DENSITY SEPARATION BY A NONAQUEOUS SOLVENT OF FINE GROUND AND AIR-CLASSIFIED FLOUR FRACTIONS.

by

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INTRODUCTION

Wheats differ in protein content from year to year depending upon planting dates, available rainfall, and other environmental and agronomic conditions. However, mills having the technics of fine grinding and air classification can furnish wheat flours of constant levels despite seasonal and area variations.

Fine grinding and air classifications permit the miller to produce more uniform flours, a wider range of them from a given wheat supply and to handle wheats of abnormal protein contents due to variations in growing conditions or available supply.

Increased attention has been directed toward study and development of methods for production of protein concentrates and isolates. These processes involve aqueous wet milling fractionation of the cereal flour or separation of its components by mechanical means.

The dry operation of protein shifting by air classification has reached the status of a commercial operation in the wheat milling industry. By this process, the intracellular constituents are concentrated on the basis of size and shape through the use of an air classifier.

The principles of air classification are based on the differences which exist between endosperm chunks and the individual protein particles and starch granules. Particles of different shape, size and density can be separated and concentrated into groups more uniform in chemical and physical characteristics than the heterogeneous parent flour.

REVIEW OF LITERATURE

Introduction

The baking properties of a flour depend upon a number of factors of which protein content is one of the most important. A miller, when producing flour for a particular baking purpose, must select and blend his wheats accordingly, and he is often faced with having to use expensive wheats to satisfy particular requirements. Air classification and fine grinding present an alternative method of adjusting the protein level in flour and it can have important economic advantages in permitting the miller to make greater use of cheaper wheats.

Fractionation of wheat flour by fine grinding and air classification has made it possible to produce a wide variety of materials differing markedly in chemical composition and in physical properties. The ability to regulate the protein level of a flour fraction results from the nature of the structure of the wheat kernel and the proper application of processing methods.

In order to understand the reason why a selective shift of protein and starch occurs when a conventionally milled or finely ground flour is subjected to air classification, one must be familiar with the particle size and relationship of the various endosperm constituents.

Structure of Wheat Endosperm

In a simplified description of the starchy endosperm, Elias (12,13) described a single endosperm cell as numerous starch granules imbedded in a protein matrix and enclosed by an extremely thin cellulose wall.

The three basic types of starchy endosperm cells found in flour particles, peripheral, prismatic and central, represent three distinct regions of the wheat kernel. The peripheral cells lie directly beneath the aleurone cell layer. The cheeks of the kernel contain the central cells while the prismatic cells extend nearly to the crease from the back to the kernel.

The cells contain starch granules, which broadly fall into two groups (26):

1. Spherical or slightly polyhedral granules ranging in diameter from under 1 to about 10 microns.
2. Lenticular granules from roughly 15 to over 40 microns.

A diagrammatic representation of the process is given in Figure 1.

The endosperm protein has been described by Hess (21). He has divided it into two fractions, the wedge protein and the adhering protein. The wedge protein occupies the wedge-shaped spaces between the curved surfaces of starch granules and represents the portion of the proteinaceous matrix which is freed during the milling process. The adhering protein is very tightly joined to the surface of the starch granule.

Two types of endosperm cell protein have been distinguished. One consisting of salt soluble albumins and globulins, has been equated with functional cytoplasmic and membrane protein. The other, the gluten forming gliadin and glutenin is considered to be storage protein produced in discrete particles by protein-forming proteoplasts comparable to the starch forming amyloplasts (42).

Fine Grinding

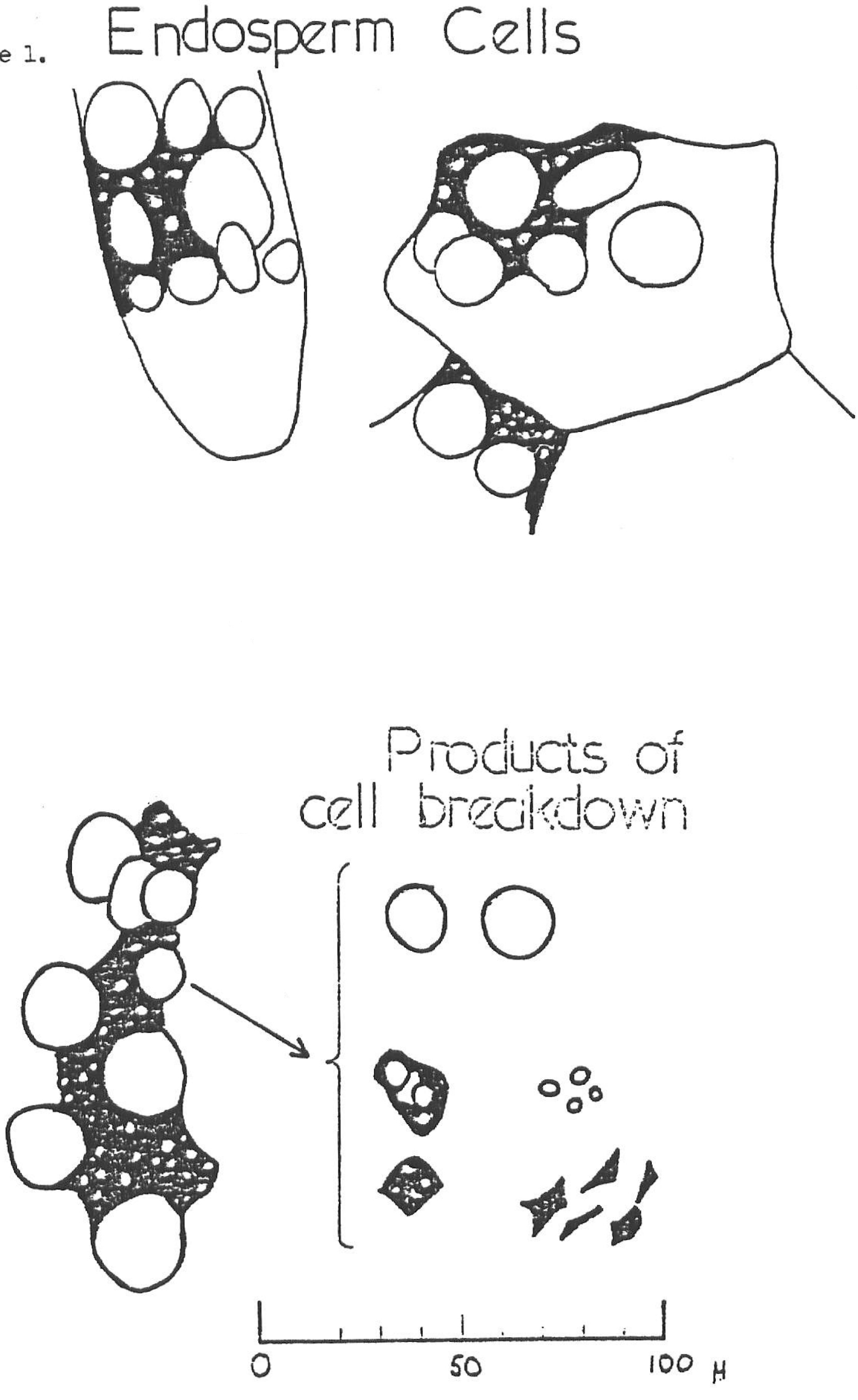
Flour consists of the starch granules, protein particles and particles of endosperm. It is generally recognized that there is an optimum granulation for flour, depending on products to be made from it. Unfortunately, however, the effect of granulation on product quality is often obscured by the fact that starch damage increases as the particles of endosperm are reduced in size. Both granulation and starch damage are related to the type (hard or soft) of wheat milled and to the type and severity of the grinding process (35).

The specifications of a patent assigned to the Pillsbury Company (45) reports that roller mills are not good for fine grinding of flour for most uses. Roller mills produce too much heat and pressure which causes changes in the properties of the protein and damage starch. The patent specifications suggested the use of impact milling to desintegrate the chunks of endosperm and fluid activated rubbing a multiple oblique impact steps to surface down the starch granules.

Ball Milling

Jones et al (26) reported that ball milling is capable of reducing flour particles very finely but the starch granules are thereby extensively shattered. This effect would result in altering the baking quality; moreover the protein in the air classified fraction would be diluted with fragments of broken starch granules. On the other hand, an appropriate grinding process should reduce the protein matrix as much as possible into particles smaller than the lenticular granules. Otherwise, the matrix may merely enter the intermediate fraction, in the form of clusters, and prevent any marked reduction in protein content of this fraction.

Figure 1.



Impact Mill

Impact grinding has been applied with the objective (15) of reducing flour to particles under 50 microns, an effect which it can accomplish without serious injury to starch granules.

When operated at a suitably high speed, the effect of the mill is undoubtedly due less impact than to the influence of alternating air disturbance on the particles traversing the orbits of the pins.

Graham (10) reported that pin mill speeds of 350 and 400 ft/sec caused remarkably small amounts of starch damage in relation to the degree of reduction. These speeds did not break up the protein adequately. At 750 ft/sec, the protein was broken up but much starch damage resulted.

Kaiser (27) described the various particle paths in pin mills (alpine types) and reported that starch granules can withstand 440-600 ft/sec with some rupture resulting at 820 ft/sec. He mentioned the importance of maintaining baking quality by not having excessive damage starch and keeping temperature rises low.

Micronizer

Fluid energy mills, such as the micronizer (2), are capable of desintegrating the endosperm mass very extensively - without rupturing the starch granules to a great extent.

Air Classification

Air classification can alter the protein content by separating flour particles according to size, shape and density.

The first step is to reduce the size of particles by grinding. This facilitates air classification. The second step is to pass the flour through an air classifier. In this step, the flour is classified or separated according to size, shape and density. The smallest particles are highest in protein and also make up the smallest portion of total flour. The largest portion is lower in protein than the parent flour (original flour) as a result of removing the high protein fraction. The next step is to air classify the largest portion to obtain another fraction of differing protein content (46).

The response of hard wheat to fine grinding and air classification is not the same as the response obtained with soft wheat flours. Stringfellow and Peplinski (49) found that Kansas hard red winter wheat could be separated into fraction ranging from 5.4 to 27.9% in protein content. If fine grinding was employed, the range was from 4.3 to 31.7% protein. However, not all of the hard red winter wheat varieties were similar in their response. Bison and Triumph varieties, when reground three times and classified into eight fractions, had protein shifting values of 60 and 59%, respectively. This is compared to protein shifting value of 39 and 36% for Comanche and Pawnee, respectively. The hard red winter wheat (HRW) were classified without prior fine grinding. The percent of protein shift was 34, 34, 20 and 20 for Bison, Triumph, Comanche and Pawnee varieties, respectively; thus, not only variety affect the amount of size reduction during milling, but it also influenced the response to fine grinding.

Density Separation

Sedimentation in aqueous media is a classic method for separating wheat flour into four fractions: water-soluble material, starch tailings, gluten and prime starch. Together with the lipids, these four fractions have constituted the basis of flour chemistry.

Water, which is a highly polar and reactive solvent, disrupts, aggregates and extracts water-soluble material; gluten proteins are hydrated and agglutinated to form gluten, and starch is released. The properties of water that make it such an effective fractionation medium, however, can cause irreversible chemical and physical changes, and isolated fractions may not represent components as they appear in the original flour or endosperm (16, 28). Relocations, reactions, and interactions caused by exposure to water, however, can be detrimental to studies in which the objectives are location, isolation or characterization of components in their native states or a combination of these. They can also be detrimental when it is desirable to maintain specific dissociations (eg, starch-protein), particle size distributions or other physical characteristics of the unfractionated flour.

To avoid solvent effects, density separations in nonaqueous media have been used for isolation and purification of small amounts of specific wheat fractions and for morphologic studies (4, 5).

The flotation technique developed by Hess (22, 23) separates flour components on the basis of their particle density. The use of nonaqueous solvents ensures that the protein fractions remain dry, and that gluten formation is prevented. As in the case of air classification, the high protein fractions are enriched in the gluten forming components (gliadin-glutenin proteins) while the starch rich fractions contain a higher proportion of water soluble proteins than the original flour (52).

The technique works better on protein or starch concentrates from the air classification process, but may be applied directly to straight run flour after pin milling.

Solvents usually employed are chloroform, carbon tetrachloride, or tetrachloroethylene, in which the density has been adjusted to between 1.31 and 1.51 by the addition of appropriate amounts of benzene or toluene. Wedge-shaped pieces of storage protein (density approximately 1.30) rise in solvent mixtures of density 1.32 to 1.34.

Starch granules (density approximately 1.49) sediment in mixtures of density 1.47 to 1.49, but float in mixtures of density 1.52 in which aleurone cell contents (density approximately 1.52 to 1.54) sink (43).

According to the study done in 1978 by Dengate et al (9), they conclude that approximate densities of wheat starch are:

1.6 g/cm^3 for dry granules

1.5 g/cm^3 for air equilibrated granules (10-15% m.c., dry basis)

1.3 g/cm^3 for hydrated granules

The protein particles in flour have lower densities than the starch granules. The small starch granules are generally higher in density than the larger ones (21).

Gracza (17, 18) found that the specific gravity of the individual fractions obtained from air classification of hard spring wheat flour ranged from 1.430 g/cm^3 for the high protein fraction to 1.465 g/cm^3 for the high starch fraction. The parent flour had a specific gravity of 1.447 g/cm^3. This is in contrast to a soft wheat flour whose individual fractions ranged in specific gravity from 1.403 to 1.487 g/cm^3. From microscopic examination of air classified fractions of both hard and soft flours, Gracza noted these differences. The protein particles were smaller, thinner, and less irregular in hard wheat flour; the starch granules were flatter and more lenticular in hard wheat flour; and the surfaces of more starch granules were free of protein in the soft flour. Soft wheat flour contained more large elementary starch granules; and the endosperm chunks of hard wheat flour have polygonal shapes with distinct edges.

Baking Performances of Fine Ground and Air Classified Fractions

It is generally recognized that there is an optimum granulation for flour, depending on the product to be made from it. However, the effect of granulation on product quality is often obscured by the fact that starch damage increases as the particles of endosperm are reduced in size. Both granulation and starch damage are related to the type (hard or soft) of wheat milled and to the type and severity of the grinding process (2).

Cakes

A finer granulation is generally considered to be desirable in cake flour, and the pin milling of cake flour is widely practiced. Mertz and Nordstrom suggested that flour is improved for cake making when the coarser flour particles are reduced to an average size below 35 microns. They also suggested the importance of maintaining starch damage below 5% as measured by an enzyme misceptibility test.

According to Berry et al (2), pin milling from five to ten times in no case improved the cake more than pin milling for fewer times. The results found suggest that reduction in size of flour particles may be the most important change which occurs during pin milling. Damage to flour components other than the starch may also be important. The changes due to pin milling affect the way flour hydrates, which in turn affect the viscosity and possibly the colloidal properties of batter, as well as the quality of the baked cake.

Breakmaking

Bread performances tests were made on several air classified flour fractions by Wichser (57) and reported the following results.

The 20% protein fraction alone was not well suited for making bread, but when supplemented with the chunk fraction, gave a good bread flour.

The loaf produced from the chunk fraction had a large loaf volume, bright crumb color, close and silky cell structure, and a smooth and velvety texture.

The bread made from a low protein-starchy fraction gave poor quality loaves.

Cookies

According to Ruiz (58), the cookies made with flour with low average particle size were of poor quality even though protein content was of an acceptable level. It was also the same for parent and fraction EE when reground to the lower particle size. However, cookies made with flour blend of D, E, EE (EE unground) fraction showed an increase in cookie quality as the protein and particle size increased. Fig (2).

Fraction EE, unground, showed the largest average diameter among the fractions, however, fraction EE unground of reground flour showed the best cookie appearance.

METHODS AND MATERIALS

General Method

The objective of this study was to evaluate the amount of free protein and free starch available in the fine ground and air classified fractions performed by three fine grinders and a turbo air classifier.

The particles produced in the reduction of the endosperm cells by either classical milling operations or fine grinding can be categorized into three major groups:

- Free starch granules
- Free wedge protein particles
- Clusters of cell fragments

The breakdown of endosperm material by severe mechanical treatment resulted in the freeing of considerable protein and starch.

It was possible to concentrate the starch and protein into different fractions using the air separation method. Several regrindings have been found to be very effective in increasing the yield of the high and low protein fractions and in decreasing the amount of coarse material.

Density fractionations of all fractions obtained were performed using benzene - carbon tetrachloride solutions adjusted to density of 1.38 (free protein) and 1.48 (free starch).

Fine Grinding

An untreated, straight grade flour milled from hard red winter (HRW) analyzing 11.8% protein content on a 14% moisture basis, was used as a source material. It was reground five times in order to reduce the flour to a finer average granulation.

Three different grinders:

- The magic mill
- The pin mill
- The udy mill

were used to perform the fine grinding. A sample of each pass was collected for analysis.

Air Classification

The flour was fractionated as milled and also after regrinding by one pass through the Alpine pin mill at 14,000 rpm.

The unground flour was first fractionated by the turbo air classifier using adjustment settings known to provide good protein shifts.

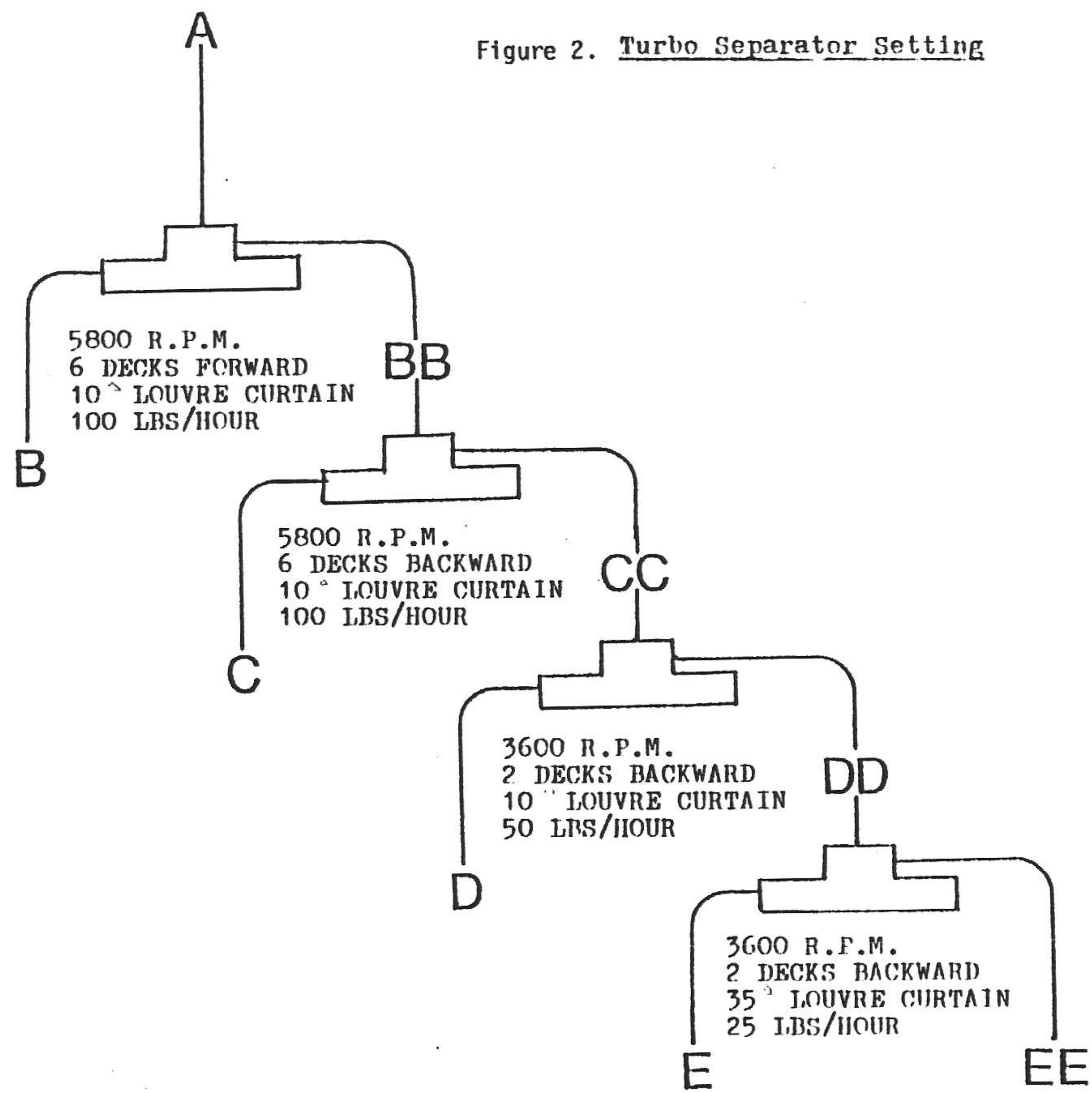
Four fine fractions were obtained by making four separations using the settings shown in Figure (2). These four separations or "cuts" were designated, in the order accomplished, as B, C, D and E. The parent flour is called A; the fine fractions were designated by single letters, B, C, D and E; and the coarse fractions were designated by double letters BB, CC, DD, and EE.

Samples from all fractions were collected for analysis. The scheme in Figure (3) shows the procedure and the samples collected.

Magic Mill

The magic mill utilizes a milling concept derived from the pharmaceutical industry. The machine utilizes high velocity impact to break down solids into uniform particles. The magic mill has no rubbing surfaces to wear off or wear out.

Figure 2. Turbo Separator Setting



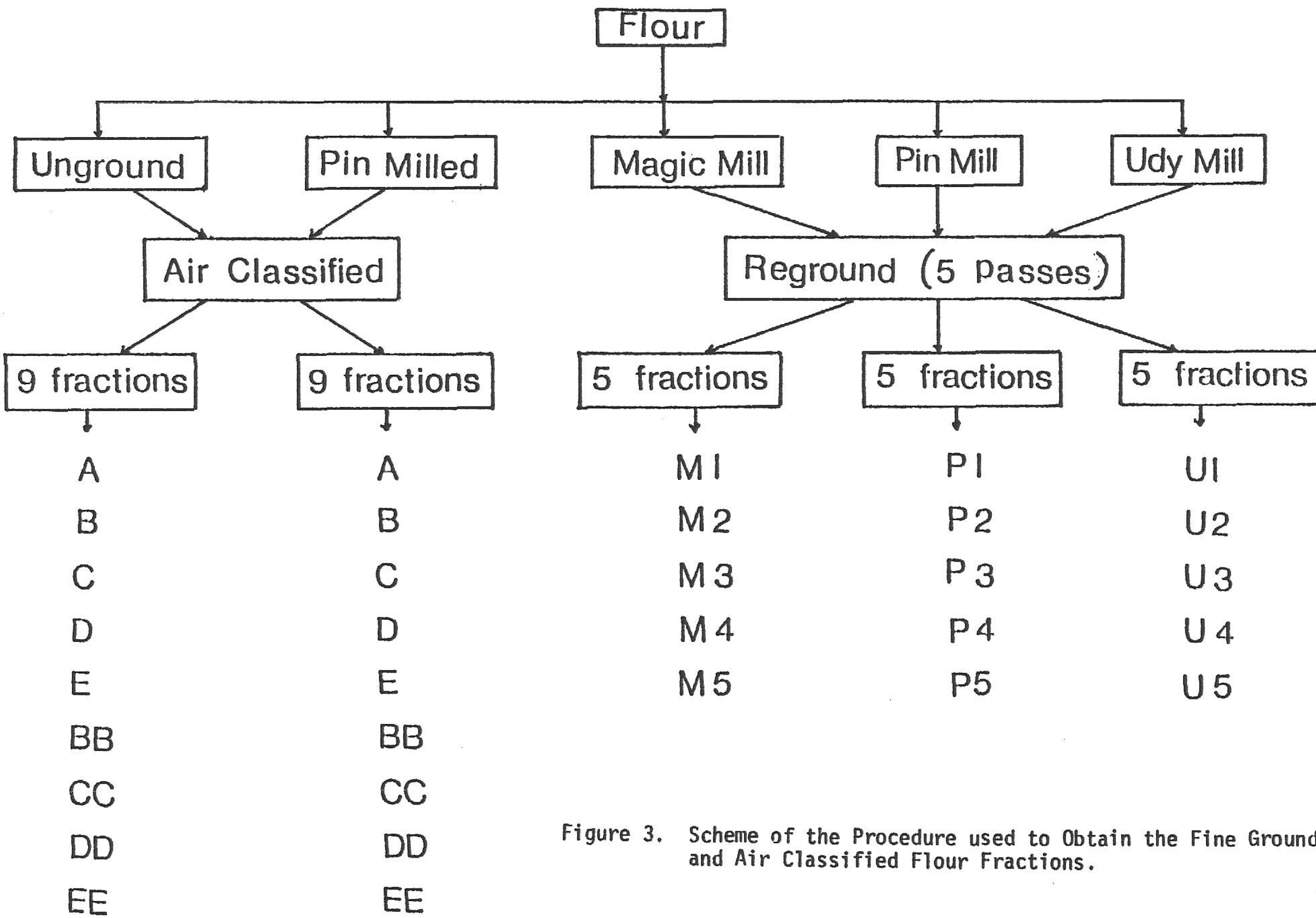


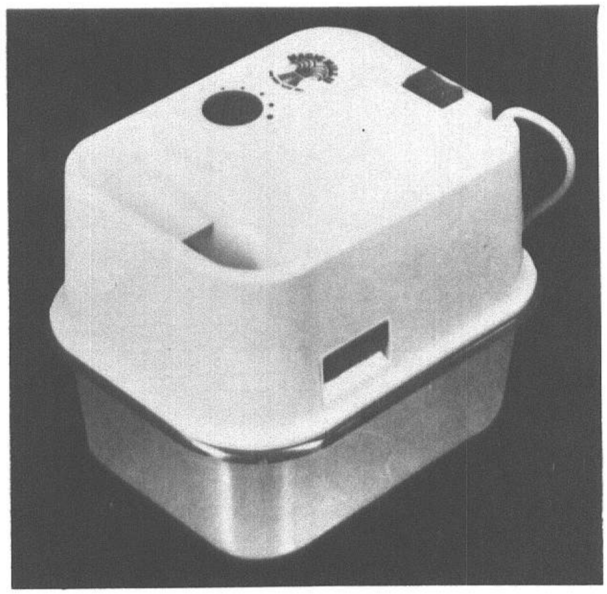
Figure 3. Scheme of the Procedure used to Obtain the Fine Ground and Air Classified Flour Fractions.

THE MAGIC MILL

A. The magic mill

B. Cut-away view of the micronizers

C. Airflow of flour created by the cyclocup



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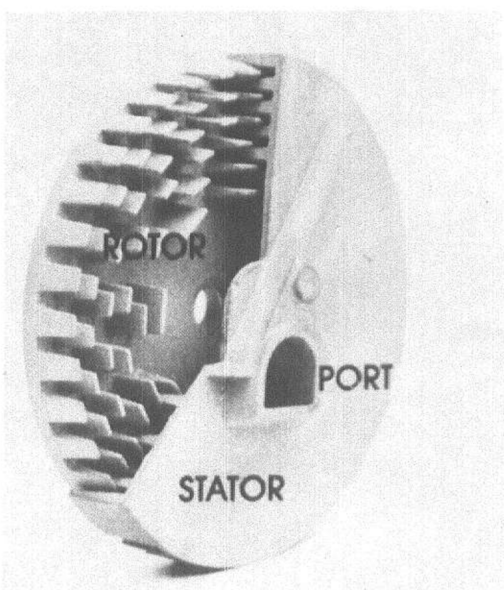

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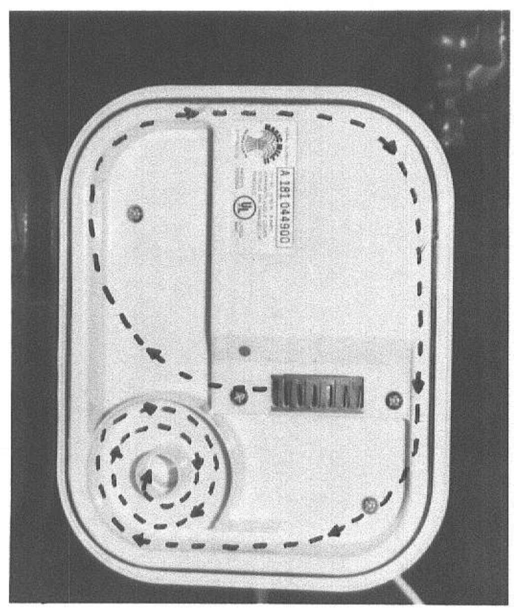


C

Magic Mill

The principle of milling utilized in micronizing is that of exploding the wheat as it comes in contact with the rotating micronizer as it moves at over 25,000 rpm.

Alpine Pin Mill

The Alpine pin mill has two pinned discs with four circular rows of pins on each disc. One disc is stationary in the Kolloplex 1602 and the other rotates at high speed. The inner rows of pins have lower lineal velocities than the outer rows of pins and the easy-to-grind material is ground by the slower pins. Harder to grind material requires higher velocities and is ground by the outer rows of faster pins. A speed of 16,000 rpm was used.

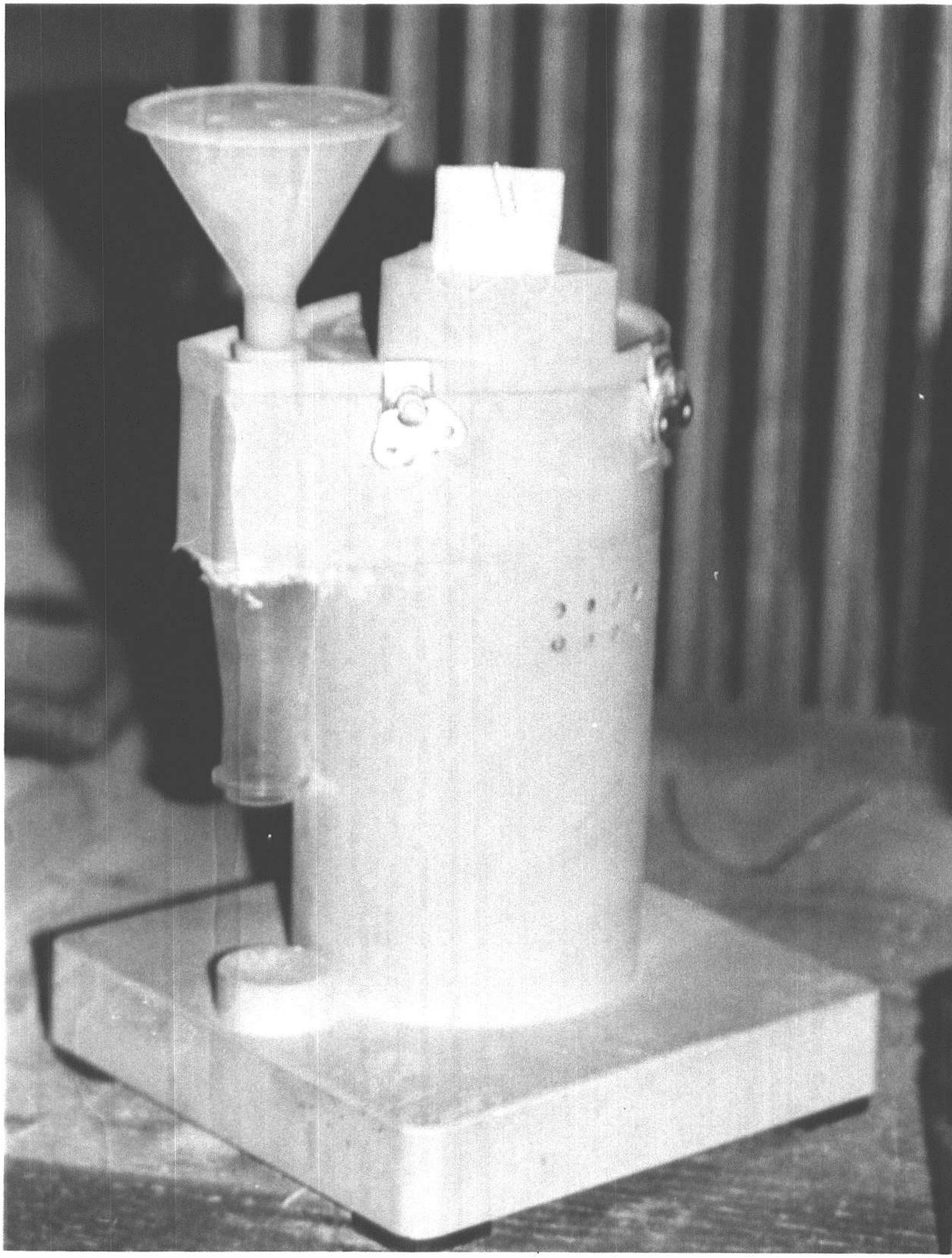
Udy Mill

Before a particle can escape from the grinding chamber it must generally be reduced in mass by the high speed impeller (about 12,000 rpm) until it is light enough to turn about 120° and follow the air flow through the screen (.020"). The baffle, as provided on the cover at the sample inlet opening, will prevent direct bombardment of the screen by incoming particles. The reduced material can be conveniently collected in a bag by connecting it at the lower cyclone outlet.

The Turbo Air Classifier

The air classifier, itself, consist of a cylindrical classifier chamber 6 inches in diameter and 4 inches high. Air was pulled by means of a fan through the 3.5 inch diameter center opening. Air entered the

Alpine Pin Mill



Udy Mill

classifier tangentially at the bottom through an inlet. Flour was fed by twin screws on top of the rotating decks.

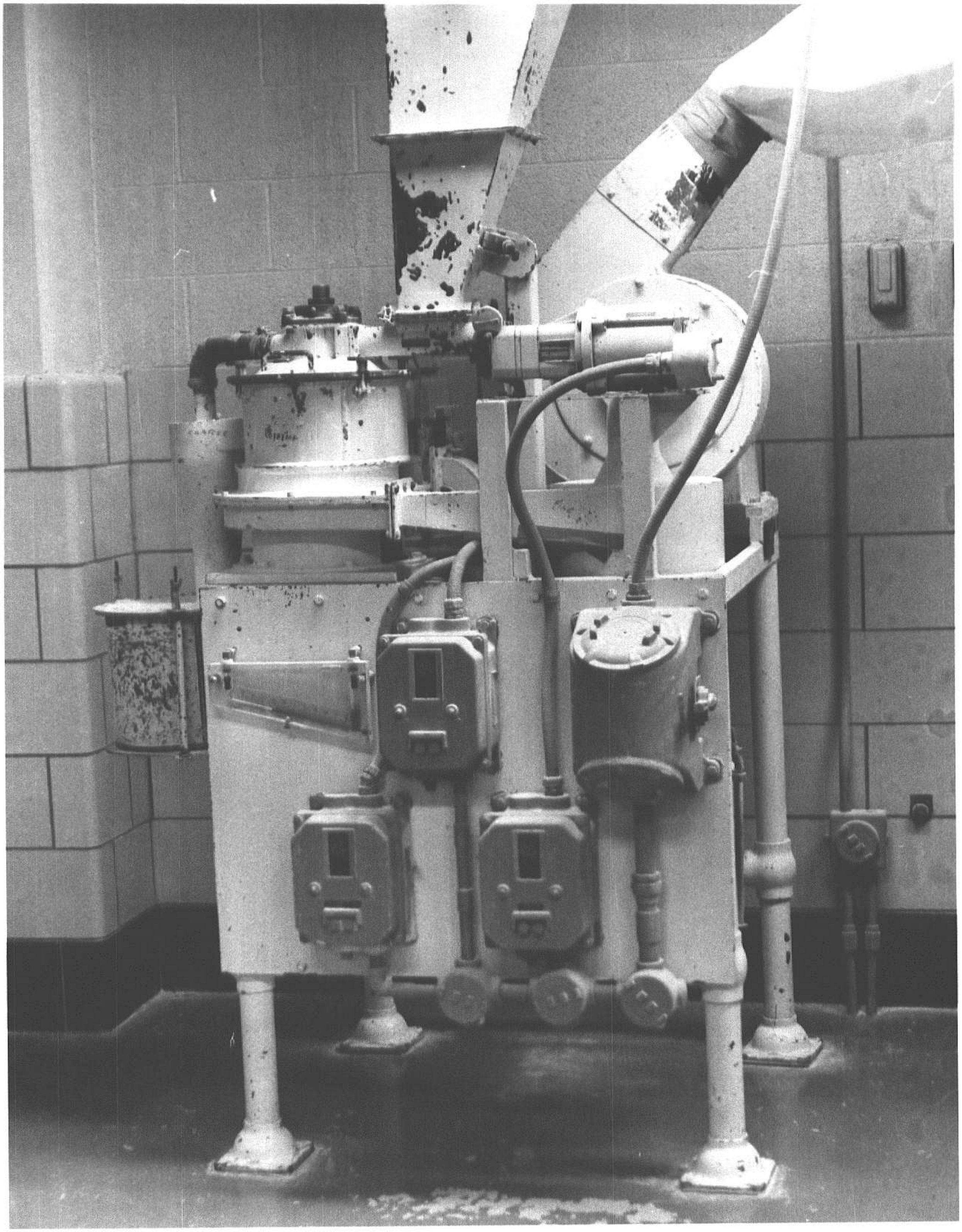
There are two forces acting on any particle at all times in the classifier: the centrifugal force which tries to fling the particle outward, and the drag force, due to the air, acting in the opposite direction. The two forces acting in opposite direction, governed the movement of the particle. A relatively coarse particle had a greater centrifugal force than the drag force. The coarse particle was thrown outward and was collected in a cyclone collector.

For the finer particles, the drag force was greater than the centrifugal force. The fine material was pulled in with the air through the center opening to a cyclone where the material was collected. The air, relatively free of particles, was blown into a filter bag by an external fan. The classifier was driven by a 3 horsepower motor operating at 3600 rpm while the fan operated at 3600 rpm with a 1.5 horsepower motor.

Density Separation

The interstitial protein of wheat endosperm can be largely separated in the native dry state from the starch by differential centrifugation of finely ground flour in mixtures of benzene and carbon tetrachloride of density intermediate between that of starch (density 1.5) and that of the protein (density 1.3).

Density separations of protein and starch fractions of flour were effective when the solvent density was intermediate between the densities of the protein and starch. Figure (4). The protein content and amount of total flour protein contained in the floating fraction was dependent on the



Air Classifier

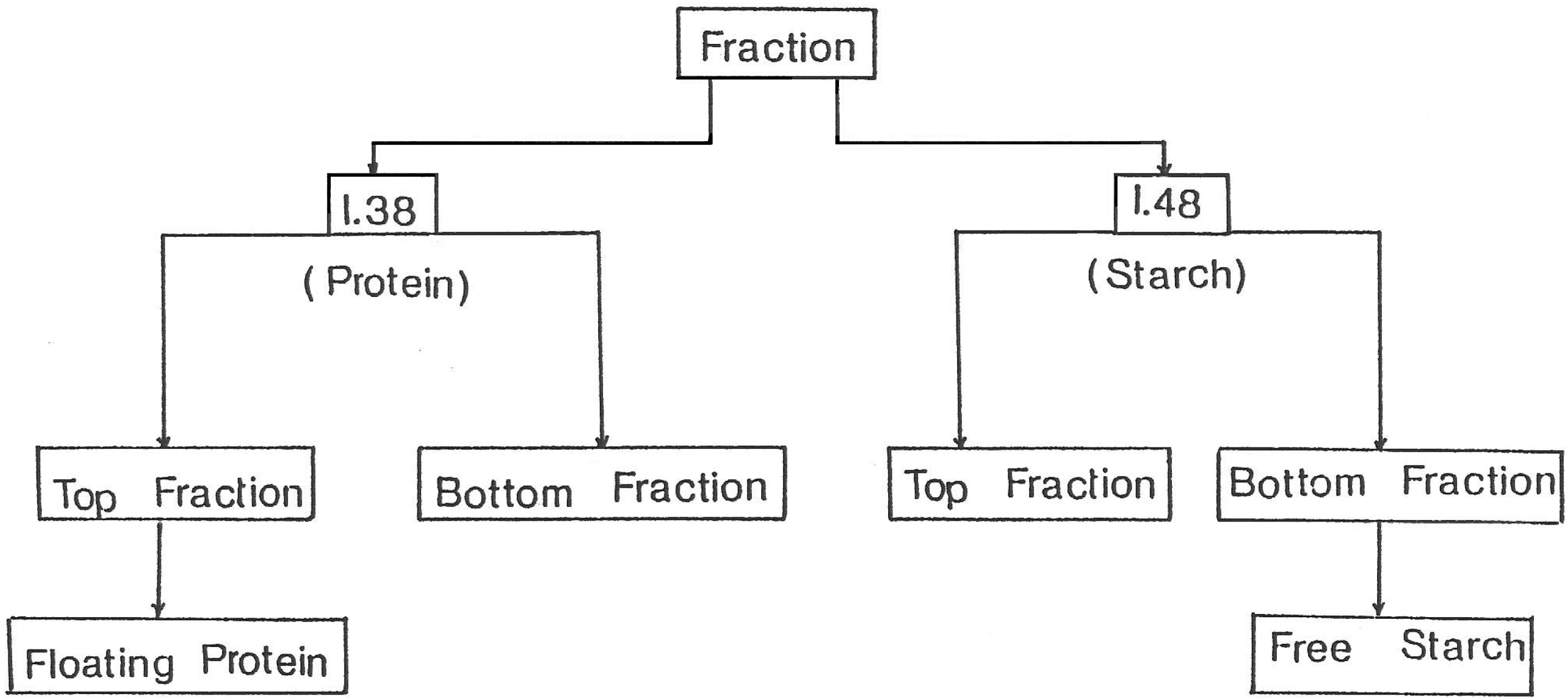


Figure 4. Scheme of the Procedure used for Density Separation

solvent and the denisty of the solvent. Additionally, the change in solvent density with temperature control was critical to the success of the protein-starch separation.

The Solvent

Carbon tetrachloride (density 1.594 g/ml at 20^{o}C) and benzene (density .875 g/ml at 20^{o}C) were blended to give mixtures with particular densities 1.38 for free protein and 1.48 for free starch.

Densities were determined with a liquid pycnometer and corrected for temperature.

The Centrifuge Bottle

A regular 125 ml pyrex bottle was used to replace a centrifuge bottle. The receptacle portion, forming the upper part and neck of the bottle, was made from a ground glass fitting (19/22) Figure (5).

Free Protein Separation

A 25 g sample of flour was placed into the centrifuge bottle which then was filled to the shoulder with the benzene - carbon tetrachloride mixture (1.38) and vigourly shaked to disperse the flour uniformely. It was then filled with solvent to the top of the neck. The bottle was wrapped with a piece of heavy rubber, ¼" thick, in order to prevent impacts against the walls of the centrifuge cup. The solvent-flour mixture was centrifuged in an International Model PR 2 centrifuge fitted with Int. 259 head at 18,000 rpm for 30 minutes.

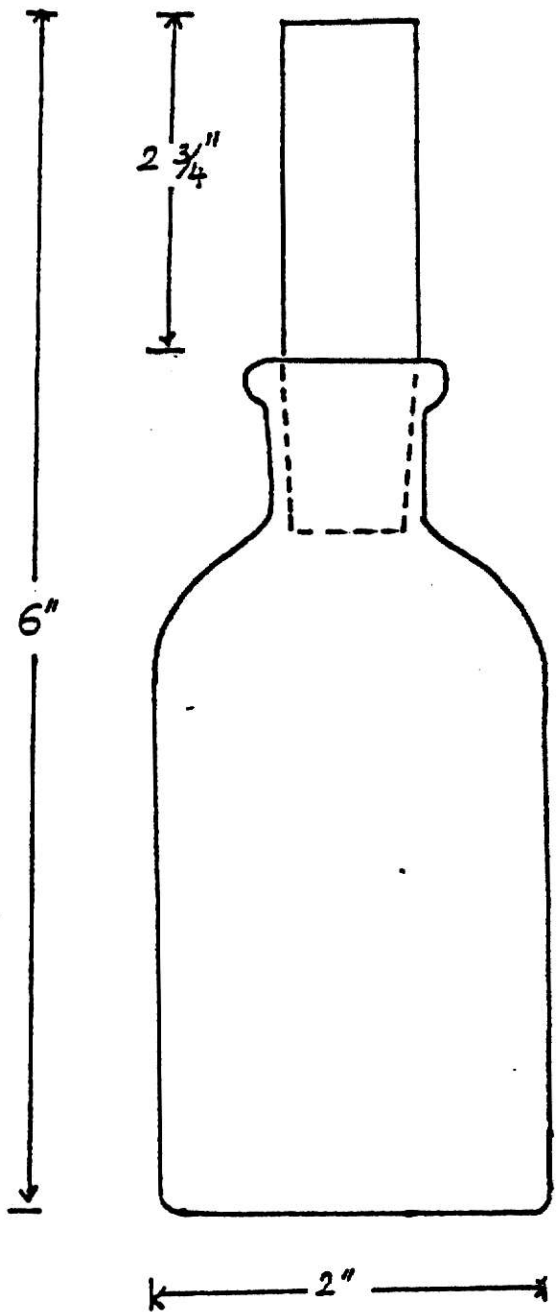


Fig 5 . The centrifuge bottle used for the density separation. The removable neck was made from a ground glass fitting ($ 19/22). The diagram is drawn in actual size.

The material less dense than the density of the solution being used as the fractionation medium was easily trapped in the stem of the centrifuge bottle.

The three phases consisted of

Top fraction -	Free protein
Intermediate fraction -	Solvent layer
Bottom fraction -	Residue

as shown in the diagram (6).

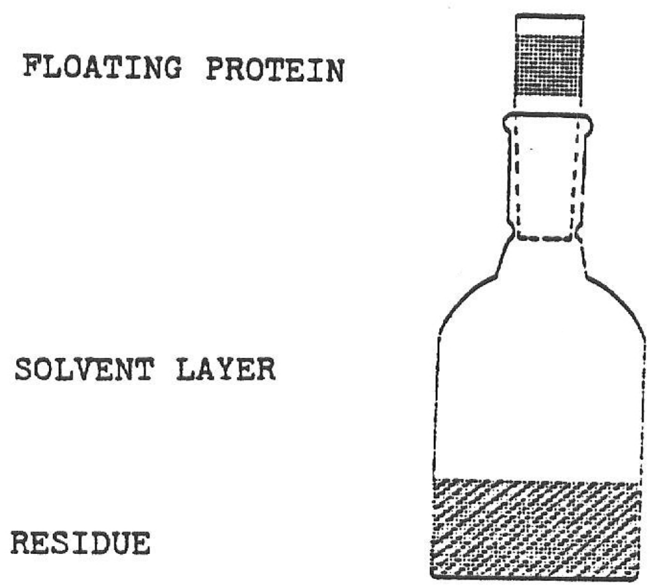


Figure (6) Separation at 1.38 Density for Free Protein

The floating protein was separated from the bottle by removing the detachable neck and washed by fresh solvent into a suction filter, then air dried at room temperature until solvent odor could no longer be detected.

The remaining solvent layer and settled bottom fraction were mixed together, then filtered and air dried. The two solid fractions were weighed then analyzed for moisture and protein content.

Free Starch Separation

For the determination of free starch, 10 g of flour was used and the same procedure was followed except for the specific density where 1.48 was used.

The three layers were as follows

Top fraction -	Residue
Intermediate fraction -	Solvent layer
Bottom fraction -	Free starch

as shown in the diagram (7).

RESIDUE

SOLVENT LAYER

SINKING STARCH

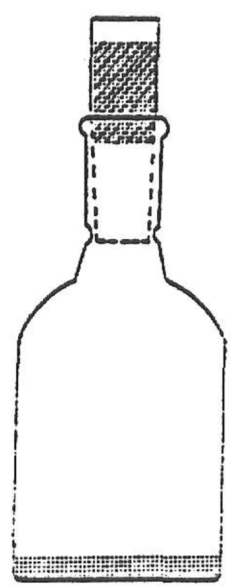


Figure (7) Separation at 1.48 Density for Free Starch

The different steps of the density separation procedure

a. The centrifuge bottle and the suction filter

b. The bottle set to receive the flour sample and the solvent

c. To fit inside the centrifuge cup and in order to protect the centrifuge bottle from breaking,the 125 ml Pyrex bottle must be wrapped with a piece of rubber 1/4" thick.

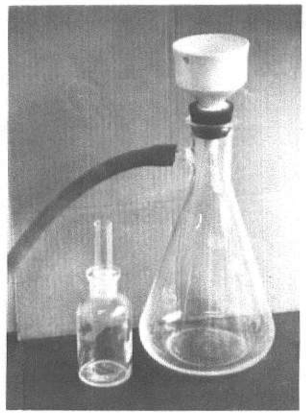
d. The solvent and flour mixture ready for centrifugation.

e. The centrifuge with its 6 cups head.

f. The appearance the mixture after centrifugation.

g. Close-up of the centrifuged mixture.The top fraction is trapped in the upper part of the neck.

h. Separation of the neck from the bottle.



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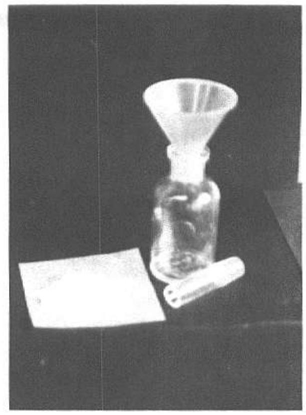

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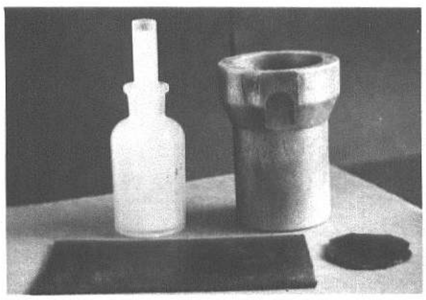

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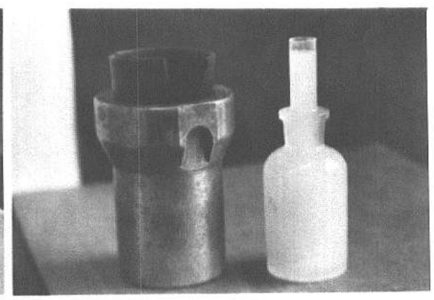

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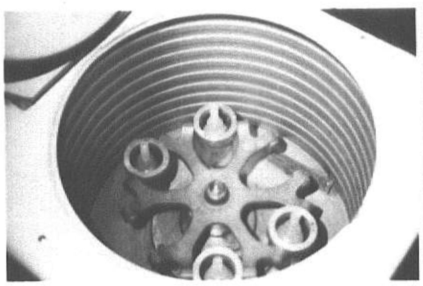

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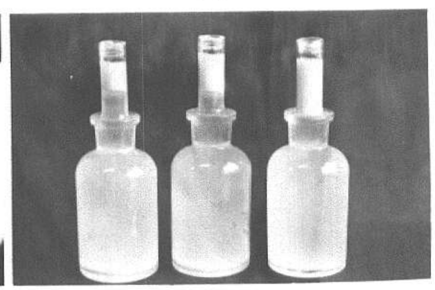

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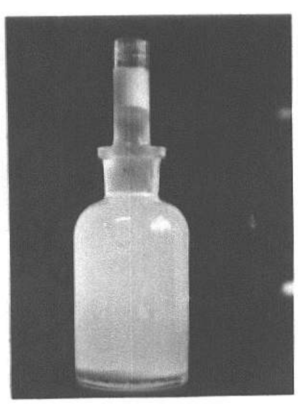


g

h

Average particle size

The Fisher Sub-Sieve Size Analyzer was used for the determination of the average particle size of a sample.

Moisture

A.A.C.C. Cereal Laboratory Methods was used for moisture determination.Evaporation of water is the principle involved.The loss in weight is calculated as percent moisture.

Protein

The Kjeldhal method was used for the Nitrogen determination. The factor used for flour is Nitrogen x 5.7.

Statistical Analysis

Comparisons employing the t-ratios were conducted to determine whether differences existed between the mean trial values of each type of separation at 1.38 density and 1.48 density.

RESULTS AND DISCUSSION

Effect of Density on Wheat Protein and Starch

A previous work was done in the early 60's by Professor Ward using 1.38 and 1.48 densities to separate free protein and free starch of whole flour.

The two densities were tested with fine ground flour and the results obtained were considered appropriate to conduct this work.

Solvent Density vs. Protein Separation

The protein content and amount of total flour protein contained in the float fraction is dependent on the solvent density.

Figure (8) shows the total flour protein separation behavior between densities of 1.34 and 1.42.

It can be seen that the protein content of the floating fraction reaches a maximum of 76.8% as the solvent density reaches 1.38. Above this solvent density, the amount of floating protein increases, but the percent protein in the float drops sharply.

Solvent Density vs. Starch Separation

The flour exhibits an increase of the amount of free starch at 1.48. The amount of the sink is 71.12% at its maximum in the curve. Figure (9). As the density is increased from 1.48, a decrease in the sink fraction is observed.

The denisty of 1.48 showed the highest amount of free starch but not the lowest in protein content. A prime interest was given to the yield but not to its purity as done for the free protein.

Fine Grinding

Fine grinding caused extensive reduction in size of endosperm particles. The results of this reduction in size were the freeing of starch granules and particles of protein matrix and an increase in the amount of small endosperm chunks.

Particle Size vs. Number of Passes

Fine grinding seemed to be quite effective in reducing endosperm to particles of small size. The magic mill, Figure (10), gave the best results, after the fifth pass, still giving finer fractions (9.5 microns). The pin mill, in contrast, seemed to reach its optimum at the third pass (11.5 microns). The udy mill was inefficient after the 2nd pass.

In the case of the pin mill, some finer particles were sucked into the filters; this may have affected the particle size of the final product.

Density Separation

Free Protein vs. Number of Passes

The histograms in Figures (11, 12, 13) show the amount of protein freed by regrinding several times.

The udy mill, Figure (11), seemed to be ineffective after the second pass while the protein purity seemed to improve slightly.

The pin mill, Figure (12), gave good results at the first pass with its purest protein content. At the second pass there was no change in the amount of free protein, but the protein purity decreased. The reason for that was the loss of finer particles (free protein) sucked through the

Specific densities vs floating protein

DENSITIES	% FLOAT	% PROTEIN
1.34	9.60	69.80
1.36	10.60	71.90
1.38	23.00	76.80
1.40	26.60	76.40
1.42	34.80	64.50

Specific densities vs sinking starch

DENSITIES	% SINK	% PROTEIN
1.46	59.70	3.60
1.47	68.24	3.50
1.48	71.12	3.20
1.49	32.60	2.40
1.50	3.20	.90
1.51	0	0

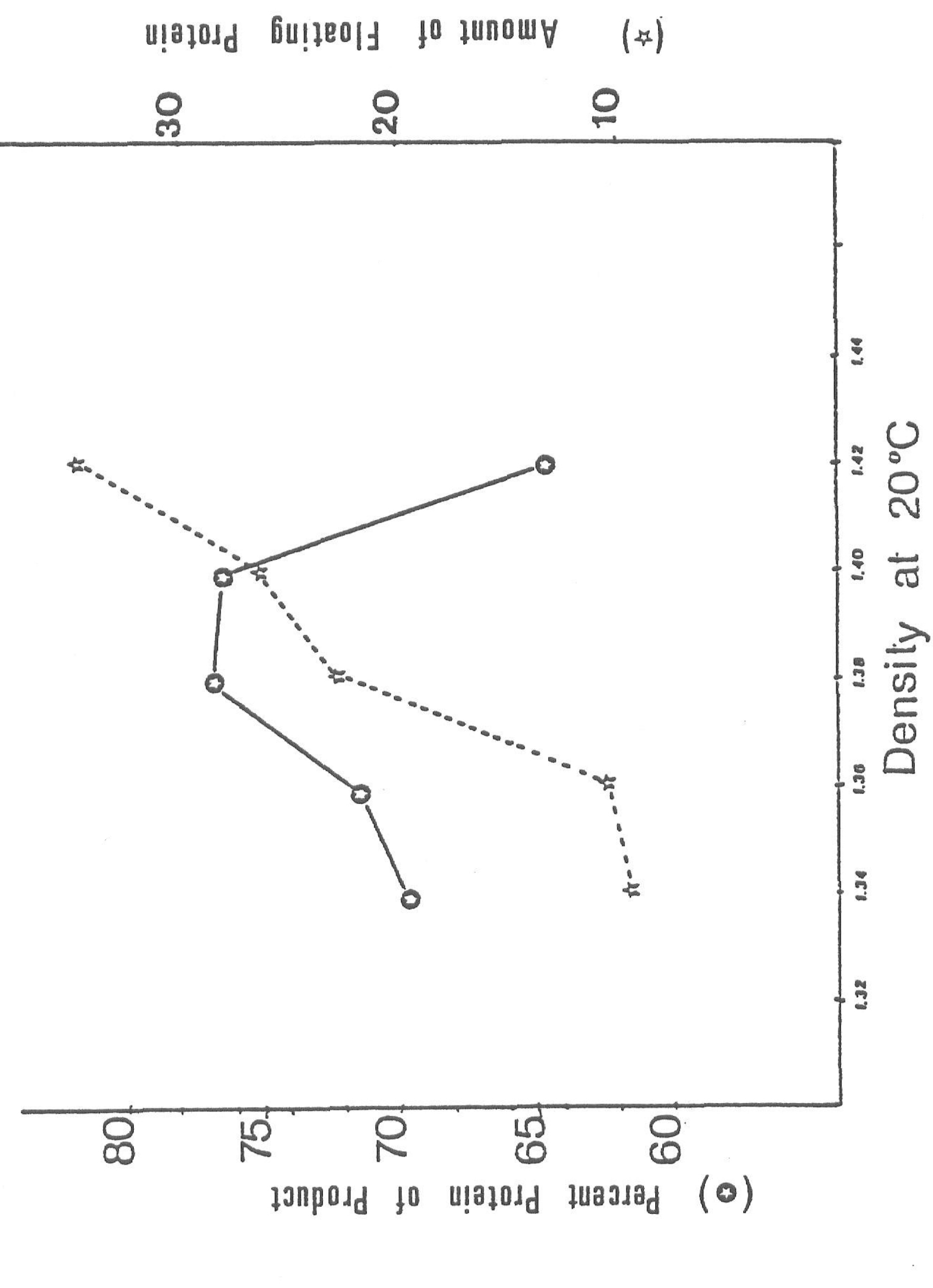


Figure 8. Solvent density vs Protein separation

Figure 9. Solvent density vs Starch separation

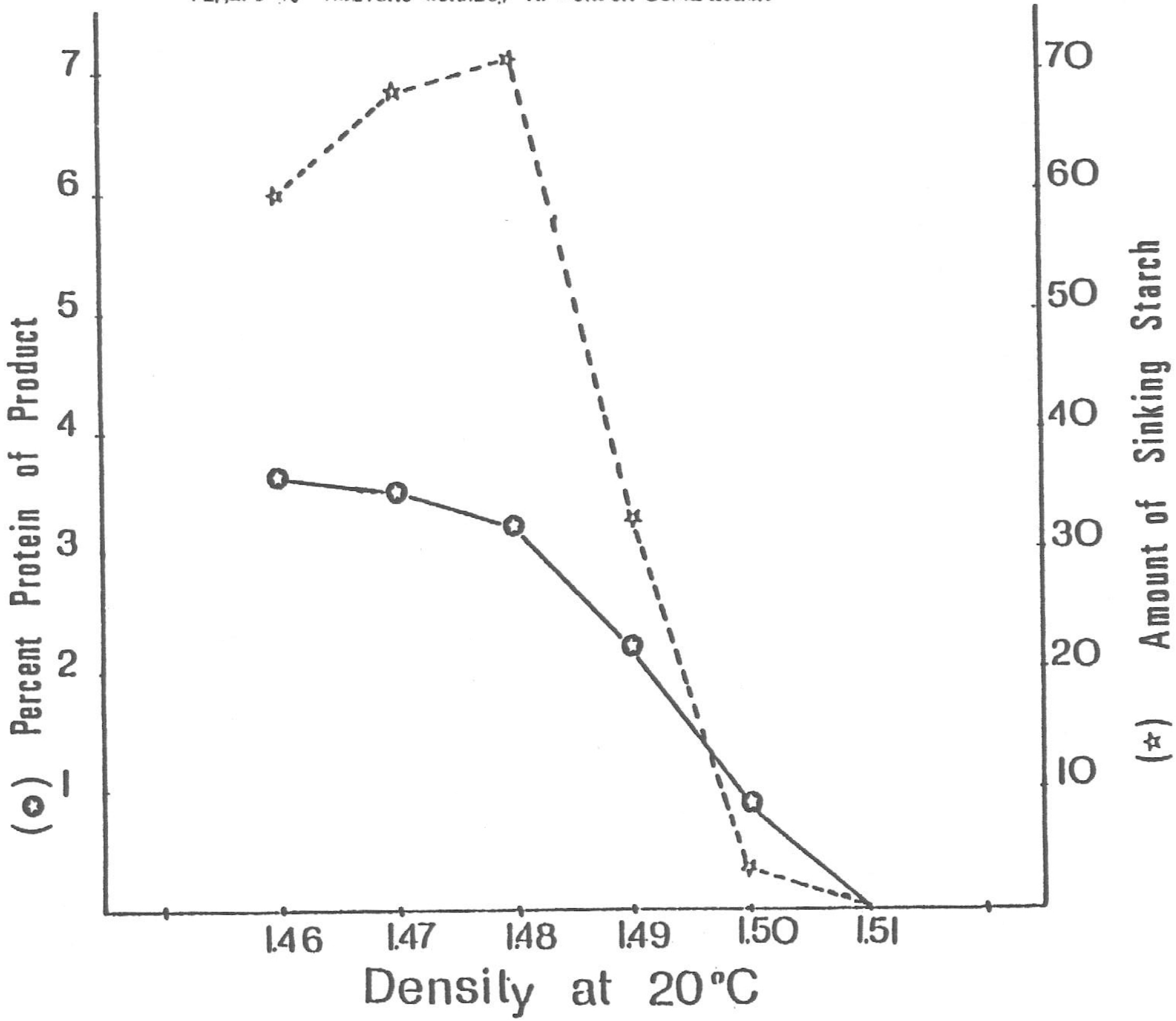


Figure 10. Relationship Between Number of Passes and Particles Size of Three Different Grinders.

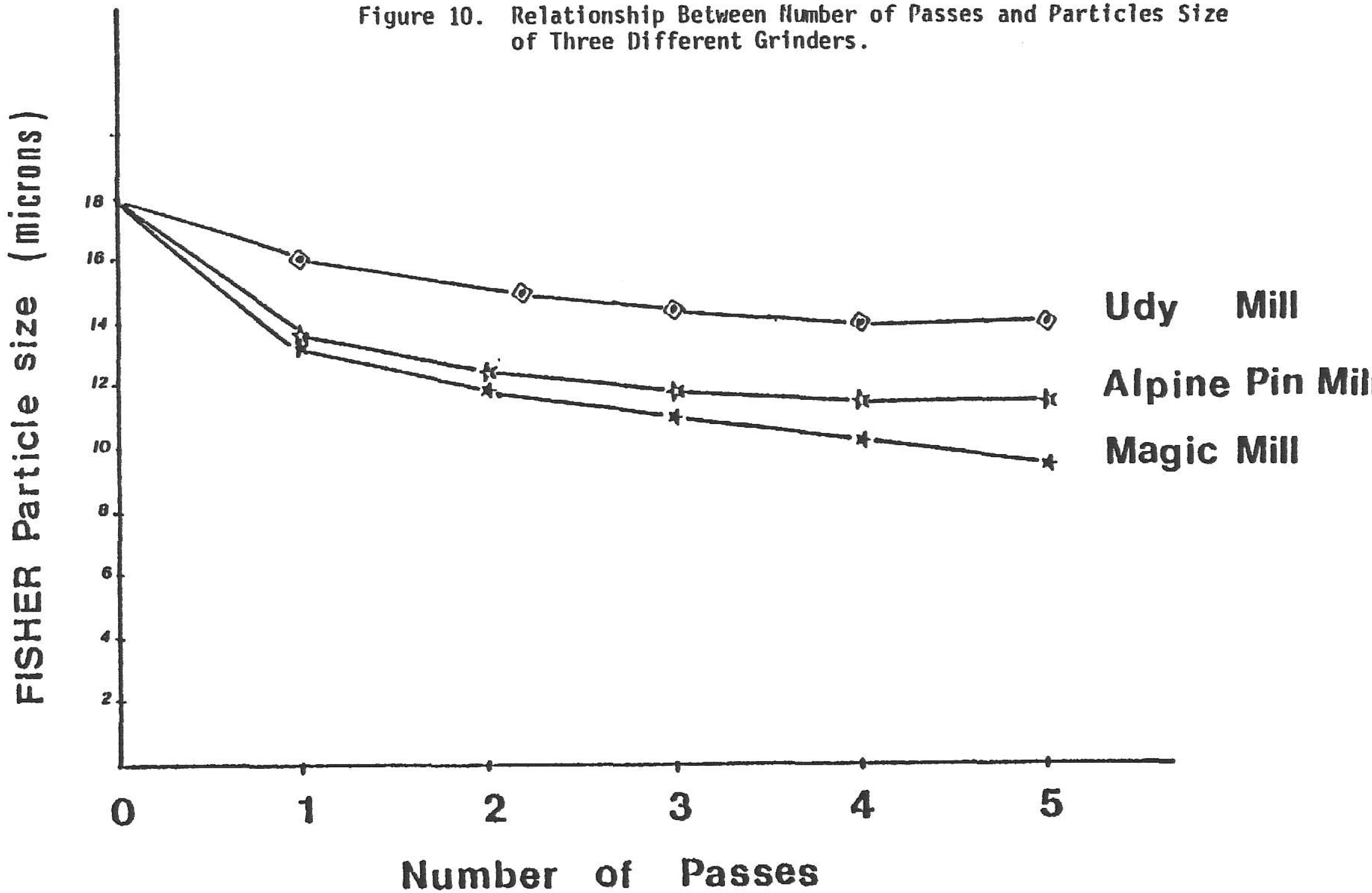


Figure 11. Amount and Purity of Free Protein Obtained at Different Passes by the Udy Mill.

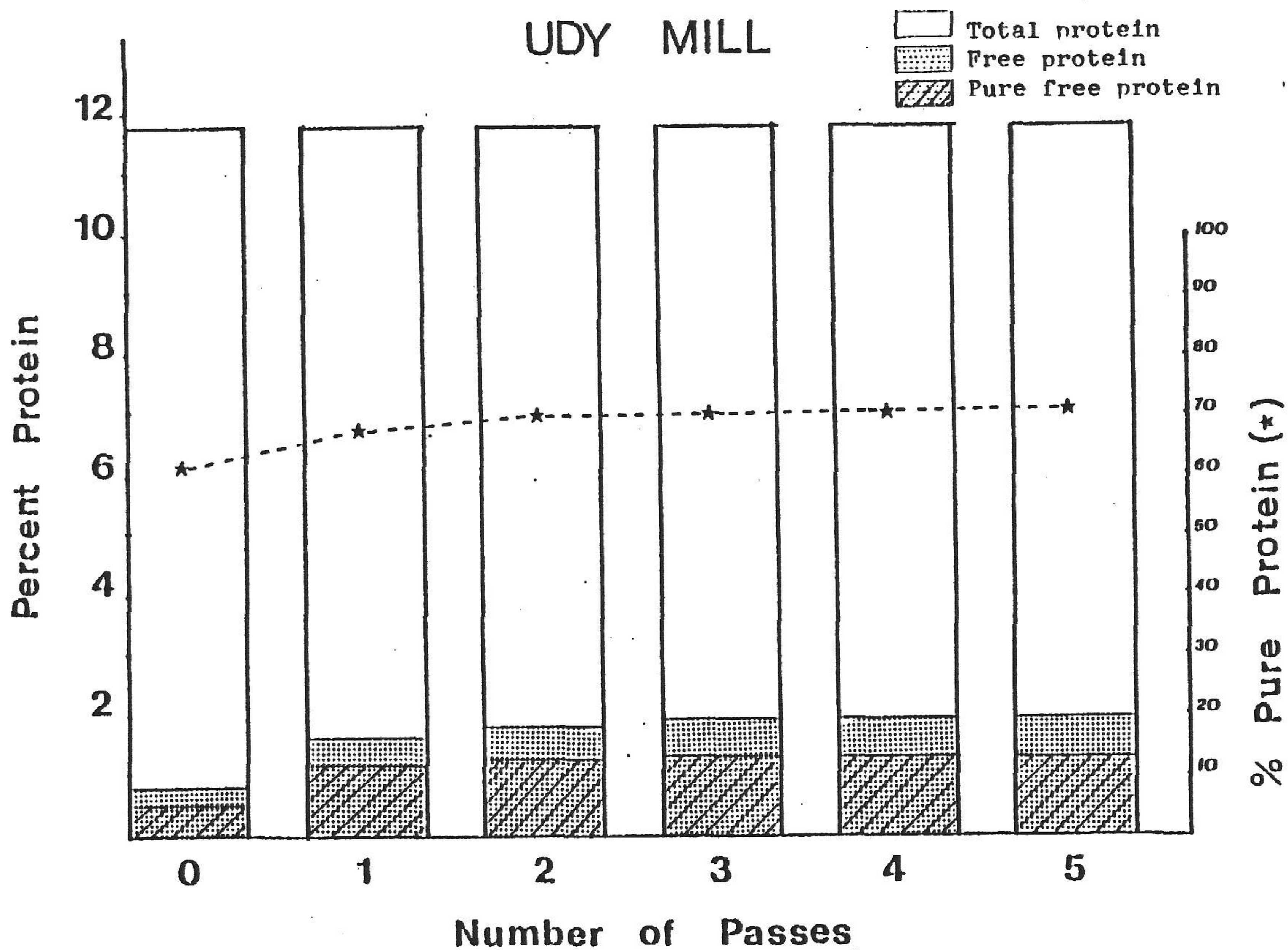
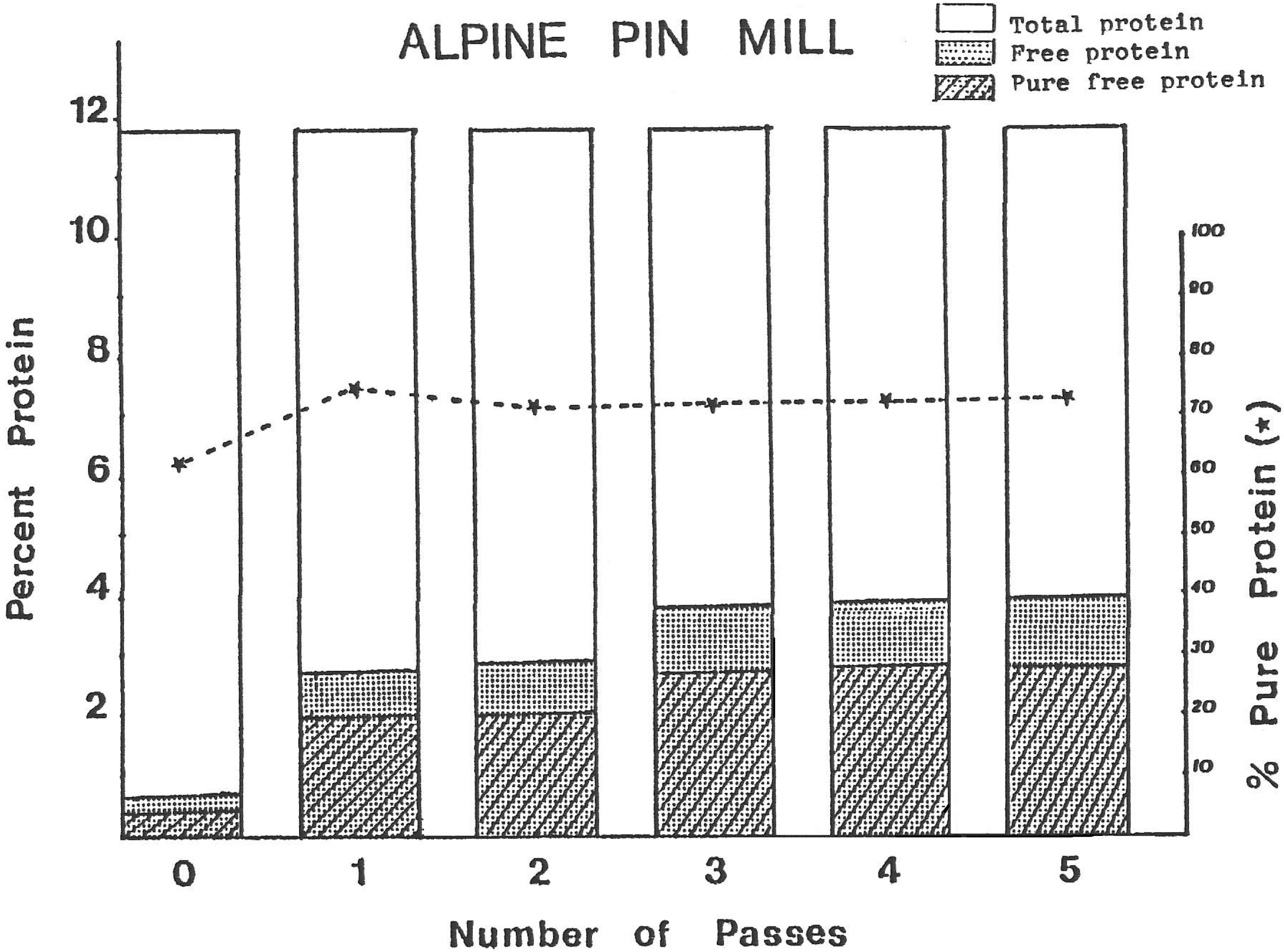


Figure 12. Amount and Purity of Free Protein Obtained at Different Passes by the Alpine Pin Mill.



filter. As it can be seen, the amount of free protein loss was not negligible, the purity of its protein showed it very clearly. The third pass was good but the purity remained almost at the same level. No changes were noticed at the fourth and fifth passes.

The magic mill, Figure (13), gave excellent results; the free protein and its purity increased progressively after each pass. The advantage of the magic mill was that all the material ground was recovered and there were no filters.

Results of the preceding separations are shown in Tables (2, 3, 4) and Figure (4). The percent of protein in the floating fraction was increased by finer grinding, as evidenced by the fact that the percent of protein of the float of magic mill flour (80.52%) was greater than that of pin milled flour (74.02%) which in turn was greater than that of the Udy mill (71.60%).

The percentage of floating protein followed the same sequence (5.16, 3.70, 1.78, respectively). Regrinding several times, however, does improve the separation significantly in the magic mill, yielding nearly 80.52% of the protein in the floating fraction. The protein content of the starch fraction is extremely low, less than 3%, which suggests that the starch could be of significant use to the food industry.

Figure (15) compares the amounts of protein freed by the three grinders.

The magic mill freed progressively greater amounts through the five passes.

With the pin mill there was an increase through the third pass interrupted at the second pass by the loss of finer particles through suction.

TABLE 2.

Magic Mill

PASSES	PARTICLE SIZE	% FLOAT (1.38)	% SINK (1.48)
1	13.25	2.66	28.20
2	11.50	3.68	45.60
3	11.00	4.12	53.00
4	10.25	4.78	61.40
5	9.50	5.16	65.00

Pin Mill

PASSES	PARTICLE SIZE	% FLOAT (1.38)	% SINK (1.48)
1	13.50	2.68	26.20
2	12.50	2.82	34.30
3	11.75	3.74	37.20
4	11.50	3.78	39.10
5	11.50	3.70	41.00

Udy Mill

PASSES	PARTICLE SIZE	% FLOAT (1.38)	% SINK (1.48)
1	16	1.50	22.20
2	15	1.72	22.80
3	14.50	1.74	23.60
4	14	1.78	24.00
5	14	1.78	23.40

TABLE 3.

Magic Mill

PASSES	% PROTEIN	% FLOAT (1.38)	% PURITY	% TOTAL FREE PROTEIN
1	11.8	2.66	70.99	15.93
2	11.8	3.68	74.69	23.22
3	11.8	4.12	76.39	27.07
4	11.8	4.78	79.68	32.20
5	11.8	5.16	80.52	35.25

Pin Mill

PASSSES	% PROTEIN	% FLOAT (1.38)	% PURITY	% TOTAL FREE PROTEIN
1	11.8	2.68	74.90	16.95
2	11.8	2.82	71.53	17.12
3	11.8	3.74	72.38	22.88
4	11.8	3.78	73.43	23.56
5	11.8	3.70	74.02	23.22

Udy Mill

PASSES	% PROTEIN	% FLOAT (1.38)	% PURITY	% TOTAL FREE PROTEIN
1	11.8	1.50	67.08	8.47
2	11.8	1.72	69.52	10.17
3	11.8	1.74	70.30	10.34
4	11.8	1.78	71.50	10.85
5	11.8	1.78	71.60	10.86

TABLE 4.

Magic Mill

PASSES	% PROTEIN	% SINK (1.48)	% PURITY	% TOTAL FREE PROTEIN
1	11.80	28.20	96.81	27.30
2	11.80	45.60	96.31	43.92
3	11.80	53.00	96.11	50.94
4	11.80	61.40	95.99	58.94
5	11.80	65.00	95.91	62.34

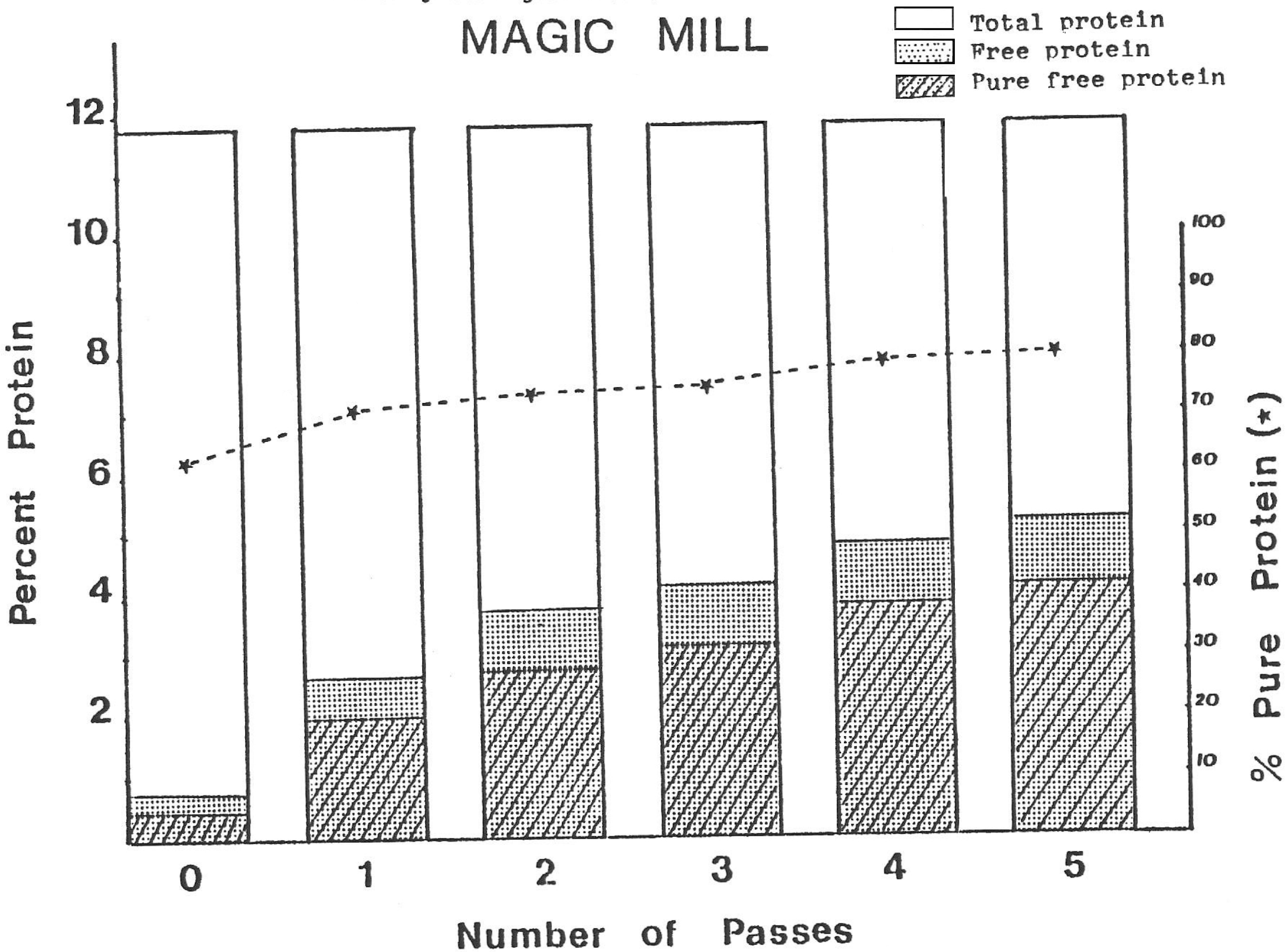
Pin Mill

PASSSES	% PROTEIN	% SINK (1.48)	% PURITY	% TOTAL FREE PROTEIN
1	11.80	26.20	97.71	25.60
2	11.80	34.30	97.20	33.34
3	11.80	37.20	97.42	36.24
4	11.80	39.10	97.49	38.12
5	11.80	41.00	97.51	39.98

Udy Mill

PASSES	% PROTEIN	% SINK (1.48)	% PURITY	% TOTAL FREE PROTEIN
1	11.80	22.20	96.58	21.44
2	11.80	22.80	96.32	21.96
3	11.80	23.60	95.93	22.64
4	11.80	24.00	96.58	23.18
5	11.80	23.40	96.84	22.66

Figure 13. Amount and Purity of Free Protein Obtained at Different Passes by the Magic Mill.



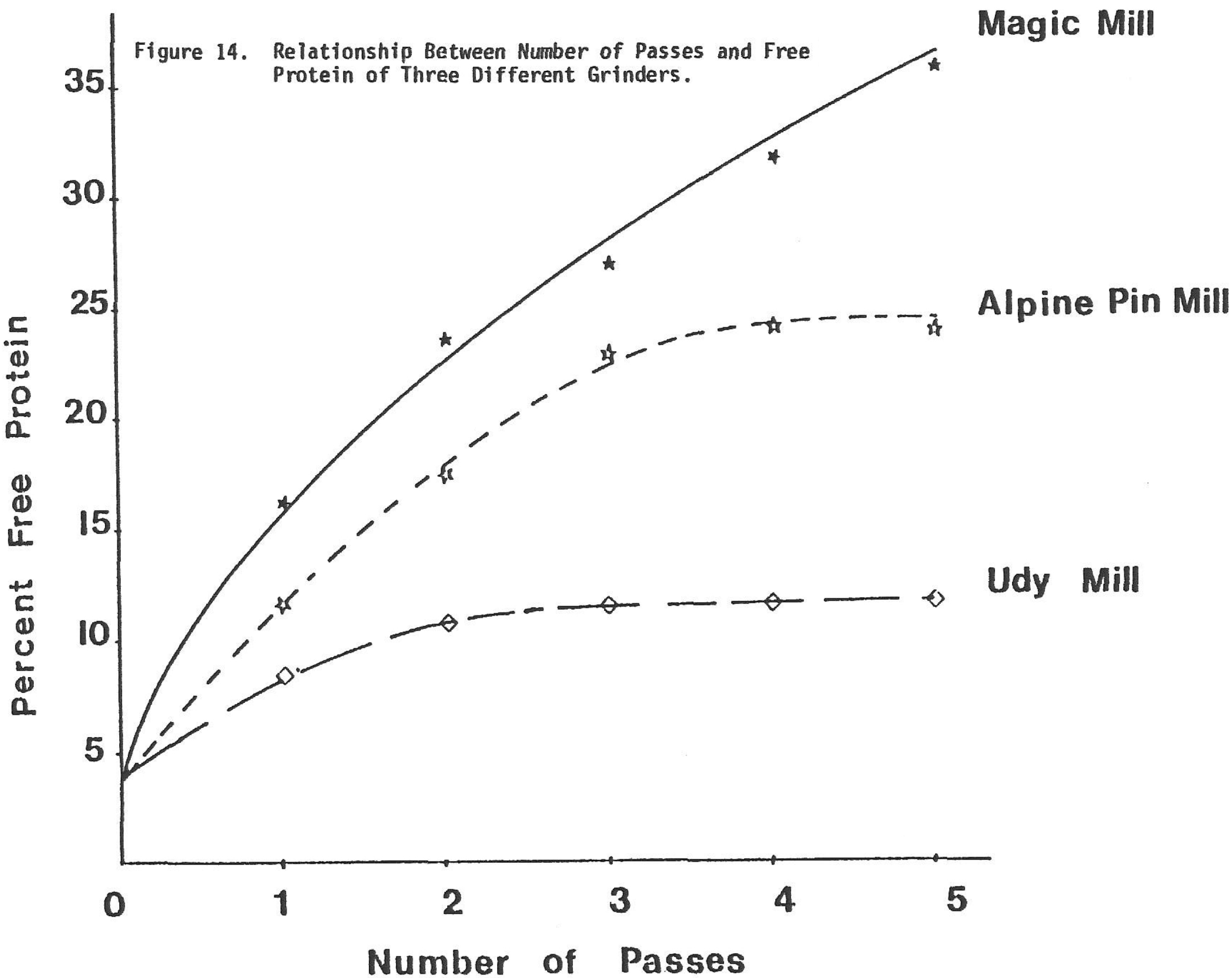
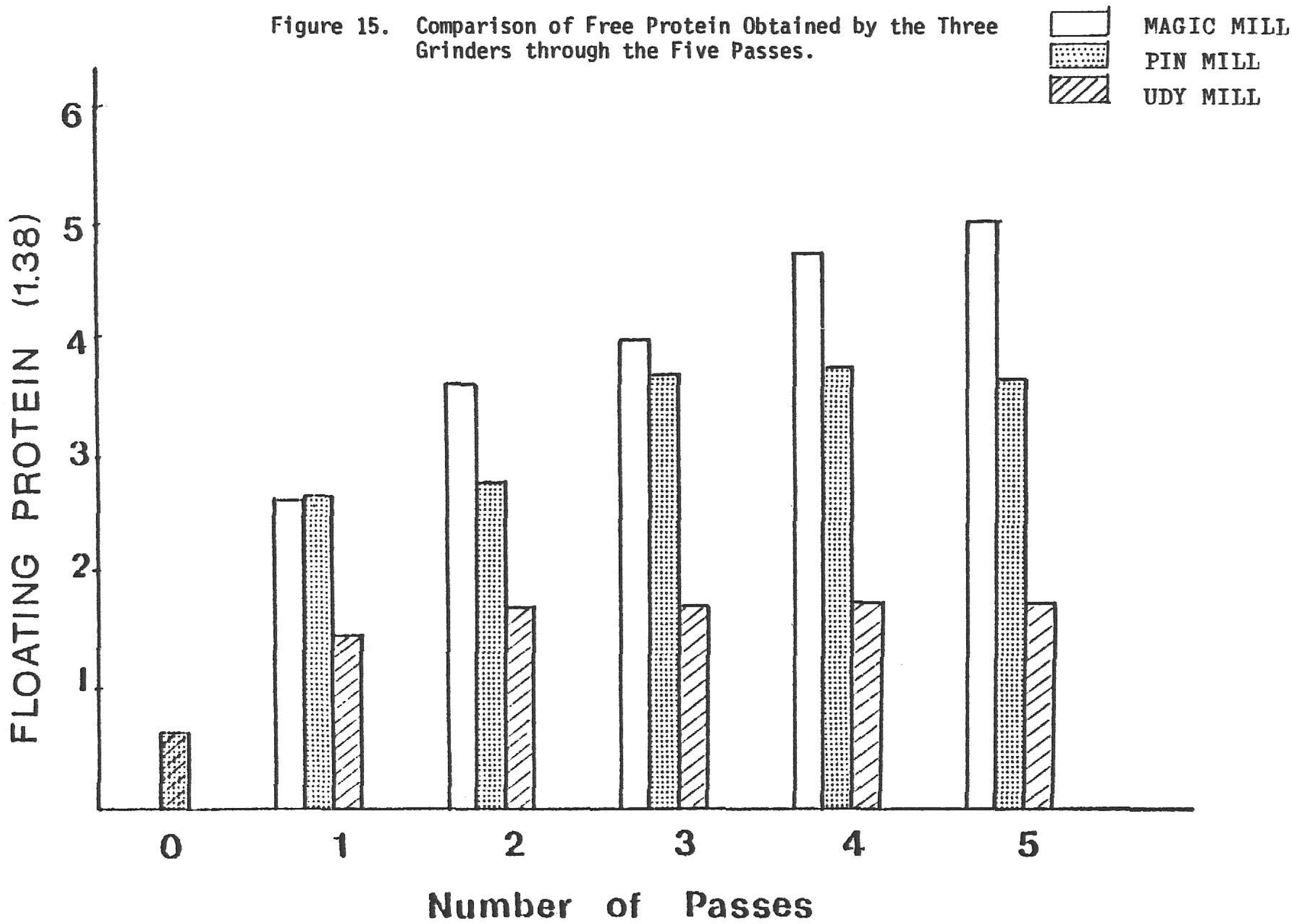


Figure 14. Relationship Between Number of Passes and Free Protein of Three Different Grinders.

Figure 15. Comparison of Free Protein Obtained by the Three Grinders through the Five Passes.



The Ūdy mill was not efficient at all after the first pass.

Figure (16) compares the amounts of starch freed by the three grinders. The results were similar to the protein pattern.

The magic mill freed higher amounts of starch through the five passes followed by the pin mill and then by the poor yields of the udy mill.

A complete comparison is shown in Figure (17) where the evaluation of their efficiency can be seen.

The best result was given by the magic mill at the fifth pass, 6% free protein of total flour, 66% free starch of total flour and 28% fine endosperm chunks.

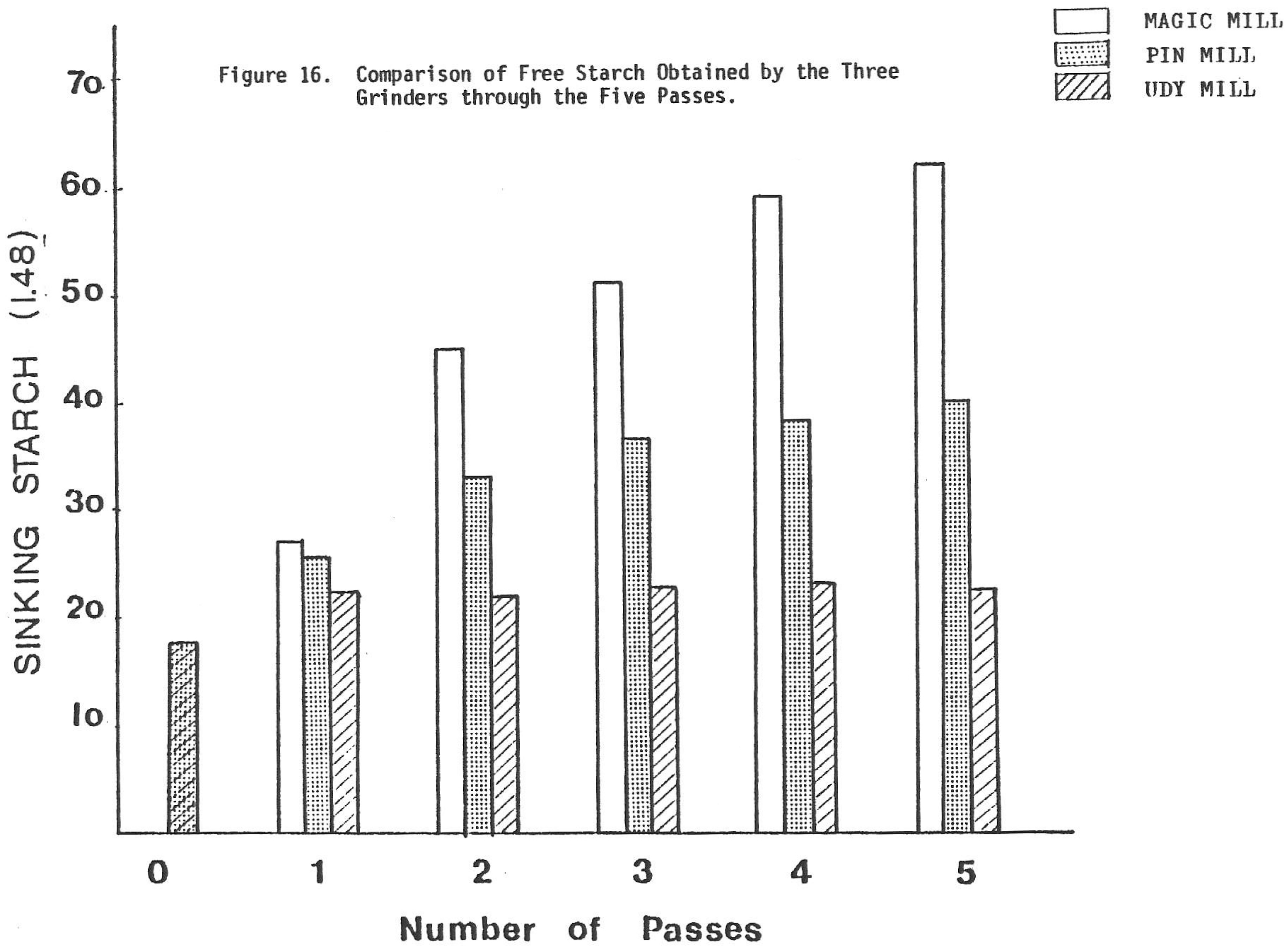
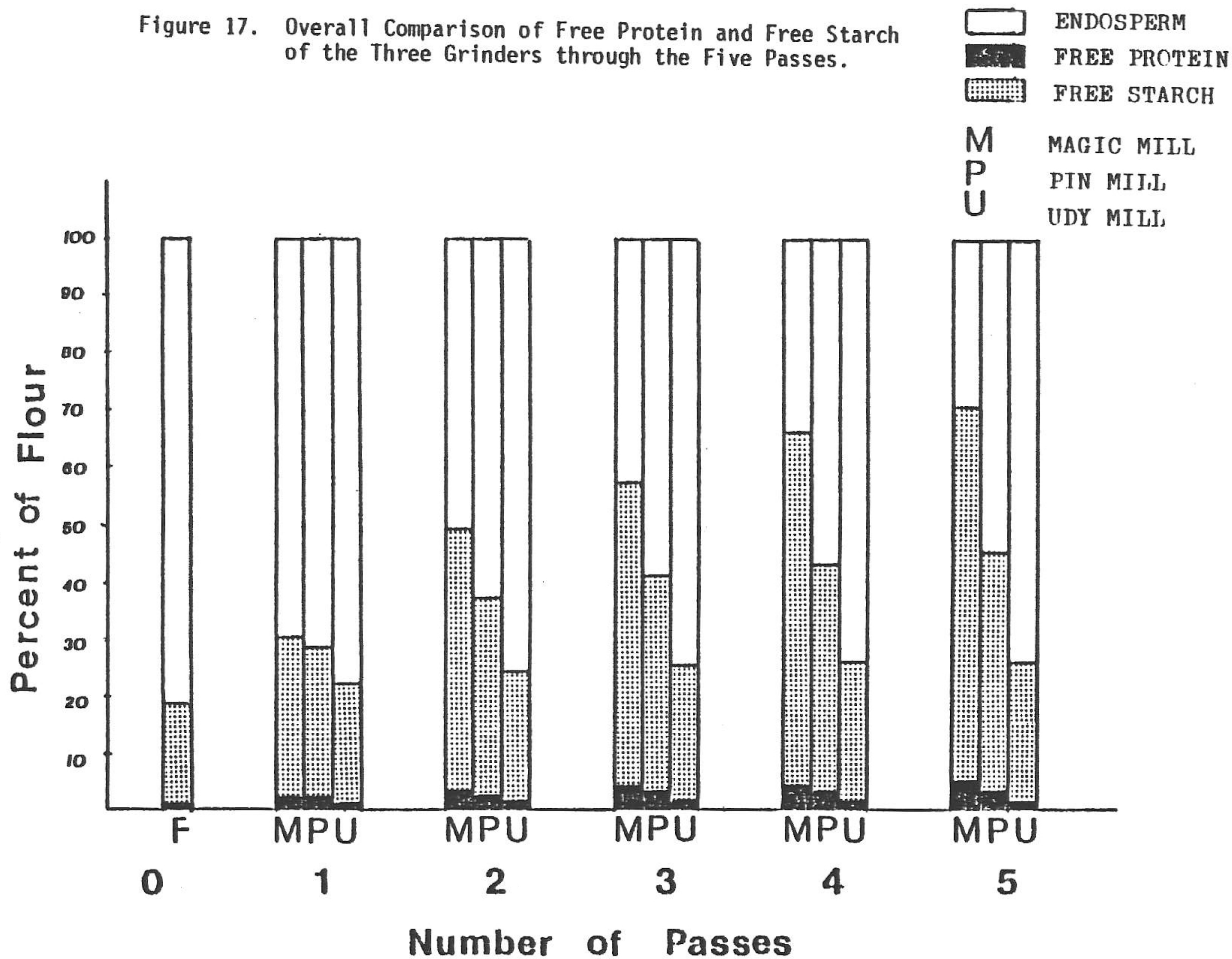


Figure 16. Comparison of Free Starch Obtained by the Three Grinders through the Five Passes.

Figure 17. Overall Comparison of Free Protein and Free Starch of the Three Grinders through the Five Passes.



An attempt was made in Figure 18 and 19 to draw curves which show the relationship between floating protein and particle size and between sinking starch and particle size. The two curves have the same pattern but more accentuated with starch since this component is the more important in quantity in flour.

Air Classification

The as milled flour gave fractions containing up to about 27.2% protein, while the lowest protein obtained was 7.50%, Figure (20).

The reground flour, before classification, was quite effective in increasing yields of high and low protein fractions, and the range of protein content was also extended, 30.20% for the highest fraction and 7.10% for the lowest fraction, Figure (21).

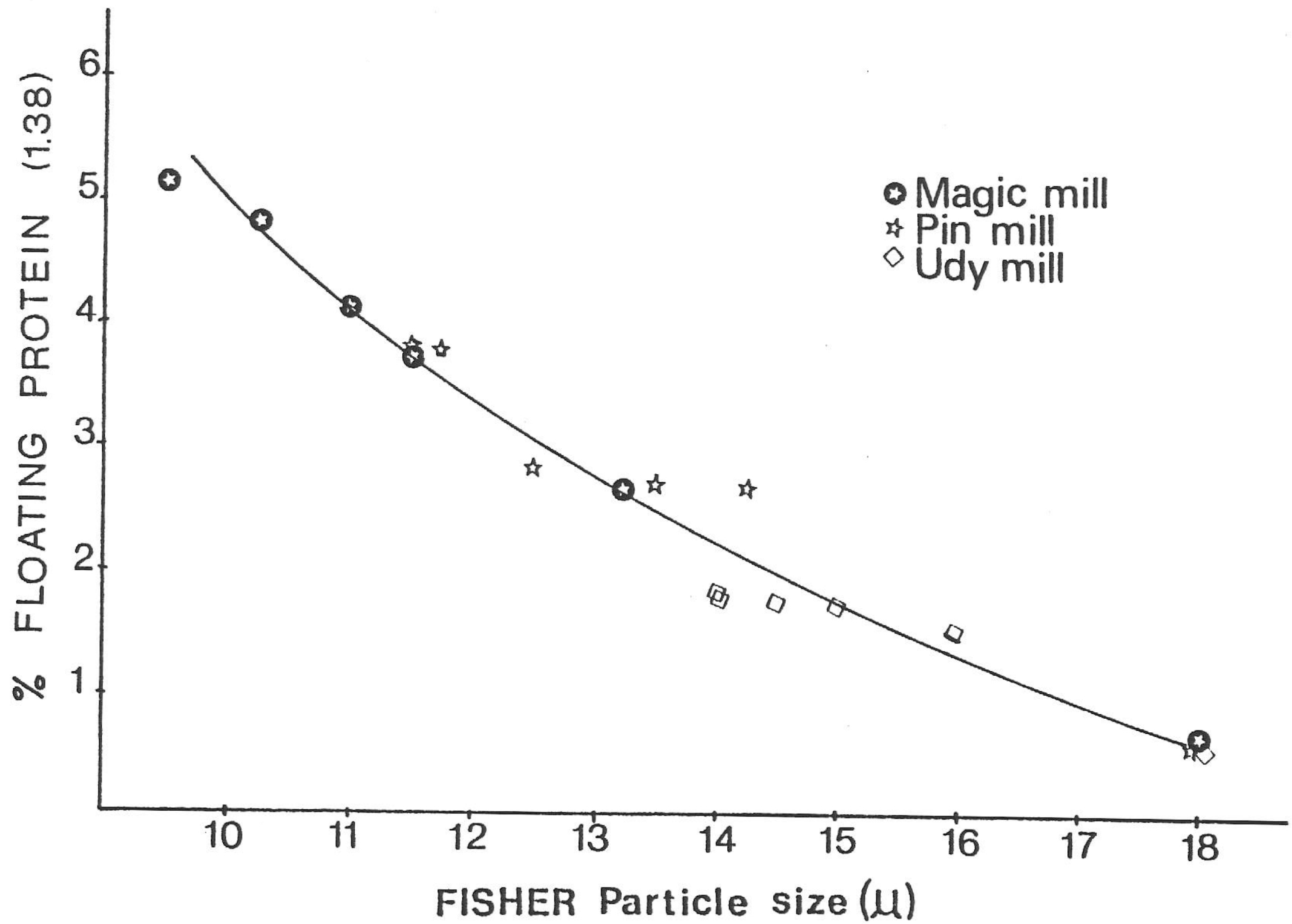
Protein Content

The histograms in Figure 22 show that the protein for all the flour samples was concentrated in the B and C fractions. The protein content of these fractions was higher than that of the parent flour. The protein content of D and E fractions was lower than that of the parent flour. The coarsest fraction (EE) had a protein content similar to that of the parent.

Particle Size

The Fisher numbers of the four fine fractions (A, B, C, D) and fraction EE are presented in the histogram form in Figure (23). The broken line denotes the Fisher number of the parent flour used for each of the six fractionations. The Fisher number was greatest for the EE fraction; in all

Figure 18. Relationship Between Free Protein and Particle Size Obtained by Three Different Grinders.



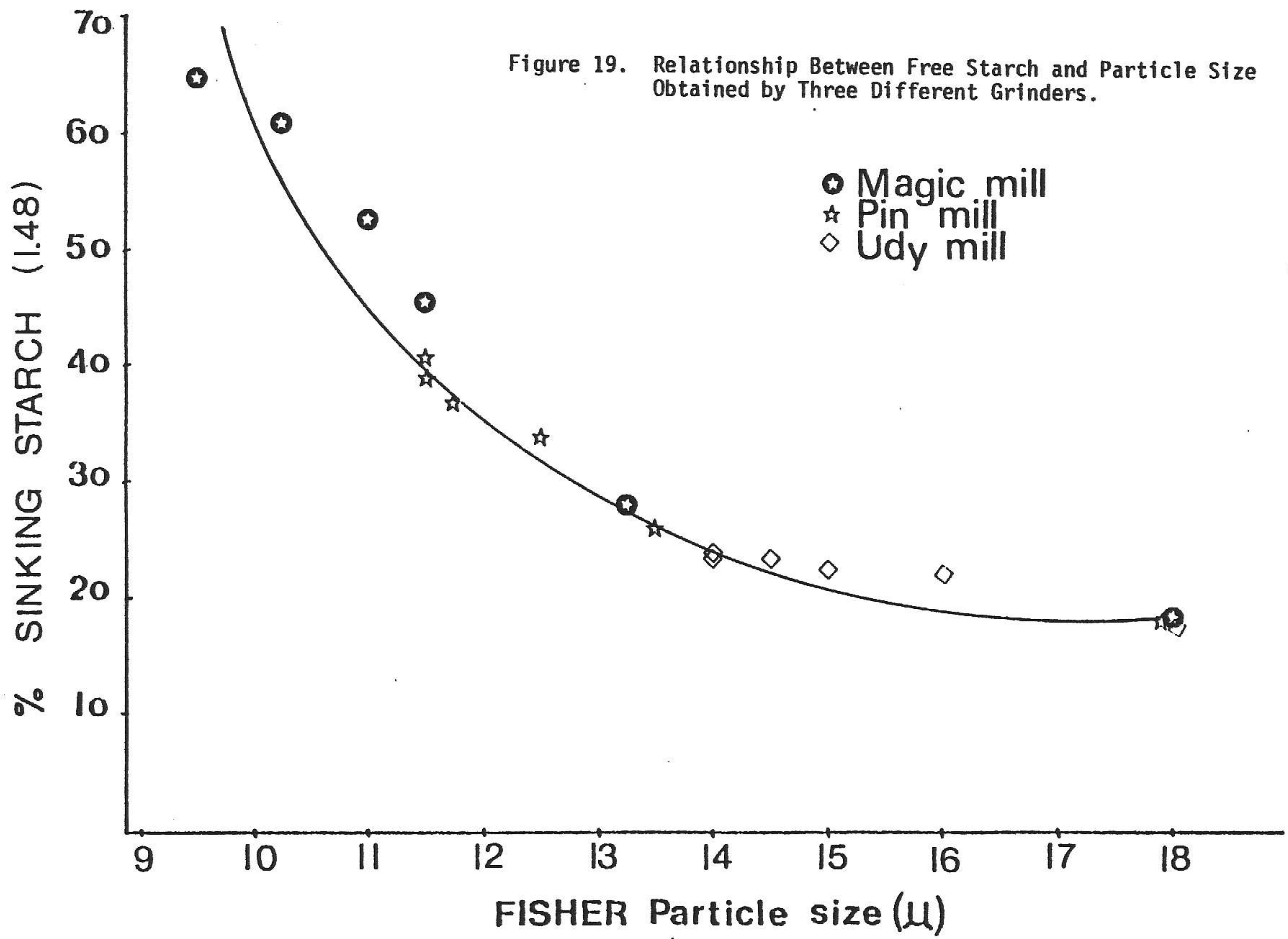


Figure 19. Relationship Between Free Starch and Particle Size Obtained by Three Different Grinders.

Figure 20. Air Classification of Unground Flour.

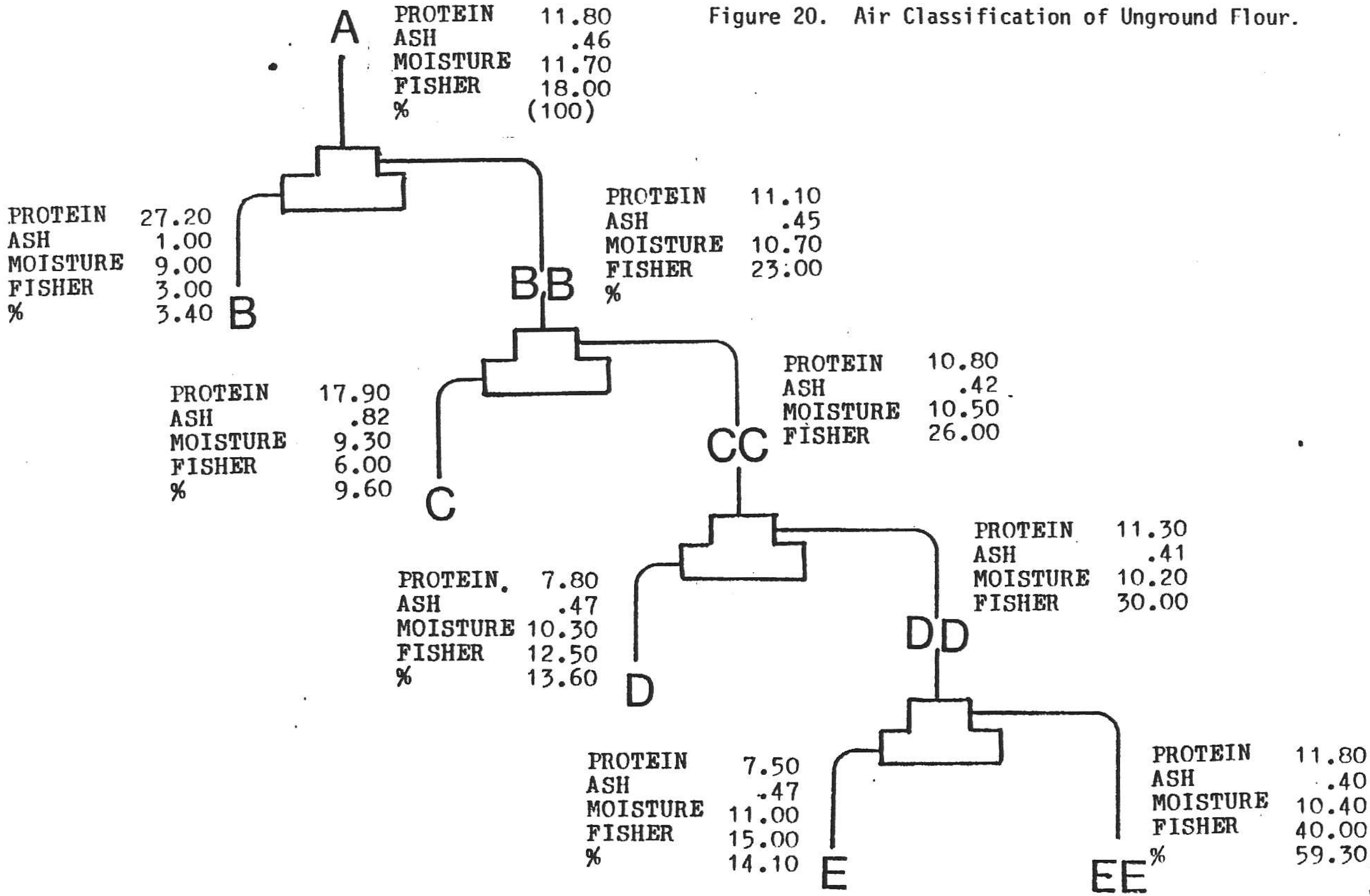


Figure 21. Air Classification of Pin Milled Flour.

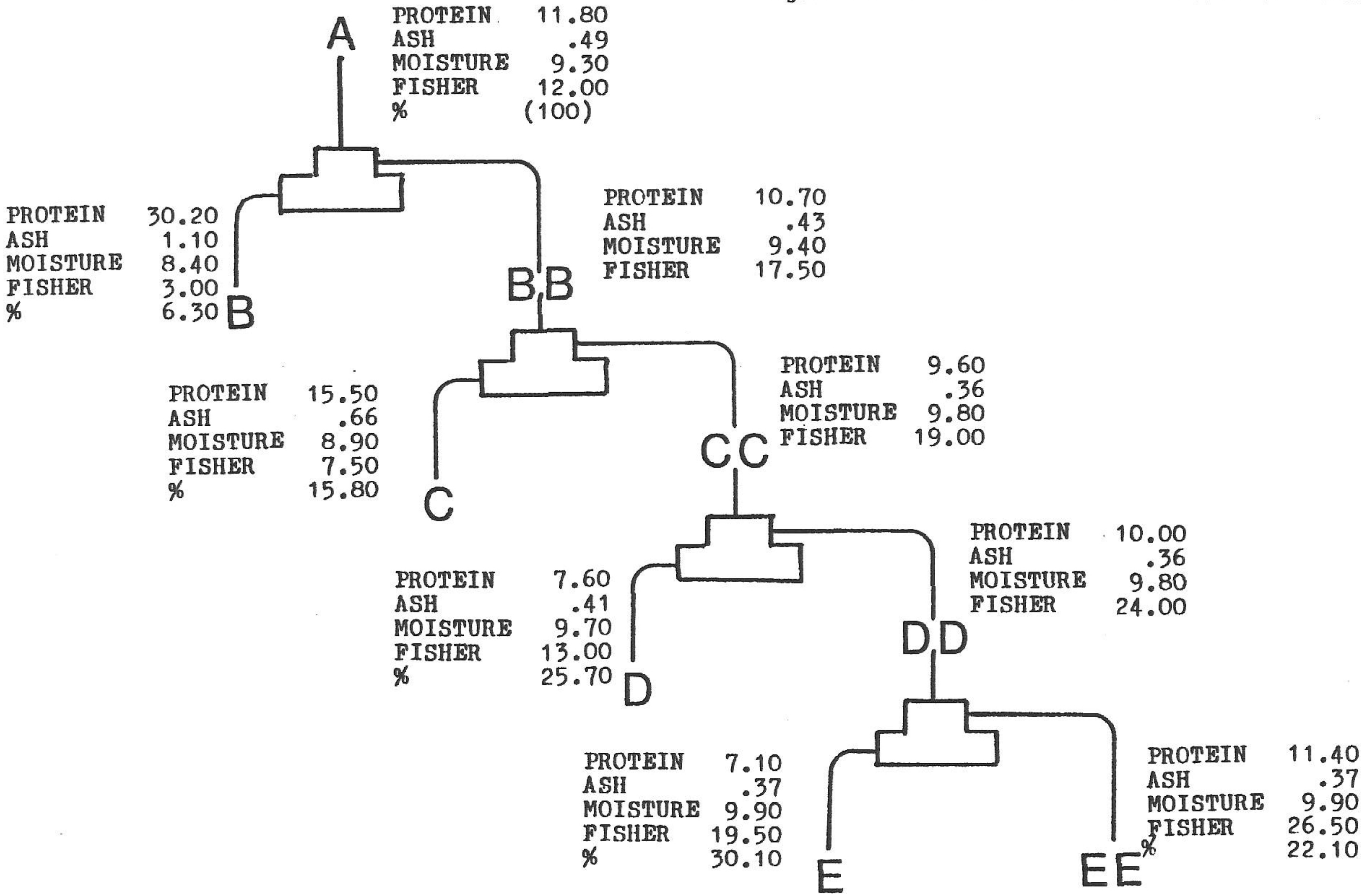
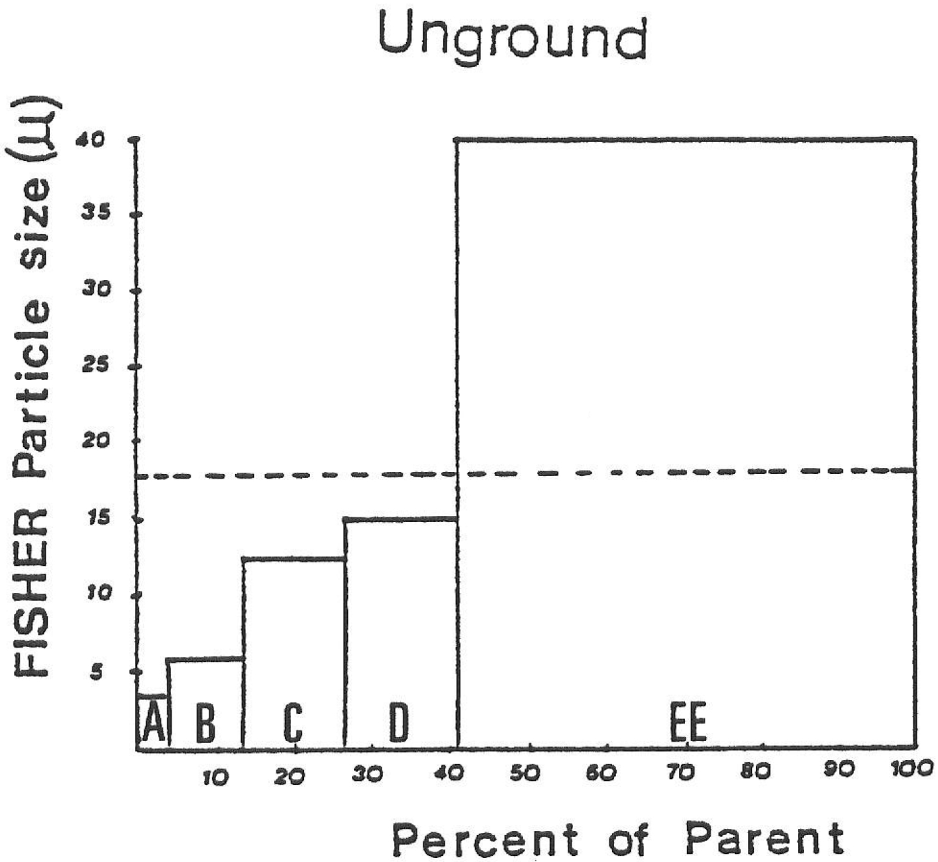


Figure 23. Air-Classified Flour Fractions



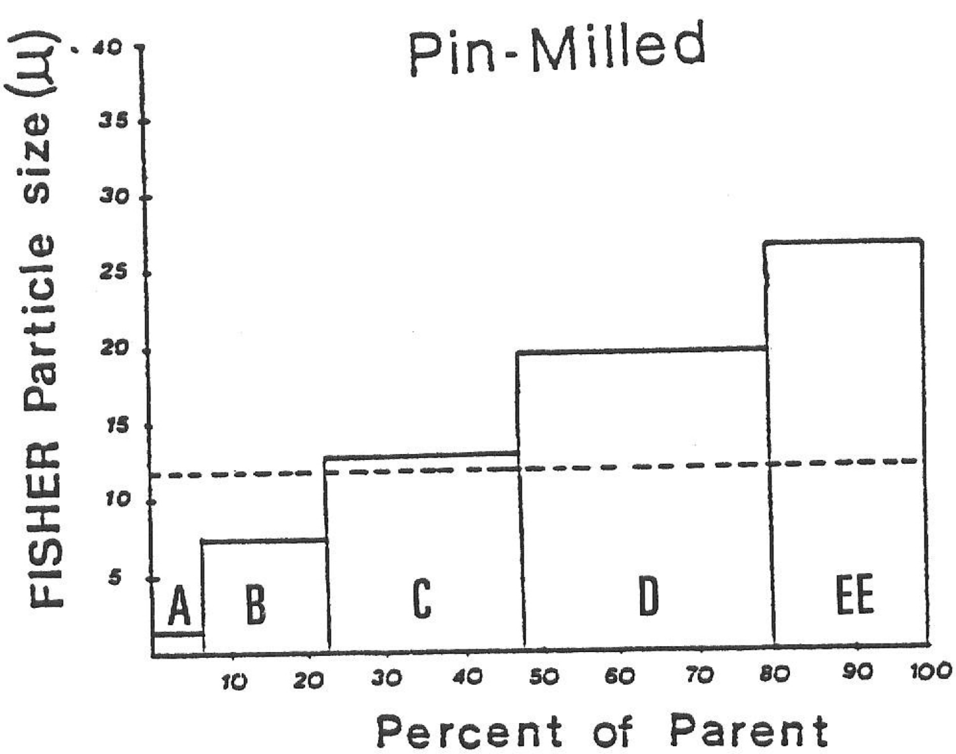
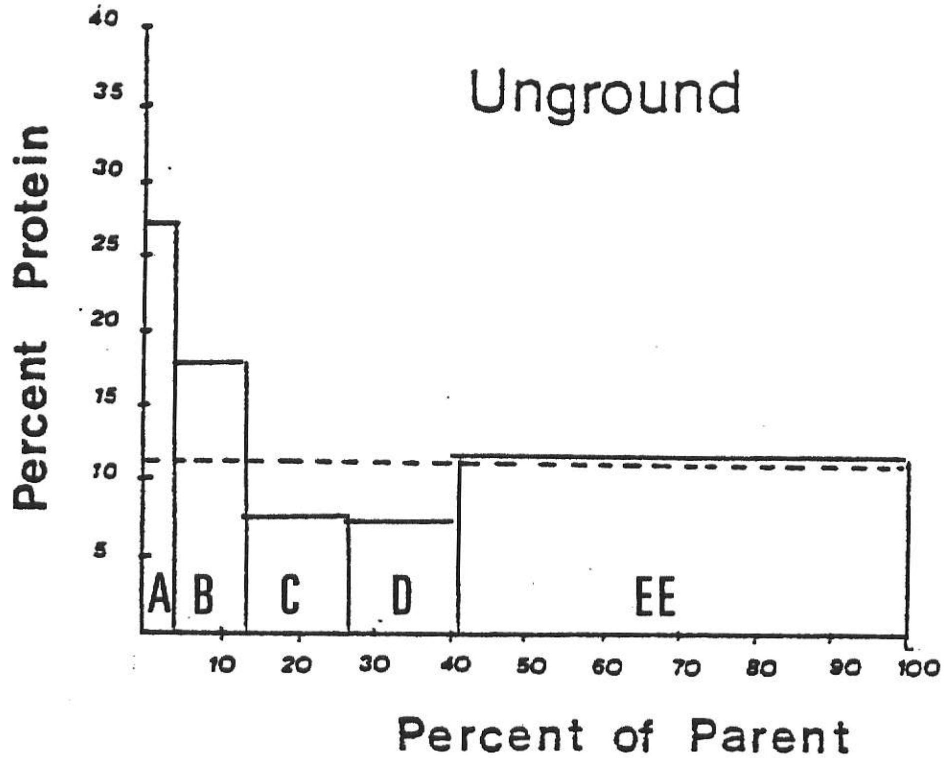
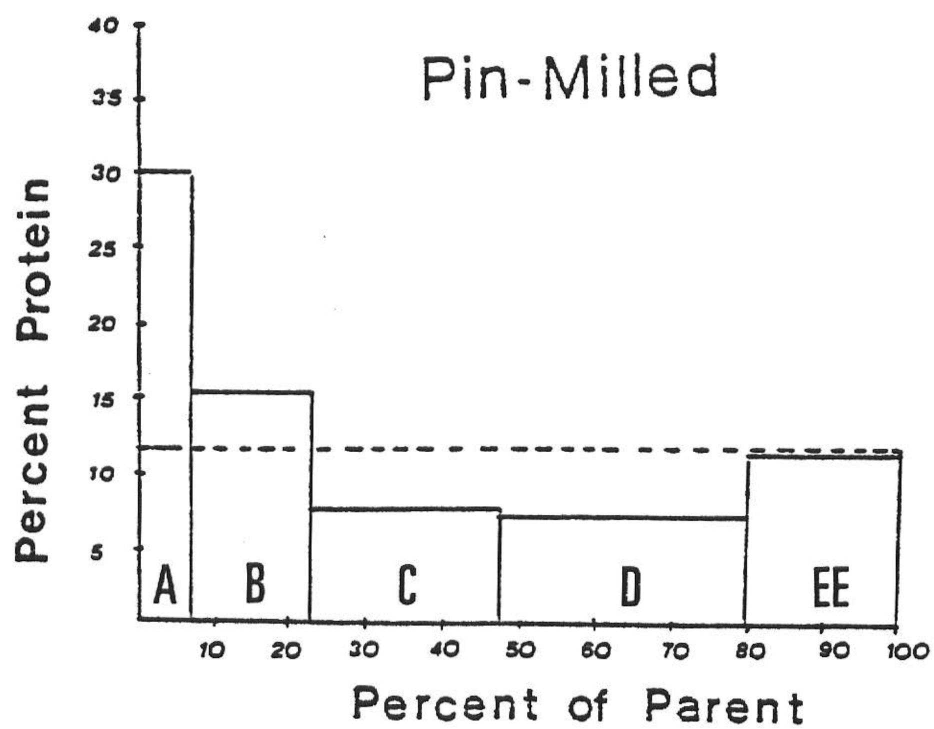


Figure 22. Air-Classified Flour Fractions





six instances it was greater than the Fisher number of the parent. The four fractionations which involved using pin milled flour had C, D and EE greater than the parent's.

High protein was associated with high ash and low protein was associated with low ash. The particle size increased with each successive air separation stage. As protein content increased from E, D, C to B fraction, the particle size decreased.

Density Separation

The effect of fine grinding on the amount of free protein is quite evident in Figure (24) and Table (5).

The high protein fraction, fraction B, was reduced in size while at the same time its protein level was increased. 76.47% of the 27.2% protein content were free protein in the unground fraction B. The same fraction pin milled gave 82.78% free protein. Fraction C was the second fraction from which an important amount of free protein was produced; 53.4% for the unground flour and 39.5% in the pin milled flour. From these figures it can be concluded that by fine grinding there was a tendancy to concentrate all the free protein in one fraction, the B fraction. In the unground flour the free protein was concentrated in two fractions, B and C.

The fractions BB, CC, DD and EE showed lower free protein content than the parent.

Even the protein purity is associated with high floating protein.

The purest protein was obtained in fraction B for both flours (unground and pin milled), 73% and 79.90%, respectively.

TABLE 5.

Unground

FRACTIONS	MOISTURE	PROTEIN (14% M.B)	ASH (14% M.B)	PARTICLE SIZE (MICRONS)
A	10.70	11.80	.46	18
B	9.00	27.20	1.00	3
C	9.30	17.90	.82	6
D	10.30	7.80	.47	12.50
E	11.00	7.50	.47	15
BB	10.70	11.10	.45	23
CC	10.50	10.80	.42	26
DD	10.20	11.30	.41	30
EE	10.40	11.80	.40	40

Pin Milled

FRACTIONS	MOISTURE	PROTEIN (14% M.B)	ASH (14% M.B)	PARTICLE SIZE (MICRONS)
A	9.30	11.80	.49	12
B	8.40	30.20	1.10	3
C	8.90	15.50	.66	7.50
D	9.70	7.60	.41	13
E	9.90	7.10	.37	19.50
BB	9.40	10.70	.43	17.50
CC	9.80	9.60	.37	19
DD	9.80	10.00	.36	24
EE	9.90	11.40	.37	26.50

Figure 24. Amount and Purity of Free Protein of Unground Air Classified Fractions.

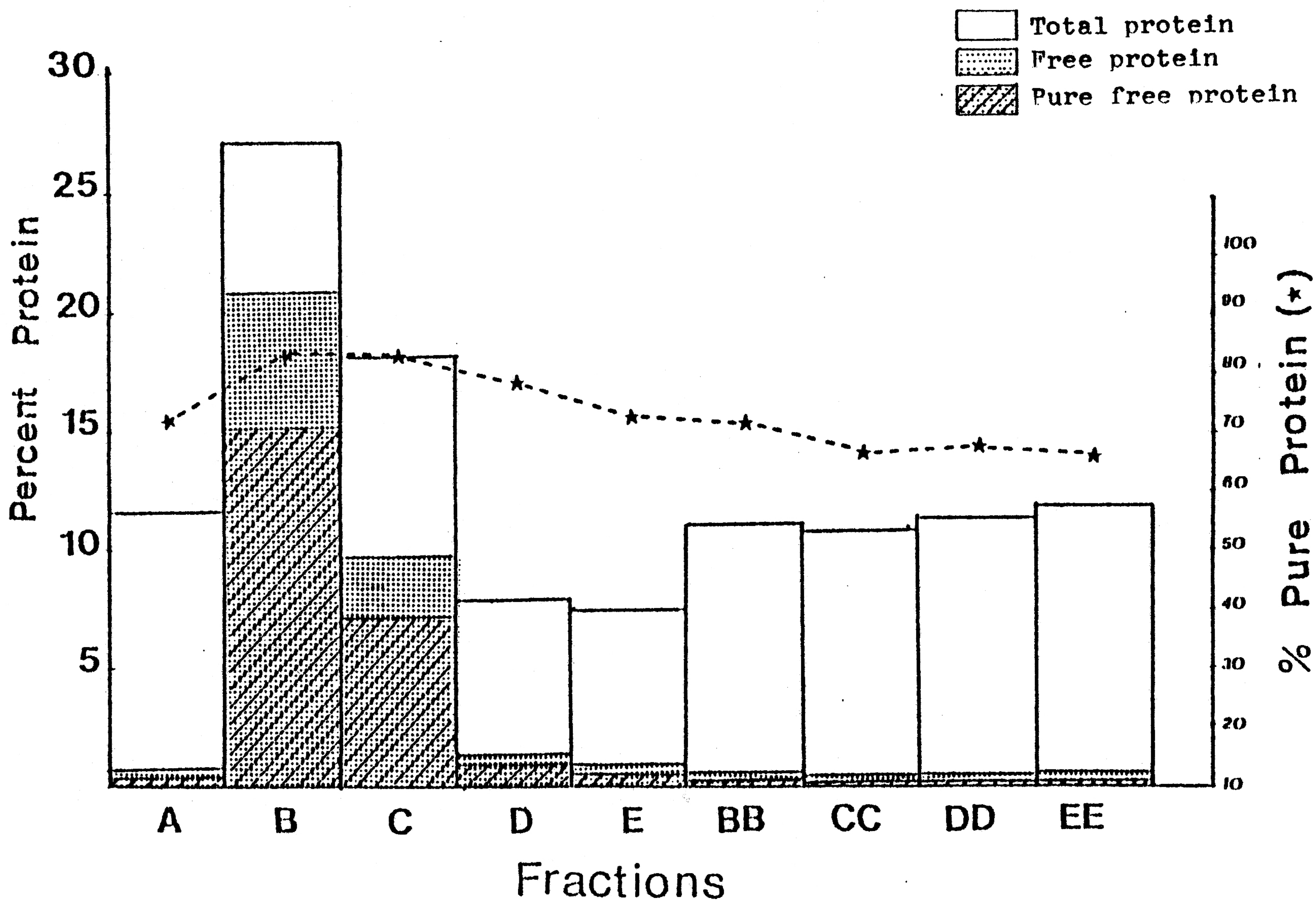
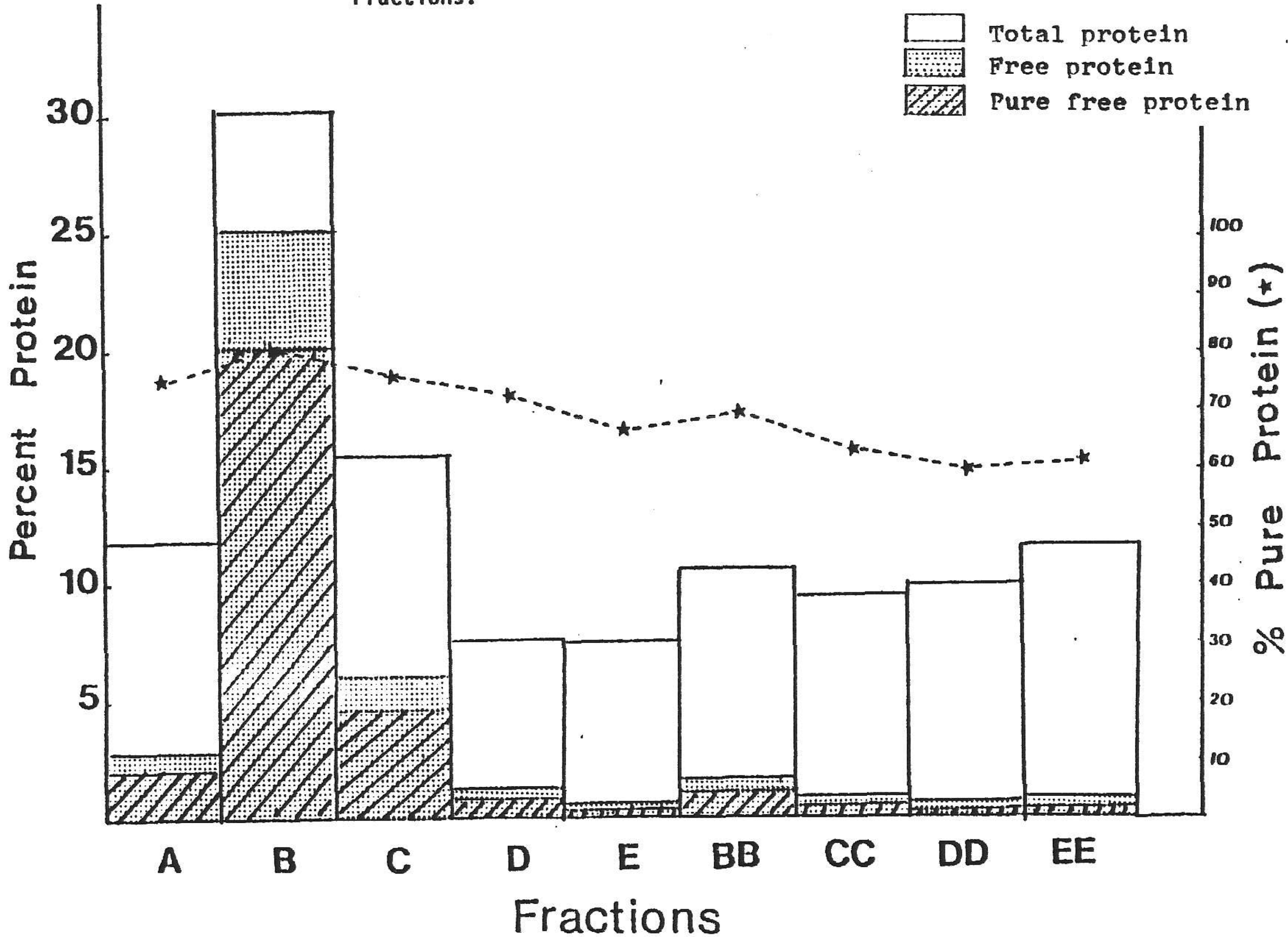


Figure 25. Amount and Purity of Free Protein of Pin Milled Air Classified Fractions.



Free Protein vs. Particle Size

The relation of particle size to free protein distribution is shown in Figure (26). As is well known, protein is highest in the smallest size range, 1 to 16 microns, because more thin, light-density protein fragments are included. The protein content decreases rapidly from 1 to 20 microns because a greater proportion of starch granules is included in the highest micron size. In fact, the fraction from about 16 to 35 microns is considerably below the protein content of the original flour because this range includes an increased concentration of free starch granules.

The comparative histograms of free protein, free starch and overall grinding, Figures (27, 28, 29), show the differences between unground flour and pin milled flour fractions. The B and C fractions were the more important according to the amount of protein freed (Figure 27). As it was said earlier, the concentration of free protein was in fraction B and C with the unground flour, but with pin milled flour, the free protein had a tendancy to concentrate in B fraction.

The coarser particles, mainly starch granules, were concentrated in D and E fractions with the pin milled flour (Figure 28). All the fractions with unground flour had between 15 and 30% free starch. The pin milled flour considerably increased the amount of free starch in the coarser fractions (between 35 and 72%), more than doubling it in some instances. The fractions D and E had the highest amount of free starch and the finest, fraction B, had the lowest amount of free starch, even lower than the parent.

TABLE 6.

Unground

FRACTIONS	% PROTEIN	% FLOAT (1.38)	% PURITY	% TOTAL FREE PROTEIN
A	11.80	.68	62.80	3.73
B	27.20	20.80	73.00	55.80
C	17.90	9.56	72.30	38.66
D	7.80	1.40	67.70	12.05
E	7.50	.80	62.80	6.67
BB	11.10	.60	61.90	3.24
CC	10.80	.30	55.90	1.48
DD	11.30	.20	57.70	1.06
EE	11.80	.20	56.00	1.02

Pin Milled

FRACTIONS	% PROTEIN	% FLOAT (1.38)	% PURITY	% TOTAL FREE PROTEIN
A	11.80	2.70	74.80	17.12
B	30.20	25.00	79.90	66.16
C	15.50	6.10	75.20	29.55
D	7.60	1.40	72.10	13.28
E	7.10	.60	66.40	5.63
BB	10.70	1.62	69.80	10.65
CC	9.60	1.00	63.80	6.66
DD	10.00	.74	59.80	4.40
EE	11.40	.78	61.80	4.21

TABLE 7.

Unground

FRACTIONS	% PROTEIN	% SINK (1.48)	% PURITY	% TOTAL FREE STARCH
A	11.80	18.10	95.03	17.20
B	27.20	15.34	92.83	14.24
C	17.90	31.00	96.52	29.92
D	7.80	28.50	97.61	27.82
E	7.50	25.50	97.49	24.86
BB	11.10	19.10	96.34	18.40
CC	10.80	20.80	96.54	20.08
DD	11.30	16.80	95.95	16.12
EE	11.80	11.10	94.77	10.52

Pin Milled

FRACTIONS	% PROTEIN	% SINK (1.48)	% PURITY	% TOTAL FREE STARCH
A	11.80	26.40	95.68	25.26
B	30.20	12.00	94.83	11.38
C	15.50	45.22	95.71	43.28
D	7.60	70.40	96.70	68.08
E	7.10	71.12	96.79	68.84
BB	10.70	51.62	96.40	49.76
CC	9.60	49.62	96.29	47.78
DD	10.00	43.02	96.00	41.30
EE	11.40	36.14	94.80	34.26

Figure 26. Relationship Between Free Protein and Particle Size of Unground Flour and Air Classified Flour.

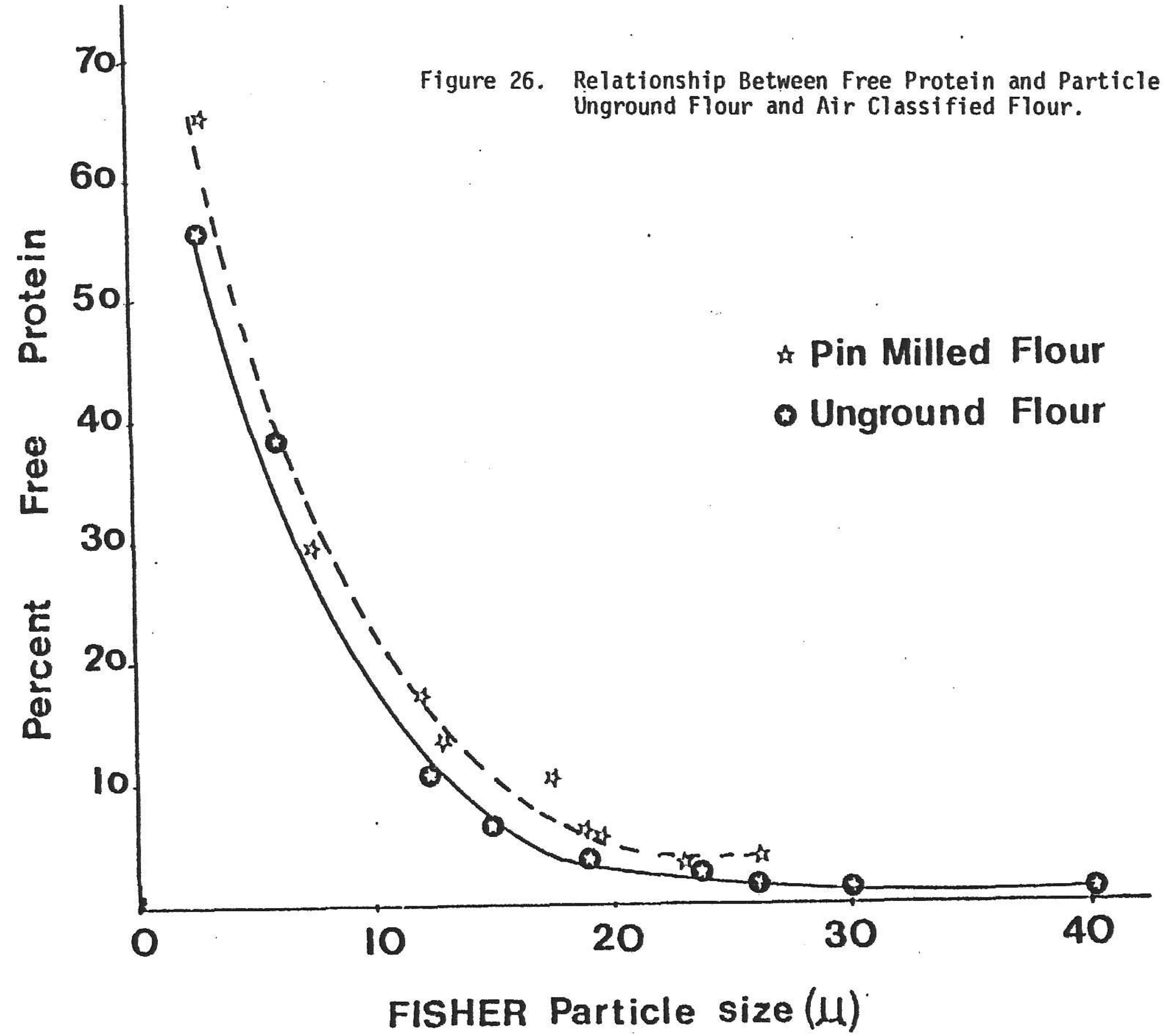


Figure 27. Comparison of Free Protein Obtained by Unground and Pin Milled Air Classified Fractions.

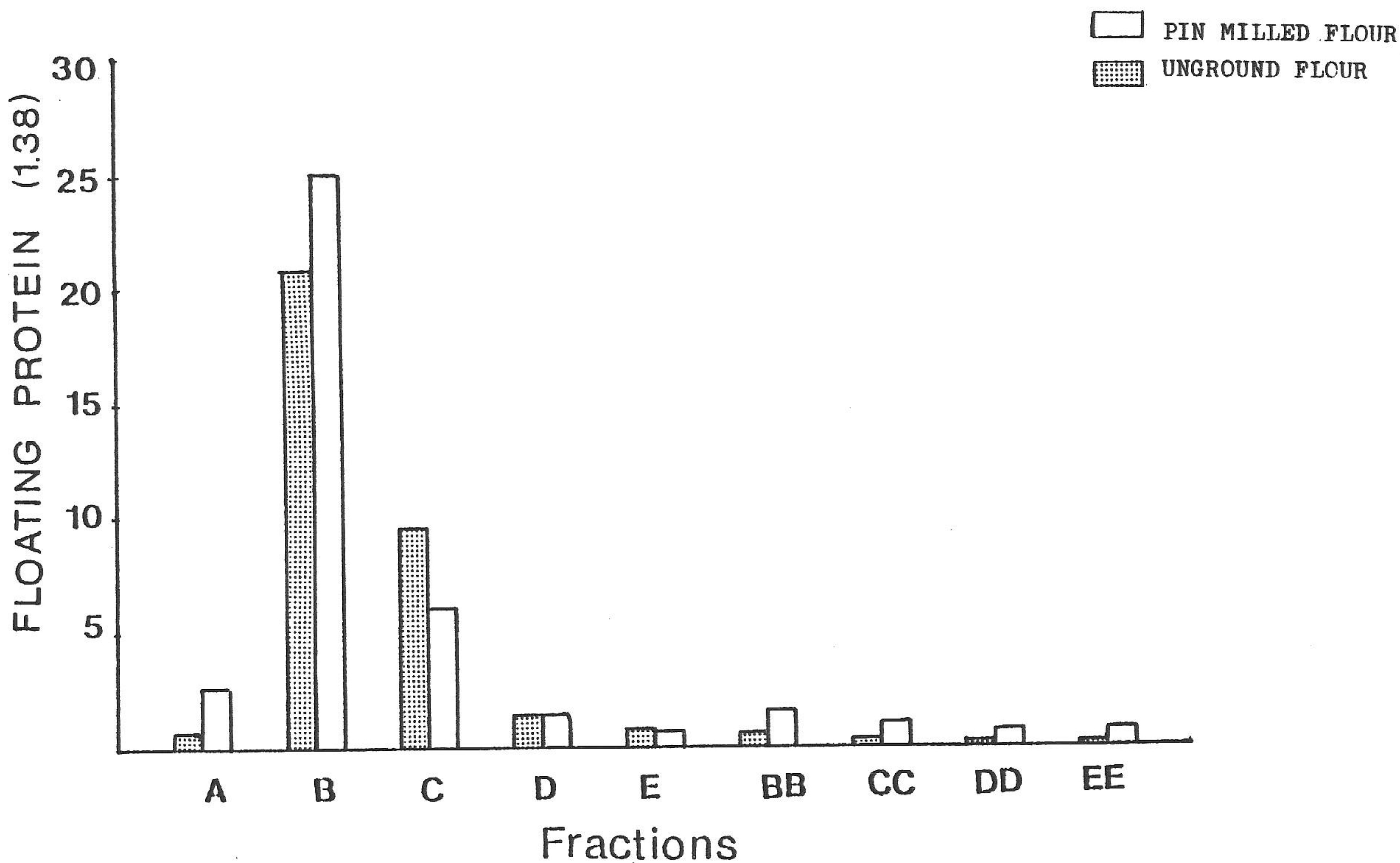


Figure 28. Comparison of Free Starch Obtained by Unground and Pin Milled Air Classified Fractions.

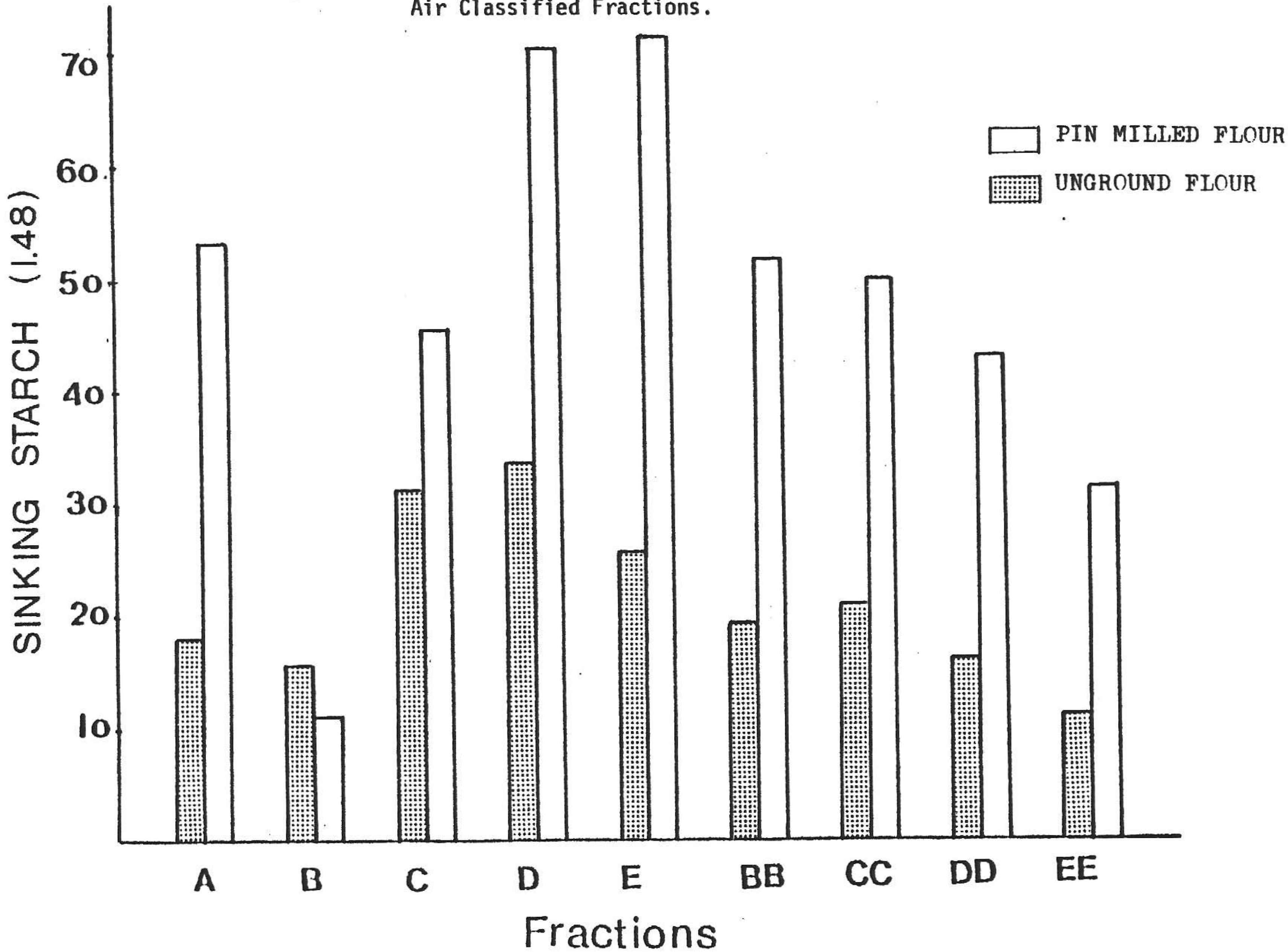


Figure (29) shows the effect of air classification and fine grinding on the different fractions, showing the amount of free starch and free protein in the fraction itself.

Statistical results

The results of the data collected from both trial 1 and trial 2 of different types of grinding at levels of 1.38 density and 1.48 density of this study are presented in this section.

For better clarity and understanding of statistical test, this section was subdivided into two sections.

Section 1 reports the results of null hypothesis regarding the no significant mean trial values of different types of grinding at a level of 1.38 density (Table 8).

Section 2 presents the results of null hypothesis regarding the no significant mean trial values of different types of grinding at a level of 1.48 density (Table 9).

An examination of t values in tables (8, 9) indicates that none of the mean trial value differences of both the different types of grinding and the total samples appeared to be significantly.

The high correlation between trial 1 and trial 2 for each type of grinding and total samples, as shown in tables (8, 9), provide the evidence for the test of no significant mean trial value differences.

The correlation of trial 1 and trial 2 of all samples for both densities is at .001 label of significance.

Figure 29. Overall Comparison of Free Protein and Free Starch of Unground and Pin Milled Air Classified Fractions.

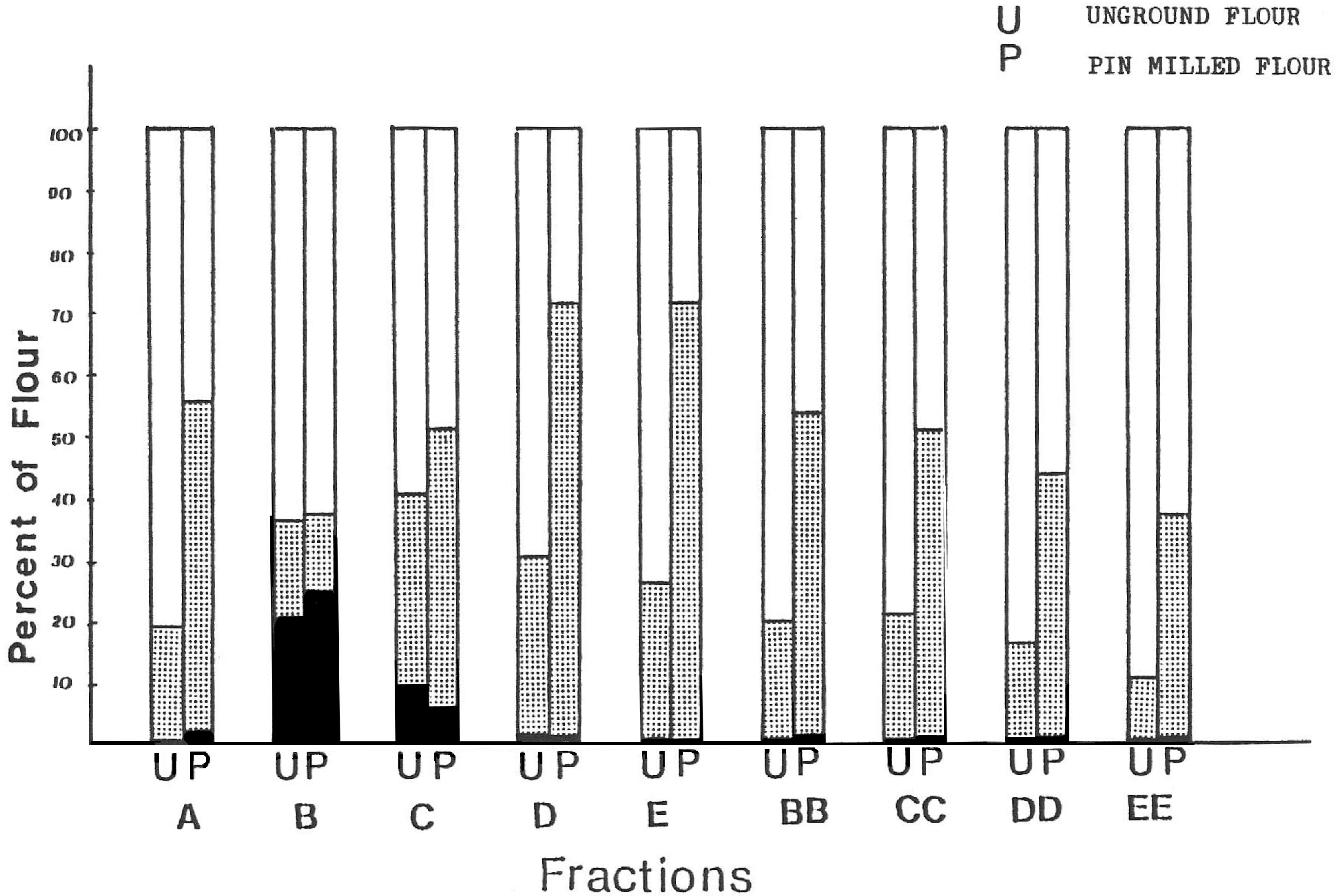


TABLE 8.

t-test between two mean trial values of different types of grinding at level of 1.38 density.

Types of grinding		Trial 1	Trial 2	t	Significance (P)	Correlation
Unground Air classified	M	3.694	3.980	-.99	N.S.	.999***
	SD	6.630	7.429			
	N	9	9			
Pin milled Air classified	M	4.403	4.471	-1.47	N.S.	1.000***
	SD	7.841	7.961			
	N	9	9			
Magic mill	M	4.124	4.036	1.31	N.S.	.989***
	SD	.997	.967			
	N	5	5			
Pin mill	M	3.304	3.384	-1.93	N.S.	.988**
	SD	.530	.564			
	N	5	5			
Udy mill	M	1.696	1.710	-.26	N.S.	.888*
	SD	.173	.066			
	N	5	5			
Total samples	M	3.591	3.688	1.20	N.S.	.998***
	SD	5.288	5.536			
	N	33	33			

Note:

*** $P \leq .001$
** $P \leq .01$
* $P \leq .05$

M: mean
SD: standard deviation
N: number of cases
N.S: no significance

TABLE 9.

t-test between two mean trial values of different types of grinding at level of 1.48 density.

Types of grinding		Trial 1	Trial 2	t	Significance (P)	Correlation
Unground Air classified	M	20.747	20.653	.27	N.S.	.987**
	SD	6.523	6.456			
	N	9	9			
Pin milled Air classified	M	45.029	45.090	-.18	N.S.	.999***
	SD	19.232	19.922			
	N	9	9			
Magic mill	M	50.552	50.728	-.36	N.S.	.999***
	SD	14.147	15.123			
	N	5	5			
Pin mill	M	35.356	35.762	-1.08	N.S.	.992***
	SD	5.572	6.026			
	N	5	5			
Udy mill	M	23.030	23.370	-1.53	N.S.	.785*
	SD	.701	.795			
	N	5	5			
Total samples	M	34.444	34.575	-.82	N.S.	.999***
	SD	16.575	16.714			
	N	33	33			

Note;

*** $P \leq .001$ M: mean

** $P \leq .01$ SD: standard deviation

* $P \leq .05$ N: number of cases

N.S: no significance

CONCLUSIONS

The results of this study provide guidelines for a technique that is not new but that has been employed only to a limited extent for fractionation of whole flours. It appeared that non-aqueous separations work on free starch and free protein; to achieve good separations, the technique requires only that the material be finely ground.

Since the solvent does not interact with flour components, the separation process should not affect their properties. Because of this lack of interaction, however, aggregated material is not broken down, and separation into homogeneous fractions requires prior disruption of aggregates.

The more the protein matrix is broken up by means of fine grinding without damage to the starch granules, then the more free protein and free starch will be available. There is a limit, however, to the amount of breaking up which can be done without causing starch damage.

Complete solvent removal is important in the preparation of products which may be used for human or animal food. The ease of removal is a prime economic factor, especially with respect to energy conservation. The development of an efficient process with non-toxic solvents to separate these small particles from the rest of the flour could have wide uses in food processing.

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Appendix

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION A

50 Grams

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	.34
Moisture %	11.70
Protein % (N x 5.7)	62.80
Grams Protein in Fraction	.22
% of Total Protein	3.73

BOTTOM FRACTION

Amount recovered grams	49.37
Moisture %	11.40
Protein % (N x 5.7)	11.10
Grams Protein in Fraction	5.48
% of Total Protein	94.48

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.22	3.73
In Bottom Fraction	5.48	94.48
Total	5.70	98.21

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

FRACTION

50 Grams

TOP FRACTION

Amount recovered grams	40.60
Moisture %	11.60
Protein % (N x 5.7)	13.10
Grams Protein in Fraction	5.32
% of Total Protein	90.17

BOTTOM FRACTION

Amount recovered grams	9.05
Moisture %	11.90
Protein % (N x 5.7)	5.00
Grams Protein in Fraction	.45
% of Total Protein	7.63

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.32	90.17
In Bottom Fraction	.45	7.63
Total	5.77	97.80

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION B

50 GRAMS

Original Flour

Moisture	9.00
Protein	27.20
Ash	1.00
Fisher Size	3

TOP FRACTION

Amount recovered grams	10.40
Moisture %	11.20
Protein % (N x 5.7)	73.00
Grams Protein in Fraction	7.59
% of Total Protein	55.80

BOTTOM FRACTION

Amount recovered grams	38.80
Moisture %	11.40
Protein % (N x 5.7)	15.40
Grams Protein in Fraction	5.97
% of Total Protein	43.90

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	7.59	55.80
In Bottom Fraction	5.97	43.90
Total	13.56	99.70

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	31.51
Moisture %	11.90
Protein % (N x 5.7)	40.20
Grams Protein in Fraction	12.67
% of Total Protein	93.16

BOTTOM FRACTION

Amount recovered grams	7.67
Moisture %	11.30
Protein % (N x 5.7)	7.20
Grams Protein in Fraction	.55
% of Total Protein	4.04

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	12.67	93.16
In Bottom Fraction	.55	4.04
Total	13.22	97.20

Note: Protein + Ash corrected on 14% M.B.

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION C

50 GRAMS

Original Flour

Moisture	9.30
Protein	17.90
Ash	.82
Fisher Size	6.00

TOP FRACTION

Amount recovered grams	4.78
Moisture %	11.30
Protein % (N x 5.7)	72.30
Grams Protein in Fraction	3.46
% of Total Protein	38.66

BOTTOM FRACTION

Amount recovered grams	44.86
Moisture %	11.60
Protein % (N x 5.7)	12.10
Grams Protein in Fraction	5.43
% of Total Protein	60.67

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	3.46	38.66
In Bottom Fraction	5.43	60.67
Total	8.89	99.33

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	33.05
Moisture %	11.20
Protein % (N x 5.7)	24.60
Grams Protein in Fraction	8.13
% of Total Protein	90.84

BOTTOM FRACTION

Amount recovered grams	15.55
Moisture %	11.40
Protein % (N x 5.7)	3.80
Grams Protein in Fraction	.59
% of Total Protein	6.59

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	8.13	90.84
In Bottom Fraction	.59	6.59
Total	8.72	97.43

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION D

50 GRAMS

Original Flour

Moisture	10.30
Protein	7.80
Ash	.47
Fisher Size	12.50

TOP FRACTION

Amount recovered grams	.70
Moisture %	11.90
Protein % (N x 5.7)	67.70
Grams Protein in Fraction	.47
% of Total Protein	12.05

BOTTOM FRACTION

Amount recovered grams	48.80
Moisture %	11.80
Protein % (N x 5.7)	6.80
Grams Protein in Fraction	3.32
% of Total Protein	85.13

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.47	12.05
In Bottom Fraction	3.32	85.13
Total	3.79	97.18

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	34.10
Moisture %	11.00
Protein % (N x 5.7)	10.40
Grams Protein in Fraction	3.55
% of Total Protein	91.02

BOTTOM FRACTION

Amount recovered grams	14.25
Moisture %	11.80
Protein % (N x 5.7)	2.40
Grams Protein in Fraction	.34
% of Total Protein	8.72

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	3.55	91.02
In Bottom Fraction	.34	8.72
Total	3.89	99.74

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION E

50 GRAMS

Original Flour

Moisture	11.00
Protein	7.50
Ash	.47
Fisher Size	15.00

TOP FRACTION

Amount recovered grams	.40
Moisture %	11.70
Protein % (N x 5.7)	62.80
Grams Protein in Fraction	.25
% of Total Protein	6.67

BOTTOM FRACTION

Amount recovered grams	49.20
Moisture %	10.60
Protein % (N x 5.7)	6.90
Grams Protein in Fraction	3.39
% of Total Protein	90.40

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.25	6.67
In Bottom Fraction	3.39	90.40
Total	3.64	97.07

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	36.87
Moisture %	10.90
Protein % (N x 5.7)	9.20
Grams Protein in Fraction	3.39
% of Total Protein	90.40

BOTTOM FRACTION

Amount recovered grams	12.75
Moisture %	11.80
Protein % (N x 5.7)	2.50
Grams Protein in Fraction	.32
% of Total Protein	8.53

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	3.39	90.40
In Bottom Fraction	.32	8.53
Total	3.71	98.93

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION BB

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.10
Ash	.45
Fisher Size	23.00

TOP FRACTION

Amount recovered grams	.30
Moisture %	11.20
Protein % (N x 5.7)	61.90
Grams Protein in Fraction	.18
% of Total Protein	3.24

BOTTOM FRACTION

Amount recovered grams	49.31
Moisture %	10.90
Protein % (N x 5.7)	10.70
Grams Protein in Fraction	5.27
% of Total Protein	95.07

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.18	3.24
In Bottom Fraction	5.27	95.07
Total	5.45	98.31

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	40.30
Moisture %	11.10
Protein % (N x 5.7)	12.70
Grams Protein in Fraction	5.11
% of Total Protein	92.07

BOTTOM FRACTION

Amount recovered grams	9.55
Moisture %	11.00
Protein % (N x 5.7)	3.70
Grams Protein in Fraction	.35
% of Total Protein	6.31

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.11	92.07
In Bottom Fraction	.37	6.31
Total	5.48	98.38

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION CC

50 GRAMS

Original Flour

Moisture	10.50
Protein	10.80
Ash	.42
Fisher Size	26.00

TOP FRACTION

Amount recovered grams	.15
Moisture %	11.50
Protein % (N x 5.7)	55.90
Grams Protein in Fraction	.08
% of Total Protein	1.48

BOTTOM FRACTION

Amount recovered grams	49.60
Moisture %	11.90
Protein % (N x 5.7)	10.30
Grams Protein in Fraction	5.09
% of Total Protein	94.26

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.08	1.48
In Bottom Fraction	5.09	94.26
Total	5.17	95.74

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	39.30
Moisture %	11.40
Protein % (N x 5.7)	12.70
Grams Protein in Fraction	4.99
% of Total Protein	92.41

BOTTOM FRACTION

Amount recovered grams	10.40
Moisture %	11.70
Protein % (N x 5.7)	3.50
Grams Protein in Fraction	.36
% of Total Protein	6.67

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.99	93.41
In Bottom Fraction	.36	6.67
Total	5.35	99.08

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION DD

50 GRAMS

Original Flour

Moisture	10.20
Protein	11.30
Ash	.41
Fisher Size	30.00

TOP FRACTION

Amount recovered grams	.10
Moisture %	11.50
Protein % (N x 5.7)	57.70
Grams Protein in Fraction	.06
% of Total Protein	1.06

BOTTOM FRACTION

Amount recovered grams	49.80
Moisture %	12.10
Protein % (N x 5.7)	10.70
Grams Protein in Fraction	5.33
% of Total Protein	94.34

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.06	1.06
In Bottom Fraction	5.33	94.34
Total	6.39	95.40

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	39.80
Moisture %	12.10
Protein % (N x 5.7)	12.60
Grams Protein in Fraction	5.24
% of Total Protein	92.74

BOTTOM FRACTION

Amount recovered grams	9.40
Moisture %	11.10
Protein % (N x 5.7)	4.10
Grams Protein in Fraction	.34
% of Total Protein	6.02

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.24	92.74
In Bottom Fraction	.34	6.02
Total	5.58	98.76

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UNGROUND FLOUR
FRACTION EE

50 GRAMS

Original Flour

Moisture	10.40
Protein	11.80
Ash	.40
Fisher Size	40.00

TOP FRACTION

Amount recovered grams	.10
Moisture %	11.20
Protein % (N x 5.7)	56.00
Grams Protein in Fraction	.06
% of Total Protein	1.02

BOTTOM FRACTION

Amount recovered grams	49.80
Moisture %	12.10
Protein % (N x 5.7)	10.70
Grams Protein in Fraction	5.78
% of Total Protein	97.97

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.06	1.02
In Bottom Fraction	5.78	97.97
Total	5.84	98.99

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	43.95
Moisture %	11.30
Protein % (N x 5.7)	12.30
Grams Protein in Fraction	5.40
% of Total Protein	91.52

BOTTOM FRACTION

Amount recovered grams	5.55
Moisture %	12.30
Protein % (N x 5.7)	5.30
Grams Protein in Fraction	.29
% of Total Protein	4.92

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.40	91.52
In Bottom Fraction	.29	4.92
Total	5.69	96.44

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION A

50 GRAMS

Original Flour

Moisture	9.30
Protein	11.80
Ash	.49
Fisher Size	12.00

TOP FRACTION		BOTTOM FRACTION	
Amount recovered grams	1.35	Amount recovered grams	48.00
Moisture %	11.30	Moisture %	11.90
Protein % (N x 5.7)	74.80	Protein % (N x 5.7)	10.20
Grams Protein in Fraction	1.01	Grams Protein in Fraction	4.89
% of Total Protein	17.12	% of Total Protein	82.18

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.01	17.12
In Bottom Fraction	4.89	82.18
Total	5.90	99.30

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION		BOTTOM FRACTION	
Amount recovered grams	36.45	Amount recovered grams	13.20
Moisture %	12.10	Moisture %	12.40
Protein % (N x 5.7)	14.40	Protein % (N x 5.7)	4.30
Grams Protein in Fraction	5.25	Grams Protein in Fraction	.57
% of Total Protein	88.98	% of Total Protein	9.66

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.25	88.98
In Bottom Fraction	.57	9.66
Total	5.82	98.64

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION B

50 GRAMS

Original Flour

Moisture	8.40
Protein	30.20
Ash	1.10
Fisher Size	3.00

TOP FRACTION

Amount recovered grams	12.50
Moisture %	11.60
Protein % (N x 5.7)	79.90
Grams Protein in Fraction	9.99
% of Total Protein	66.16

BOTTOM FRACTION

Amount recovered grams	37.00
Moisture %	11.90
Protein % (N x 5.7)	11.70
Grams Protein in Fraction	4.33
% of Total Protein	28.68

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	9.99	66.16
In Bottom Fraction	4.33	28.68
Total	14.32	94.84

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	43.84
Moisture %	11.80
Protein % (N x 5.7)	32.60
Grams Protein in Fraction	14.30
% of Total Protein	94.08

BOTTOM FRACTION

Amount recovered grams	6.00
Moisture %	12.10
Protein % (N x 5.7)	5.20
Grams Protein in Fraction	.31
% of Total Protein	2.00

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	14.30	94.08
In Bottom Fraction	.31	2.00
Total	14.61	96.08

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION C

50 GRAMS

Original Flour

Moisture	8.90
Protein	15.50
Ash	.66
Fisher Size	7.50

TOP FRACTION

Amount recovered grams	3.05
Moisture %	12.10
Protein % (N x 5.7)	75.20
Grams Protein in Fraction	2.29
% of Total Protein	29.55

BOTTOM FRACTION

Amount recovered grams	46.10
Moisture %	11.80
Protein % (N x 5.7)	11.60
Grams Protein in Fraction	5.35
% of Total Protein	69.03

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	2.29	29.55
In Bottom Fraction	5.35	69.03
Total	7.64	98.58

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	26.95
Moisture %	10.50
Protein % (N x 5.7)	24.50
Grams Protein in Fraction	6.60
% of Total Protein	85.16

BOTTOM FRACTION

Amount recovered grams	22.61
Moisture %	11.90
Protein % (N x 5.7)	4.30
Grams Protein in Fraction	.97
% of Total Protein	12.52

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	6.60	85.16
In Bottom Fraction	.97	12.52
Total	7.57	97.69

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION D

50 GRAMS

Original Flour

Moisture	9.70
Protein	7.60
Ash	.41
Fisher Size	13.00

TOP FRACTION

Amount recovered grams	.70
Moisture %	12.30
Protein % (N x 5.7)	72.10
Grams Protein in Fraction	.50
% of Total Protein	13.28

BOTTOM FRACTION

Amount recovered grams	48.20
Moisture %	11.90
Protein % (N x 5.7)	6.80
Grams Protein in Fraction	3.27
% of Total Protein	86.25

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.50	13.28
In Bottom Fraction	3.27	86.25
Total	3.77	99.53

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	14.32
Moisture %	11.30
Protein % (N x 5.7)	17.60
Grams Protein in Fraction	2.52
% of Total Protein	66.32

BOTTOM FRACTION

Amount recovered grams	35.20
Moisture %	10.60
Protein % (N x 5.7)	3.30
Grams Protein in Fraction	1.16
% of Total Protein	30.53

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	2.52	66.32
In Bottom Fraction	1.16	30.53
Total	3.68	96.85

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION E

50 GRAMS

Original Flour

Moisture	9.90
Protein	7.10
Ash	.37
Fisher Size	19.50

TOP FRACTION

Amount recovered grams	.30
Moisture %	11.30
Protein % (N x 5.7)	66.40
Grams Protein in Fraction	.20
% of Total Protein	5.63

BOTTOM FRACTION

Amount recovered grams	49.00
Moisture %	11.60
Protein % (N x 5.7)	6.80
Grams Protein in Fraction	3.33
% of Total Protein	93.80

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.20	5.63
In Bottom Fraction	3.33	93.80
Total	3.53	99.43

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	14.10
Moisture %	11.90
Protein % (N x 5.7)	16.50
Grams Protein in Fraction	2.33
% of Total Protein	65.63

BOTTOM FRACTION

Amount recovered grams	35.56
Moisture %	11.60
Protein % (N x 5.7)	3.20
Grams Protein in Fraction	1.14
% of Total Protein	32.11

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	2.33	65.63
In Bottom Fraction	1.14	32.11
Total	3.47	97.74

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION BB

50 GRAMS

Original Flour

Moisture	9.40
Protein	10.70
Ash	.43
Fisher Size	17.50

TOP FRACTION

Amount recovered grams	.81
Moisture %	11.30
Protein % (N x 5.7)	69.80
Grams Protein in Fraction	.57
% of Total Protein	10.65

BOTTOM FRACTION

Amount recovered grams	48.23
Moisture %	11.70
Protein % (N x 5.7)	9.80
Grams Protein in Fraction	4.72
% of Total Protein	88.35

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.57	10.65
In Bottom Fraction	4.72	88.35
Total	5.29	98.80

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	23.86
Moisture %	11.70
Protein % (N x 5.7)	17.70
Grams Protein in Fraction	4.22
% of Total Protein	78.88

BOTTOM FRACTION

Amount recovered grams	25.81
Moisture %	11.80
Protein % (N x 5.7)	3.60
Grams Protein in Fraction	.93
% of Total Protein	17.38

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.22	78.88
In Bottom Fraction	.93	17.38
Total	5.15	96.26

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION CC

50 GRAMS

Original Flour

Moisture	9.80
Protein	9.60
Ash	.37
Fisher Size	19.00

TOP FRACTION

Amount recovered grams	.50
Moisture %	11.00
Protein % (N x 5.7)	63.80
Grams Protein in Fraction	.32
% of Total Protein	6.66

BOTTOM FRACTION

Amount recovered grams	48.75
Moisture %	12.10
Protein % (N x 5.7)	9.20
Grams Protein in Fraction	4.41
% of Total Protein	91.88

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.32	6.66
In Bottom Fraction	4.41	91.88
Total	4.73	98.54

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	24.25
Moisture %	11.00
Protein % (N x 5.7)	15.30
Grams Protein in Fraction	3.71
% of Total Protein	77.30

BOTTOM FRACTION

Amount recovered grams	24.81
Moisture %	11.40
Protein % (N x 5.7)	3.70
Grams Protein in Fraction	.92
% of Total Protein	19.17

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	3.71	77.30
In Bottom Fraction	.92	19.17
Total	4.63	96.47

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION DD

50 GRAMS

Original Flour

Moisture	9.80
Protein	10.00
Ash	.36
Fisher Size	24.00

TOP FRACTION

Amount recovered grams	.37
Moisture %	10.90
Protein % (N x 5.7)	59.80
Grams Protein in Fraction	.22
% of Total Protein	4.40

BOTTOM FRACTION

Amount recovered grams	48.20
Moisture %	12.30
Protein % (N x 5.7)	9.90
Grams Protein in Fraction	4.77
% of Total Protein	95.54

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.22	4.40
In Bottom Fraction	4.77	95.54
Total	4.99	99.94

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	28.00
Moisture %	12.30
Protein % (N x 5.7)	14.30
Grams Protein in Fraction	4.01
% of Total Protein	80.20

BOTTOM FRACTION

Amount recovered grams	21.51
Moisture %	11.00
Protein % (N x 5.7)	4.00
Grams Protein in Fraction	.86
% of Total Protein	17.20

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.01	80.20
In Bottom Fraction	.86	17.20
Total	4.87	97.40

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILLED FLOUR
FRACTION EE

50 GRAMS

Original Flour

Moisture	9.90
Protein	11.40
Ash	.37
Fisher Size	26.50

TOP FRACTION

Amount recovered grams	.39
Moisture %	11.90
Protein % (N x 5.7)	61.80
Grams Protein in Fraction	.24
% of Total Protein	4.21

BOTTOM FRACTION

Amount recovered grams	49.12
Moisture %	12.30
Protein % (N x 5.7)	10.80
Grams Protein in Fraction	5.30
% of Total Protein	92.98

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.24	4.21
In Bottom Fraction	5.30	92.98
Total	5.54	97.19

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	30.10
Moisture %	12.10
Protein % (N x 5.7)	15.00
Grams Protein in Fraction	4.52
% of Total Protein	79.30

BOTTOM FRACTION

Amount recovered grams	18.07
Moisture %	11.7C
Protein % (N x 5.7)	5.20
Grams Protein in Fraction	.94
% of Total Protein	16.49

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.52	79.30
In Bottom Fraction	.94	16.49
Total	5.46	95.79

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

MAGIC MILL
1 PASSE

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.34
Moisture %	12.30
Protein % (N x 5.7)	74.90
Grams Protein in Fraction	1.00
% of Total Protein	16.95

BOTTOM FRACTION

Amount recovered grams	48.11
Moisture %	11.70
Protein % (N x 5.7)	9.90
Grams Protein in Fraction	4.76
% of Total Protein	80.68

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.00	16.95
In Bottom Fraction	4.76	80.68
Total	5.76	97.63

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	36.32
Moisture %	11.70
Protein % (N x 5.7)	15.20
Grams Protein in Fraction	5.52
% of Total Protein	93.56

BOTTOM FRACTION

Amount recovered grams	13.10
Moisture %	12.20
Protein % (N x 5.7)	2.30
Grams Protein in Fraction	.30
% of Total Protein	5.08

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.52	93.56
In Bottom Fraction	.30	5.08
Total	5.82	98.64

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

MAGIC MILL
2 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.41
Moisture %	11.30
Protein % (N x 5.7)	71.53
Grams Protein in Fraction	1.01
% of Total Protein	17.12

BOTTOM FRACTION

Amount recovered grams	47.92
Moisture %	12.40
Protein % (N x 5.7)	9.80
Grams Protein in Fraction	4.69
% of Total Protein	79.49

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.01	17.12
In Bottom Fraction	4.69	79.49
Total	5.70	96.61

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	32.45
Moisture %	10.95
Protein % (N x 5.7)	16.50
Grams Protein in Fraction	5.35
% of Total Protein	90.68

BOTTOM FRACTION

Amount recovered grams	17.15
Moisture %	11.50
Protein % (N x 5.7)	2.80
Grams Protein in Fraction	.48
% of Total Protein	8.13

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.35	90.68
In Bottom Fraction	.48	8.13
Total	5.83	98.81

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

MAGIC MILL
3 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.87
Moisture %	10.80
Protein % (N x 5.7)	72.38
Grams Protein in Fraction	1.35
% of Total Protein	22.88

BOTTOM FRACTION

Amount recovered grams	47.52
Moisture %	11.50
Protein % (N x 5.7)	9.30
Grams Protein in Fraction	4.42
% of Total Protein	74.96

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.35	22.88
In Bottom Fraction	4.42	74.96
Total	5.77	97.84

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	31.12
Moisture %	11.80
Protein % (N x 5.7)	16.70
Grams Protein in Fraction	5.20
% of Total Protein	88.13

BOTTOM FRACTION

Amount recovered grams	18.60
Moisture %	12.10
Protein % (N x 5.7)	2.60
Grams Protein in Fraction	.48
% of Total Protein	8.14

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.20	88.13
In Bottom Fraction	.48	8.14
Total	5.68	96.27

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

MAGIC MILL
4 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.89
Moisture %	12.50
Protein % (N x 5.7)	73.43
Grams Protein in Fraction	1.39
% of Total Protein	23.56

BOTTOM FRACTION

Amount recovered grams	47.32
Moisture %	11.00
Protein % (N x 5.7)	9.20
Grams Protein in Fraction	4.35
% of Total Protein	73.73

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.39	23.56
In Bottom Fraction	4.35	73.73
Total	5.74	97.29

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	30.05
Moisture %	11.80
Protein % (N x 5.7)	17.80
Grams Protein in Fraction	5.35
% of Total Protein	90.67

BOTTOM FRACTION

Amount recovered grams	19.55
Moisture %	12.00
Protein % (N x 5.7)	2,50
Grams Protein in Fraction	.49
% of Total Protein	8.31

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.35	90.67
In Bottom Fraction	.49	8.31
Total	5.84	98.98

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

MAGIC MILL
5 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	2.58
Moisture %	10.50
Protein % (N x 5.7)	80.52
Grams Protein in Fraction	2.08
% of Total Protein	35.25

BOTTOM FRACTION

Amount recovered grams	46.98
Moisture %	12.20
Protein % (N x 5.7)	7.80
Grams Protein in Fraction	3.66
% of Total Protein	62.03

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	2.08	35.25
In Bottom Fraction	3.66	62.03
Total	5.74	97.23

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	18.08
Moisture %	11.80
Protein % (N x 5.7)	24.10
Grams Protein in Fraction	4.36
% of Total Protein	73.90

BOTTOM FRACTION

Amount recovered grams	32.50
Moisture %	12.60
Protein % (N x 5.7)	4.10
Grams Protein in Fraction	1.33
% of Total Protein	22.54

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.36	73.90
In Bottom Fraction	1.33	22.54
Total	5.69	96.44

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILL
1 PASSE

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.33
Moisture %	11.30
Protein % (N x 5.7)	70.99
Grams Protein in Fraction	.94
% of Total Protein	15.93

BOTTOM FRACTION

Amount recovered grams	47.88
Moisture %	11.80
Protein % (N x 5.7)	9.90
Grams Protein in Fraction	4.74
% of Total Protein	80.34

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.94	15.93
In Bottom Fraction	4.74	80.34
Total	5.68	96.27

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	35.45
Moisture %	11.70
Protein % (N x 5.7)	15.10
Grams Protein in Fraction	5.35
% of Total Protein	90.68

BOTTOM FRACTION

Amount recovered grams	14.10
Moisture %	12.20
Protein % (N x 5.7)	3.21
Grams Protein in Fraction	.45
% of Total Protein	7.63

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.35	90.68
In Bottom Fraction	.45	7.63
Total	5.80	98.31

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILL
2 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.84
Moisture %	11.90
Protein % (N x 5.7)	74.69
Grams Protein in Fraction	1.37
% of Total Protein	23.22

BOTTOM FRACTION

Amount recovered grams	47.82
Moisture %	11.40
Protein % (N x 5.7)	9.10
Grams Protein in Fraction	4.35
% of Total Protein	73.73

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.37	23.22
In Bottom Fraction	4.35	72.73
Total	5.72	96.95

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	26.82
Moisture %	10.50
Protein % (N x 5.7)	18.50
Grams Protein in Fraction	4.96
% of Total Protein	84.07

BOTTOM FRACTION

Amount recovered grams	22.80
Moisture %	11.50
Protein % (N x 5.7)	3.70
Grams Protein in Fraction	.84
% of Total Protein	14.24

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.96	84.07
In Bottom Fraction	.84	14.24
Total	5.80	98.31

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILL
3 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	2.06
Moisture %	10.80
Protein % (N x 5.7)	96.39
Grams Protein in Fraction	1.57
% of Total Protein	26.61

BOTTOM FRACTION

Amount recovered grams	47.41
Moisture %	11.60
Protein % (N x 5.7)	8.70
Grams Protein in Fraction	4.12
% of Total Protein	69.83

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.57	27.07
In Bottom Fraction	4.12	69.83
Total	5.69	96.90

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	23.17
Moisture %	11.90
Protein % (N x 5.7)	20.80
Grams Protein in Fraction	4.82
% of Total Protein	81.69

BOTTOM FRACTION

Amount recovered grams	26.50
Moisture %	12.50
Protein % (N x 5.7)	3.90
Grams Protein in Fraction	1.03
% of Total Protein	17.46

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.82	81.69
In Bottom Fraction	1.03	17.46
Total	5.85	99.15

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILL
4 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	2.39
Moisture %	11.90
Protein % (N x 5.7)	79.68
Grams Protein in Fraction	1.90
% of Total Protein	32.20

BOTTOM FRACTION

Amount recovered grams	47.15
Moisture %	11.50
Protein % (N x 5.7)	8.20
Grams Protein in Fraction	3.87
% of Total Protein	65.59

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.90	32.20
In Bottom Fraction	3.87	65.59
Total	5.77	97.79

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	18.92
Moisture %	11.20
Protein % (N x 5.7)	23.30
Grams Protein in Fraction	4.41
% of Total Protein	74.75

BOTTOM FRACTION

Amount recovered grams	30.70
Moisture %	11.80
Protein % (N x 5.7)	4.00
Grams Protein in Fraction	1.23
% of Total Protein	20.85

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	4.41	74.75
In Bottom Fraction	1.23	20.85
Total	5.64	95.60

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

PIN MILL
5 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	1.85
Moisture %	11.80
Protein % (N x 5.7)	74.02
Grams Protein in Fraction	1.37
% of Total Protein	23.22

BOTTOM FRACTION

Amount recovered grams	47.00
Moisture %	12.30
Protein % (N x 5.7)	9.40
Grams Protein in Fraction	4.42
% of Total Protein	74.96

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	1.35	23.22
In Bottom Fraction	4.42	74.96
Total	5.79	98.18

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	29.12
Moisture %	11.70
Protein % (N x 5.7)	17.90
Grams Protein in Fraction	5.21
% of Total Protein	88.31

BOTTOM FRACTION

Amount recovered grams	20.50
Moisture %	12.50
Protein % (N x 5.7)	2.50
Grams Protein in Fraction	.51
% of Total Protein	8.64

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.21	88.31
In Bottom Fraction	.51	8.64
Total	5.72	96.95

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UDY MILL
1 PASSE

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	.75
Moisture %	12.30
Protein % (N x 5.7)	67.08
Grams Protein in Fraction	.50
% of Total Protein	8.47

BOTTOM FRACTION

Amount recovered grams	48.49
Moisture %	11.90
Protein % (N x 5.7)	10.80
Grams Protein in Fraction	5.24
% of Total Protein	88.81

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.50	8.47
In Bottom Fraction	5.24	88.81
Total	5.74	97.28

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	38.60
Moisture %	12.10
Protein % (N x 5.7)	14.10
Grams Protein in Fraction	5.44
% of Total Protein	92.20

BOTTOM FRACTION

Amount recovered grams	11.10
Moisture %	12.50
Protein % (N x 5.7)	3.40
Grams Protein in Fraction	.38
% of Total Protein	6.44

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.44	92.20
In Bottom Fraction	.38	6.44
Total	5.82	98.64

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UDY MILL
2 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	.86
Moisture %	12.10
Protein % (N x 5.7)	69.52
Grams Protein in Fraction	.60
% of Total Protein	10.17

BOTTOM FRACTION

Amount recovered grams	48.35
Moisture %	10.60
Protein % (N x 5.7)	10.60
Grams Protein in Fraction	5.13
% of Total Protein	86.95

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.60	10.17
In Bottom Fraction	5.13	86.95
Total	5.73	97.12

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	38.25
Moisture %	10.70
Protein % (N x 5.7)	13.90
Grams Protein in Fraction	5.32
% of Total Protein	90.17

BOTTOM FRACTION

Amount recovered grams	11.40
Moisture %	11.80
Protein % (N x 5.7)	3.70
Grams Protein in Fraction	.42
% of Total Protein	7.12

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.32	90.17
In Bottom Fraction	.42	7.12
Total	5.74	97.29

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UDY MILL
3 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	.87
Moisture %	12.30
Protein % (N x 5.7)	70.30
Grams Protein in Fraction	.61
% of Total Protein	10.34

BOTTOM FRACTION

Amount recovered grams	48.90
Moisture %	11.40
Protein % (N x 5.7)	10.50
Grams Protein in Fraction	5.13
% of Total Protein	86.95

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.61	10.34
In Bottom Fraction	5.13	86.95
Total	5.74	97.29

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	37.80
Moisture %	11.50
Protein % (N x 5.7)	14.00
Grams Protein in Fraction	5.29
% of Total Protein	89.66

BOTTOM FRACTION

Amount recovered grams	11.80
Moisture %	12.00
Protein % (N x 5.7)	4.10
Grams Protein in Fraction	.48
% of Total Protein	8.14

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.29	89.66
In Bottom Fraction	.48	8.14
Total	5.77	97.80

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UDY MILL
4 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	.89
Moisture %	12.30
Protein % (N x 5.7)	71.50
Grams Protein in Fraction	.64
% of Total Protein	10.85

BOTTOM FRACTION

Amount recovered grams	48.85
Moisture %	11.20
Protein % (N x 5.7)	10.30
Grams Protein in Fraction	5.03
% of Total Protein	85.25

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.64	10.85
In Bottom Fraction	5.03	85.25
Total	5.67	96.10

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	37.80
Moisture %	13.00
Protein % (N x 5.7)	14.30
Grams Protein in Fraction	5.40
% of Total Protein	91.52

BOTTOM FRACTION

Amount recovered grams	11.70
Moisture %	10.90
Protein % (N x 5.7)	3.20
Grams Protein in Fraction	.37
% of Total Protein	6.27

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	5.40	91.52
In Bottom Fraction	.37	6.27
Total	5.77	97.79

Note: Protein + Ash corrected on 14% M.B.

CENTRIFUGAL SEPARATION OF FREE PROTEIN AND STARCH CELLS IN FLOUR WITH SOLVENT MIXTURE OF BENZENE AND CARBON TETRACHLORIDE

SEPARATION AT 1.38 DENSITY FOR FREE PROTEIN

UDY MILL
5 PASSES

50 GRAMS

Original Flour

Moisture	10.70
Protein	11.80
Ash	.46
Fisher Size	18.00

TOP FRACTION

Amount recovered grams	.89
Moisture %	11.70
Protein % (N x 5.7)	71.60
Grams Protein in Fraction	.64
% of Total Protein	10.85

BOTTOM FRACTION

Amount recovered grams	48.92
Moisture %	11.80
Protein % (N x 5.7)	10.70
Grams Protein in Fraction	5.23
% of Total Protein	88.64

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.64	10.86
In Bottom Fraction	5.23	88.64
Total	5.87	99.50

SEPARATION AT 1.48 DENSITY FOR FREE STARCH CELLS

50 GRAMS

TOP FRACTION

Amount recovered grams	37.20
Moisture %	12.30
Protein % (N x 5.7)	14.10
Grams Protein in Fraction	5.27
% of Total Protein	89.32

BOTTOM FRACTION

Amount recovered grams	12.00
Moisture %	11.70
Protein % (N x 5.7)	3.40
Grams Protein in Fraction	.41
% of Total Protein	6.95

PROTEIN BALANCE

	GRAMS	% OF TOTAL
In Top Fraction	.41	89.32
In Bottom Fraction	5.27	6.95
Total	5.68	96.27

Note: Protein + Ash corrected on 14% M.B.

EQUIPMENT USED FOR THE DENSITY SEPARATION

I.E.C. International centrifuge

6-place rotor (IEC 259)

125 ml Pyrex bottle ($ 19)

Pyrex ground joint inner ($ 19/22)

Erlenmeyer for filter pumps (1000 ml)

Porcelain funnel O.D. 89 mm

Filling funnel with short stem

Polypropylene funnel with short stem

Hydrometers:

1. Densities 1.2 - 1.42
2. Densities 1.4 - 1.62

All metal thermometer 0°C- 50°C

Benzene

Carbon tetrachloride

DENSITY SEPARATION BY A NONAQUEOUS SOLVENT OF FINE GROUND AND AIR-CLASSIFIED FLOUR FRACTIONS.

by

ABDERRAHMANE MOUFFOK

B.S.,University of Algiers, ALGERIA ,1976

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1982

ABSTRACT

The production of free starch and free protein by the dry operations of grinding and air classification would seem to offer certain operational and, therefore, economic advantages, particularly over the wet operations presently used.

Two separate studies were conducted on the fractions obtained by three different grinders and the turbo air classifier.

A commercially milled 11.8% protein (14% M.B.) flour was broken down into two lots: unground and pin milled.

Regrinding several times by means of different grinders (pin mill, magic mill, Udy mill) were chosen to accentuate changes in particle size and evaluate their efficiency for freeing the protein and starch from the endosperm chunks.

The fractionated flour was separated by means of the difference in density of starch and protein. Mixtures of carbon tetrachloride and benzene adjusted to the desired densities were used as the fractionating media.

The results of the investigations indicate that several regrindings were efficient for freeing the protein particle in the case of the magic mill. Three successive passes in the pin mill gave a good result but inefficient beyond that. The Udy mill did not show much change in regrinding flour.

The type of grinding was also critical. Selective grinding rather than total particle size reduction was needed.

The reground flour, before air classification, was quite effective in increasing yields of high and low protein fractions.

The percent protein in the floating fraction was increased by finer grinding. The protein content of the starch fraction was extremely low which suggests that the starch could be of significant use to the food industry.

This research work resulted in a simple technique to forecast the effect of different grinders on freeing protein and starch.

This is important for protein shifting work.