A STUDY ON THE COMPOSITION OF MILLED BARLEY PRODUCTS

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INTRODUCTION

Barley is the most ancient cultivated grain; compared to wheat, it is more widely distributed and matures more rapidly. In recent years, wheat and rye have displaced barley as an important human food.

The major use of barley is in manufacturing of malt. Owing to its structure and chemical composition, barley is considered the most suitable grain for malt. Barley is also used extensively in brewing and making distilled fermented liquors.

There is a limited amount of published data on chemical composition of barley or its milled products. The purpose of this investigation was to determine the composition of representative barley varieties milled by conventional roller milling, to air separate the flours into high-protein and low-protein fractions, and to evaluate the potential uses of the various fractions.

REVIEW OF LITERATURE

Barley is regarded as the most ancient cultivated grain. It is a winter-hardy, drought- and heat-resistant grain which matures more rapidly than wheat, oats or rye, and is ecologically more widely distributed than wheat (Kent, 1966).

Classification and Grading System of Barley. Barley is included in the genus Hordeum of the grass family Gramineae. Carson and Horne (1962) reviewed the systems of classification of barley. They concluded that the most important of the older systems was proposed by Linnaeus who classified barley into four species; H. distichon, H. vulgare, H. hexastichon, and H. zeocrition based on differences in fertility and density of the ear; and the division into two botanical varieties H. distichon var. nudum and H. vulgare var. coeleste depended on the adherence of the husk to the kernel.

The wild two-row species, H. spontaneum was first described by Koch in 1848. A monophyletic precursor of cultivated barley discovered by Åberg (1938), is wild six-row barley and named H. agriocrithon E. Åberg. More recently, Åberg and Wiebe (1946) divided barleys into three species on the basis of brittleness of the rachis and number of kernel rows of the spike; most of the cultivated barleys belong to two species: six-row barley (H. vulgare L.,) and two-row barley (H. distichum L.,); in addition, Åberg and Wiebe (1946) proposed a name for a newly defined species, H. irregulare.

In practice, barleys are divided into three main types: (a) hulled, six-row barley; (b) hulled, two-row barley; and (c) hull-less barley. The terms hulled two or six row barley refer to the arrangement of the grains on the spike (Kent, 1966).

According to the U. S. grading system, barley is separated into three classes: (a) Barley; (b) Western Barley, and (c) Mixed Barley. The main sub-classes are: (a) Malting Barley, (b) Blue Malting Barley, and (c) Barley, on the basis of areas of production and color. Each sub-class is divided into five numerical grades and sample grade according to test weight, dockage, and percentage of black barley (Leonard and Martin, 1963).

Commercially, barley is classified in grain grading into three types; six-row, two-row, and feed grade (Shellenberger, 1936).

Distribution and Production of Barley. Ancient records show that cultivated barley was used by Neolithic cultures in Egypt between 5,000 and 6,000 B. C. (Weaver, 1950). Major gene centers of barley where cultivated varieties may have developed include Abyssinia and Southern Tibet (Cherry-Downes and Macey, 1967).

Barley is grown in nearly all countries with temperate climates, and is more widely distributed than other crops. It is an annual crop having winter and spring varieties, and thrives best in cold weather. According to Shands and Dickson (1953), Russia, China, United States, Canada lead in production. North Dakota, South Dakota, Minnesota, Nebraska, and California are the leading states in the United States. Cherry-Downes and Macey (1967) pointed out that average world barley yields for the five year period 1958 to 1962 differed widely between the high yielding temperate areas of Western Europe and the lower producing Mediterranean regions. Most of the large-acreage export countries such as United States, Canada and Australia have below average yields per acre. Holland, Belgium, Denmark, and the United Kingdom are high in yield per acre.

As mentioned earlier, the two main types of barley, depending on the arrangement of grains in the ear, are two-row and six-row. The two-row barleys predominate in Britain, Northwestern Europe, and Australia; the six-row type is more resistant to extremes of temperature and is grown in North America, India, and the Middle East. Both types can be used for malting (Pomeranz, 1969). In addition, small amounts of hulless (naked) barley, that is more easily adapted to food processing, are grown in Southeastern Asia (Kent, 1966). In recent years, several high-protein and high-lysine naked barleys were found.

Processing and Technology. There are two types of milling-pearling and milling of barley to grits and flour. Pot and pearl barley are the forms used directly as human food; the third product is barley flour. Pot or pearl barley, both, are manufactured by gradually removing the hull and outer portion of the barley kernel by abrasive action; the pearling or decortication process is carried further in the manufacture of pearl barley. During the pearling, some flour is produced as a by-product. Also, the pearl barley may be ground, sifted and purified to produce the granular products, barley grits, and/or barley flour. This refined flour is known as barley patent flour. Barley flour, as well as barley grits, is also milled by a gradual reduction roller milling process, similar to that used in milling of wheat to flour (Harrel and Dirks, 1955).

Novacek et al., (1966) developed a pearling scheme for separating barley into husk, pericarp, germ, aleurone, and endosperm. The separation was made on a rebolt sifter and/or gravity separator, and was based on particle size and/or density.

Harris and Scott (1947) employed Fraser's modified air-jet method to determine the proportion of hull in North Dakota barley. They found that no preliminary tempering was necessary and that significant differences in hull percentage were found among varieties. Location and growth had less effect than variety, and length of hulling treatment had a large influence on the proportion of hull removed.

According to Swanson and Pence (1932), moisture is the most important factor in wheat milling; the variation in moisture influences the yield of flour, the flour composition, and test weight of wheat. Both tempering and milling procedures influence the separation of endosperm, pericarp, aleurone, and germ from various wheats (Bradbury, 1960).

A more recent development in the wheat flour milling industry concerns fine grinding of conventionally milled flour particles followed by air-classification to separate, in part, protein from starch (Wichser, 1958, Jones et al., 1959, Grosh et al., 1959, Sullivan et al., 1960).

The uses of air-classified flours are numerous. They include: industrial uses based on processing uniform fractions available from a single flour; channeling flour to most proper and economical uses (Stringfellow et al., 1962), and minimizing the adverse influence of climate, soil and constantly changing varieties (Wichser, 1958).

The air-classification increases the protein content of some fractions and starch content of the other fractions and is the basis of numerous patents for the production of "tailor-made" flours from a single patent flour. Griffin (1962) listed the following advantages of air-classification of wheat flour: relatively inexpensive manufacture of more uniform products from varying wheats (in spite of different crop years, locations, varieties and climate factors),

and controlled particle size and chemical composition for the manufacture of new products. Air-classification of corn, sorghum, and rice flours were described by Stringfellow et al., (1962).

The utilization of air-classified wheat flour fractions has been summarized by Wichser (1958). He proposed that the high-protein (20%) fractions be used to enrich low-protein bread flours, that the 8% protein fraction be used as all purpose cake flour, the 7% protein fraction for crackers, and the 6% protein fraction for cake. The 3% protein fraction, which is mainly starch, might be used for industrial starch.

General Uses of Barley and Its Milled Products. The most important uses of barley are as feed grain for livestock, especially in pig rations, as malt for manufacturing beverages or malt-enriched food products, as seed, as human food in the form of parched-grain, pearled grain for soups, flour for flat bread, and ground or partly ground grain to be cooked and eaten as porridge (Wiebe, 1968). The major industrial use of barley is for producing malt. Barley is best suited for malt production due to the endosperm being covered with a husk which protects the growing acrospire from damage and acting as filtering material during the brewing process. Also, barley contains less protein than other malted grains, and its amylase activities are higher than of the other cereal grains (Shellenberger and Bailey, 1936).

Harrel and Dirks (1955) listed the different by-products from such processes as pearling, milling and malting and their individual uses. Phillips and Boerner (1959) also studied the numerous uses of barley.

According to Watson (1953), pot and pearl barley are used for making groats, special flours used for infants and for invalid feeding, and special breakfast foods. On the other hand, the by-products of brewing and distilling are valuable feedstuffs.

Gross Composition of Barley and Milled Barley Products. Published information on the composition of barley and milled barley products is limited. The barley grain consists of the endosperm and the embryo enclosed within the remains of the original glumes called the husk.

On a dry matter basis, barley contains 63-65% starch, 1 to 2% sucrose, about 1% of other sugars, 1 to 1.5% soluble gums, 8 to 10% hemicellulose, 4 to 5% cellulose, 2 to 3% lipids, 8 to 11% protein (N x 6.25), 2 to 2.5% mineral matter, and 5 to 6% other components (Dickson, 1959). The cellulose and ash of barley are higher than of other cereals except oats (about 11% cellulose and 3% ash content); the lipid content in barley is higher than in wheat and rye (about 2%) but lower than in oats and corn (around 5%); and the total protein content in barley is higher than in rice but lower than in rye, oats, and wheat.

Kent (1966) compiled data on the chemical composition of milled barley products. In general, the hull or bran fractions are high in cellulose, pentosans, and ash; germ is high in lipid, protein, sugar, and ash. The endosperm fraction contains mostly starch, but has a lower protein content than the germ, and is low in fat and ash.

Comprehensive tables on food composition have been issued by U.S.D.A. for the last 70 years (Watt and Merrill, 1963). Pearled light barley and pearled pot (or scotch) barley contain per 100 gm.: 11.1% moisture, 8.2 gm. of protein, 1.0 gm. of fat, 78.8 gm. of total carbohydrates, 0.5 gm. of fiber, 0.9 gm. of ash; and 10.8% moisture, 9.6 gm. of protein, 1.1 gm. of fat and 77.2 gm. of carbohydrates, 0.9 gm. of fiber and 1.3 gm. of ash, respectively.

The proximate composition and characteristics of three types of barley were studied by Kneen and Dickson (1967). The barleys were: (a) midwestern six-row Manchurian type, (b) western six-row (coast or bay) brewing barley

grown in California, and (c) western two-row barley. The chemical composition of the barleys was similar, except the California six-row barley had higher fiber and ash content. Among these three types, the midwestern six-row barley was high in enzyme activity after malting. About 90% of malt is made from this type.

During the milling process, the hull, germ and bran (which are high in minerals and vitamins) are largely removed (Kent, 1966). Changes during pearling of barley in ash, protein, and vitamin content were determined by Kuzminskaya (1963) and by Belilovskaya (1964). The water-soluble carbohydrates and free sugars were determined by Preece and Mackenzie (1952a, 1952b) and by MacLeod (1952) in barley fractionated with a small pearling machine.

Proteins of Barley. The proteins of barley are the most important factor in the malting process and determine the character and stability of beer. Early work was done by Osborne (1907) and Bishop (1928). Four fractions of protein were found based on solubility. In the protein, water-soluble albumins comprise 3 to 4%, 10 to 20% are salt-soluble globulins, 35 to 45% are prolamins (70% alcohol soluble fraction), and 35 to 45% are acid or alkali soluble glutelins. deClerck (1958) summarized methods for separation of major groups of protein and estimation of nonprotein nitrogen in barley.

Combining refined precipitation methods with some modern fractionation techniques, such as electrophoresis and column chromatography, Scandinavian workers have established that four globulins and at least five hordein fractions are present in barley (Cherry-Downes and Macey, 1967). The prolamin fraction in barley (known as hordein) is rich in glutamic acid (27.0%) and proline (14.5%): the lysine content is 1.4% and is higher than in prolamins of other cereal grains (McElroy et al., 1949; Robrlich and Thomas, 1967).

Orr and Watt (1957) observed that barley contains significant amounts of nineteen amino acids, and is low in lysine and methionine. Recent work has indicated increased growth rate of animals fed barley supplemented with lysine (Dickson, 1959).

Preece (1954) reviewed composition of barley in relation to malting, among the four groups of protein, as classified by Bishop (1928), he found the salt-soluble barley globulin was a mixture of alpha, beta, gamma, and delta fractions. Barley is the only cereal that contains beta and delta globulins. In some barleys beta-globulin is resistant to modification during malting and brewing; this may be important in formation of hazes in beer. Water-soluble albumins and alcohol-soluble hordein are mixtures of several proteins; hordein is completely degraded during malting. Protein content of barley is influenced by climate, weather, soil, and variety, and is an important index of its quality for manufacture of food products (Bishop, 1930).

Carbohydrates in Barley. Carbohydrates are quantitatively the most important constituent; they constitute about 83% of the total dry matter in barley. Starch is one of the most important carbohydrate fractions and constitutes about two-thirds of the dry weight of barley (Harris, 1962). The barley starch contains 23% amylose which consisted of chains about 400 α-1:4- linked glucose residues. The amylopectin component had a unit chain length of 26 $\pm$ 2 glucose residues and at least 86% of the branched linkages were 1:6-α glucosidic bonds (Harris, 1962, Kent, 1966). Cellulose is the main component of most of the cell walls of cereal grains; assayed as crude fiber, it is about 4 to 5% of the weight of the grain, the hemicellulose and gums consist of 10 to 11% and 1 to 2%, respectively, of the weight of the grain (Harris, 1962).

Free sugars are important in the malting of barley. The free sugars were studied by Harris (1962). Barley contains per 100 gm. dry matter: 20-93 mg. glucose, 33-159 mg. fructose, 0-135 mg. maltose, 343-1690 mg. sucrose, 144-832 mg. raffinose, 70-433 mg. glucodifructose, 97-536 mg. ethanol-soluble fructosans, and 40-900 mg. water-soluble fructosans. Meredith et al., (1951) isolated the gum-like polysaccharides from barley, malt, and wort. They found the principal sugars in all gums were glucose, arabinose, and xylose; and there were trace amounts of galacturonic acid, galactose, and mannose. The ratio of glucose : arabinose : xylose in raw gums of barley was found to be 1 : 0.1 : 0.09, in malt it was 1 : 0.23 : 0.13, and in wort it was 1 : 0.18 : 0.14.

Lipids in Barley. Barley and malt lipids were investigated by Walsh et al., (1965) and by Banasik and Gilles (1966). The total fat content of barley is 2 to 3% of the dry weight of the grain. The value depends on the method of extraction.

MacLeod and White (1961) examined the distribution of petroleum-ether-extracted lipids in barley fractionated by small pearling machine. They observed that the husk or hull contains about 10% of the total fat of barley; aleurone cells, embryo, and scutellum contributed nearly 60%; and the residual 30% was found in the flour, endosperm, and furrow tissues. Fatty acids in barley fat were determined as 2.6% stearic acid, 7.4% palmitic acid, 26.5% oleic acid, 43.7% linoleic acid, and 0.44% linolenic acid; and fat contained 5.4% of unsaponifiable matter.

Walsh et al., (1965) used thin-layer chromatography and colorimetric methods for qualitative and quantitative determination of barley and malt lipids.

Four classes were identified as (a) phospholipids, (b) mono- and di-glycerides, (c) triglycerides, and (d) hydrocarbon and sterol esters.

MacLeod and White (1961) showed a 43% decrease in fat during malting. The decrease was mainly due to the losses of about 75% of the oleic acid and 50% of linoleic and linolenic acid of the barley fat. On the other hand, palmitic acid and stearic acid were significantly increased.

Minerals and Vitamins. The barley kernel contains, on a dry matter basis, 2 to 2.5% minerals. About 95% of the minerals are phosphates and sulphates of potassium, magnesium, and calcium (Kent, 1966).

According to the data quoted by Kent (1966), barley contains in 100 gm. of dry matter : 580 mg. potassium, 440 mg. phosphorus, 160 mg. sulfur, 180 mg. magnesium, 120 mg. chloride, 50 mg. calcium, 77 mg. sodium, and 420 mg. silicon. Potassium, chloride, and silicon are higher than in wheat. Minor minerals include iron, zinc, manganese, and copper. The husk of barley contains 6.0% ash, and 65.8% of silica in the ash.

The vitamins in barley (per gm., dry matter basis) are: 6.5 µg. thiamin, 1.2 µg. riboflavin, 115 µg. nicotinic acid, 4.4 µg. pantothenic acid, 11.5 µg. pyridoxin, and 1100 µg. of choline.

Watson (1953) reported that whole barley meal contained little carotene, no vitamin C, and moderate amounts of the vitamin B complex.

During the pearling process, despite the reduction in protein and fat, and increase in carbohydrates, the mineral content decreases (Watson, 1953). Calcium was found to decrease from 0.075% to 0.016%, phosphorus was reduced from 0.373% to 0.189%, and iron from 0.005% to 0.002%. The decrease in vitamins per 100 gm. of grain was: 500-600 µg. thiamine to about 180 µg., riboflavin decreased from 95-139 µg. to 80 µg.; vitamin A values from 70 international units to nil as a result of removing the outer layers of the barley kernel.

MATERIALS AND METHODS

Sources of Barley. Five barleys were used in this study. Some of their characteristics are given in Table 1.

Table 1. Some Characteristics of Barleys Used in the Milling Study

Variety	Crop year	Kernel weight (mg.)	Kernel Size Assortment				Plump barley (%)	Moisture (%)	Agtron color
			Over 7/64 (%)	Over 6/64 (%)	Over 5/64 (%)	Through 5/64 (%)			
Primus	1968	27.2	7.7	43.3	41.0	8.0	51.0	10.0	48
Larker	1967	33.0	18.5	65.6	14.4	1.5	84.1	11.0	84
Paragon	1968	31.8	6.0	57.4	36.6	0	63.4	11.1	80
Betzes	1967	35.2	33.0	57.5	8.4	1.1	90.5	10.5	90
Atlas	1965	38.4	30.6	54.3	15.0	0.1	84.9	9.0	66

Primus is a six-row, white malting variety; the sample used was grown in South Dakota. Larker is the leading six-row malting variety for the Red River Valley in the United States. The samples were from Casselton, North Dakota. Paragon is a six-row, blue-aleurone barley, released as a malting variety in Canada. The sample was from Casselton, North Dakota. Betzes is a two-row, white malting variety, grown extensively in Montana, Colorado, and Idaho. The sample was grown in Aberdeen, Idaho. Atlas is a six-row coast type barley, formerly used for malting in England, and now used for malting to a limited extent in California. The sample was grown in the Sacramento Valley, California. Atlas is considered of poor quality in malting compared with the other varieties.

Conventional Roller Milling Process. The samples were milled in a Miag Multomat mill. The flow sheet for the mill is given in Fig. 1. The samples of barley were tempered with 0.5% water 30 minutes before milling. The feed rate was set at approximately 400 gm. per minute. Fourteen fractions were collected. The break shorts, reduction shorts, and red dog fractions were combined and ground on an Alpine Pin Mill at 15,000 r.p.m. The ground material was sifted through a 10XX sieve to separate the throughs (tailings flour) and shorts fractions. Later the throughs were mixed with the ten original flour fractions to make a straight grade flour of 65% extraction. The scheme is summarized in Fig. 2.

Air-classification Process. The straight grade flours (65% extraction) from conventional roller milling were fractionated into high- and low-proteins streams, according to scheme in Fig. 3. Fractionations were made in a Pillsbury Laboratory Model No. 1 Classifier employing indicated feed rates, speed, and internal set-up. The fractionation procedure involved removing fine fractions B, C, D and E from the original flour, A; the residue having coarse particle size flour was designated as EE.

Analytical Determinations. Analytical determinations were made on milled products or in materials ground to pass a 40-mesh Micro Wiley mill. Moisture, ash, Kjeldahl protein, and crude fiber were determined as described in Cereal Laboratory Methods (AACC, 1962). Kjeldahl nitrogen was converted to protein by the factor 6.25. Protein by the Udy dye absorption method (Olson and Heiges, 1962) and color of barley and milled barley products (Agtron) were determined according to manufactures' instruction. Particle size (average diameter in μ) was determined in a Fisher Sub-Sieve Sizer (No. 14-311), as described by Croteau (1960).

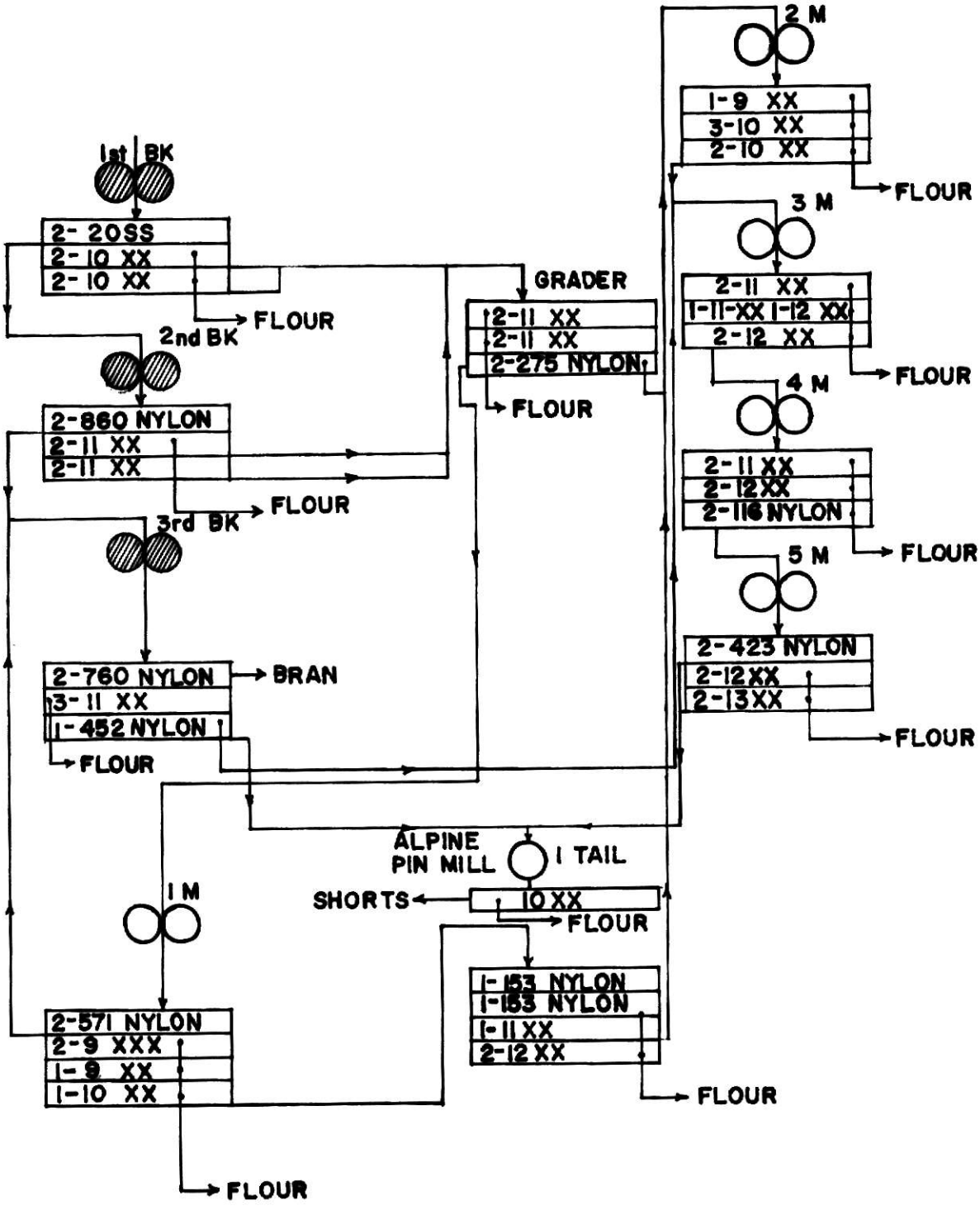

Fig. 1. Flow sheet of Miag Multomat used to mill barley samples.

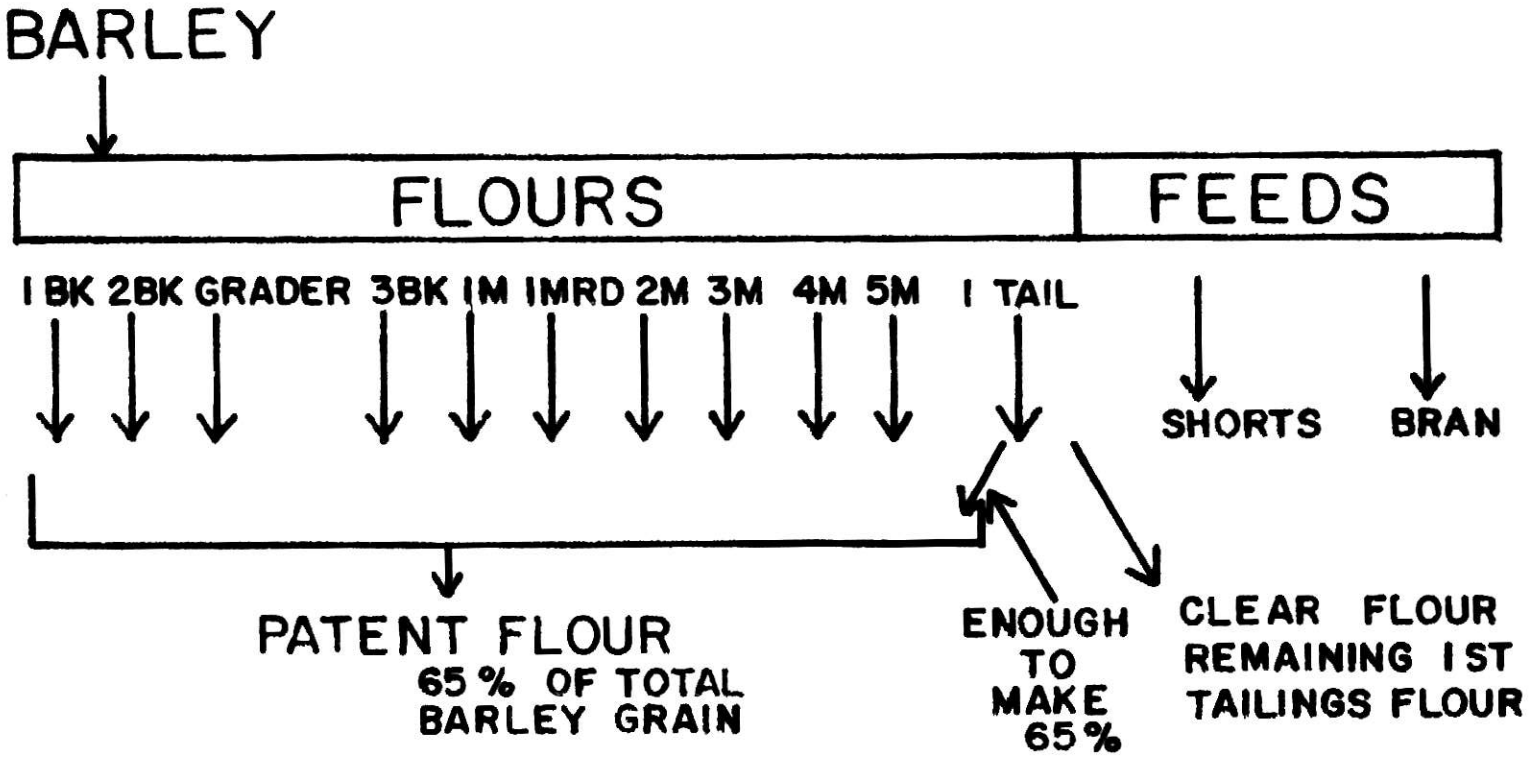

Fig. 2. Streams used in preparing a patent barley flour.

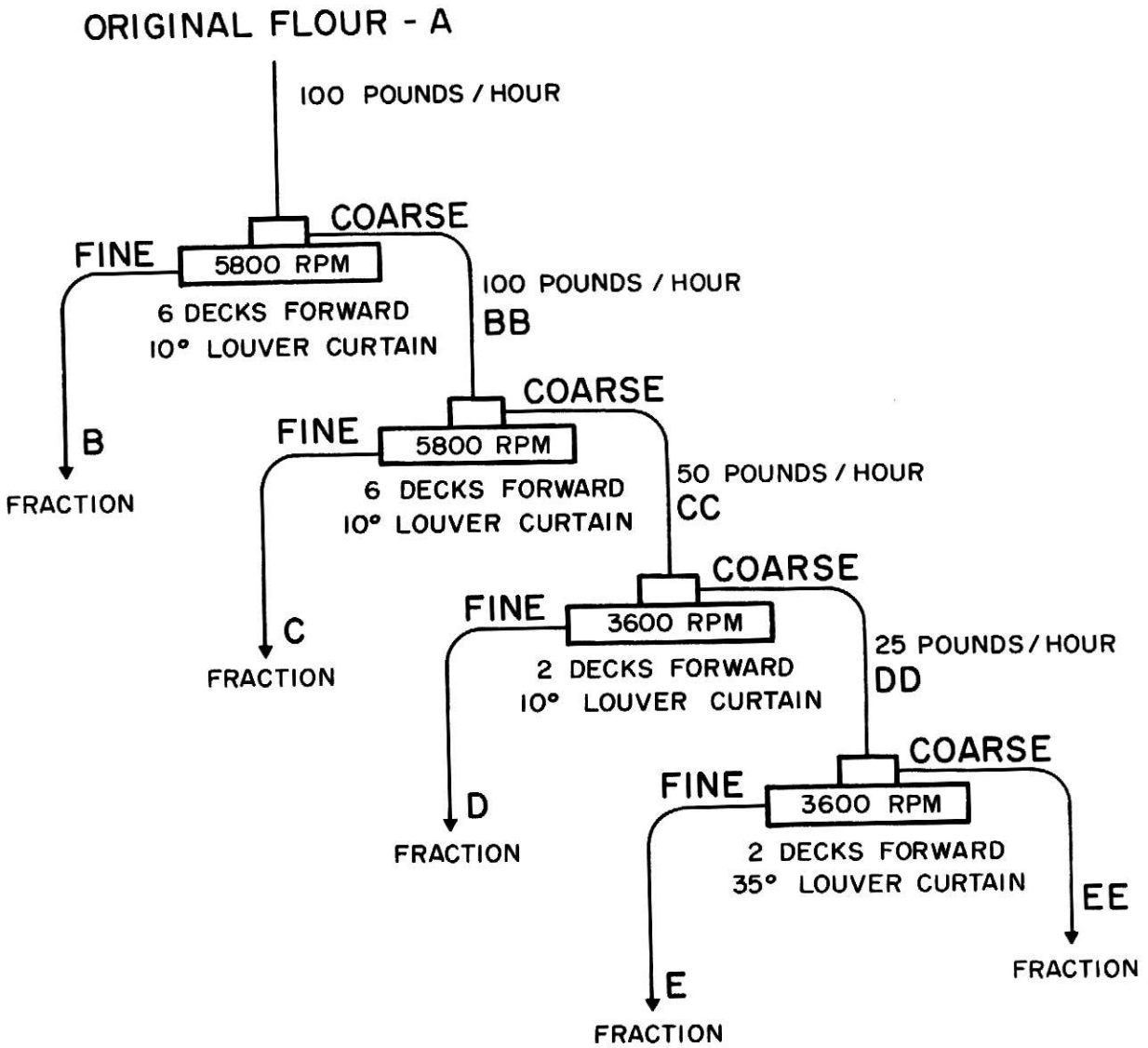

Fig. 3. Flow diagram of procedure used in air fractionation of barley flour.

Free lipids were extracted exhaustively (10 to 12 hours) with petroleum-ether (b.p. 35 to 60°C) in a Goldfish extractor. Petroleum-ether in the flour was evaporated at room temperature, the products were re-extracted twice with 100 ml. and 90 ml. water-saturated 1-butanol, as described elsewhere (Daftary and Pomeranz, 1965). The combined butanol extract (bound lipids) was filtered, evaporated under reduced pressure, redissolved in petroleum-ether, and cleared by centrifugation at 4650 x G. The results shown are means of at least two determinations. The free and bound lipids were stored at 4°.

Thin-layer chromatography was performed on 100 γ of lipids. The lipids were fractionated on standard glass plates (20 cm. x 20 cm.) coated with a 250 micron layer of Silica Gel G (E. Merck, A. G., Darmstadt, Germany), applied in a slurry of 30 gm. Silica Gel G in 60 ml. distilled water. The plates were activated for three hours at 130°C and allowed to cool in a desiccator. Chloroform was used to fractionate nonpolar lipids; the mixture of chloroform-methanol-water (65:25:4 by volume), according to Wagner et al., (1961), was used to fractionate polar components. All solvents were of analytical grade and redistilled before use. The chromatographic chambers were lined with filter paper to ensure saturation of the enclosed space with solvent vapors. After the solvent was allowed to travel about 14 cm, the plates were taken out and dried at room temperature. Plates were sprayed with a saturated solution of potassium dichromate in 70% (by volume) of aqueous sulfuric acid and charred at 180°C for 25 minutes (Blank et al., 1964). The plates were photographed under ultraviolet light. More specific spraying methods also were employed: they included molybdenum spray (Dittmer and Lester, 1964) and modified Dragendorff reagent (Mangold, 1961). Pure compounds used for identification of lipids included triolein, 1, 2-diolein, 1-monopalmitin, linolenic

acid, and linoleic acid for nonpolar lipids; monogalactosyl diglyceride, digalactosyl diglyceride, lysolecithin, phosphatidyl ethanolamine, phosphatidyl serine, and phosphatidyl choline for polar lipids.

RESULT AND DISCUSSION

Some chemical characteristics of the whole barley (with hull) used in the milling study are summarized in Table 2. The protein and crude fiber contents in the five samples varied widely. The range of protein content was from 7.7% to 10.9%, and of crude fiber from 3.6 to 5.4%. The ash content in Atlas was significantly lower than in the other varieties. Differences in free lipid or bound lipid contents in the five samples were small. For calculation of carbohydrates, the following formula was used:

Carbohydrates (by difference) % = 100% - (moisture % + crude fiber % + ash % + protein % + free lipid %).

The carbohydrate contents varied from 66.2% to 69.9%.

Table 2. Some Chemical Characteristics[a] of Barleys Used in the Milling Study

Variety	Ash (%)	Protein (N x 6.25) (%)	Lipids		Crude fiber (%)	Carbohydrates (by difference) (%)
			Free (%)	Bound (%)		
Primus	2.29	10.5	1.56	1.33	5.4	66.2
Larker	2.30	10.7	1.59	1.37	4.2	67.2
Paragon	2.51	9.4	1.51	1.26	4.2	68.4
Betzes	2.23	10.9	1.47	1.49	3.6	67.8
Atlas	1.87	7.7	1.47	1.27	5.0	69.9

[a]Expressed on a 14% moisture basis

Table 3 to Table 7 compare the gross composition of the milled barley products; among the thirteen fractions, the first four flour fractions were from the break system, the following six flour fractions were from

Table 3. Gross Chemical Composition[a] of Milled Products from Primus Barley

Mill stream	Yield (%)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Free lipid (%)
Barley	100.0	-	2.29	10.5	1.56
1st Break flour	6.6	45.0	0.96	6.6	1.12
2nd Break flour	5.5	60.0	0.78	8.1	1.20
Grader flour	2.0	50.0	0.90	8.3	1.50
3rd Break flour	11.6	49.0	1.20	10.3	1.67
1st Middlings flour	4.0	57.0	0.80	8.3	1.36
1st Middlings reduction flour	1.3	54.0	0.78	7.9	1.33
2nd Middlings flour	6.6	55.0	1.02	10.6	2.19
3rd Middlings flour	7.6	50.0	1.27	10.6	1.95
4th Middlings flour	5.7	45.0	1.45	11.6	2.28
5th Middlings flour	3.3	39.0	1.79	12.7	2.65
Tailings flour	25.1	15.5	2.78	13.5	2.91
Shorts	14.7	-	4.18	9.8	2.53
Bran	6.0	-	5.86	4.1	0.89

[a] Expressed on 14% moisture basis.

Table 4. Gross Chemical Composition[a] of Milled Products from Larker Barley

Mill stream	Yield (%)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Lipids		Crude fiber (%)	Carbohydrates (by difference) (%)
					Free (%)	Bound (%)		
Barley	100.0	---	2.30	10.7	1.59	1.37	5.42	67.2
1st Break flour	5.4	49.0	1.07	7.2	1.13	0.79	0.86	75.8
2nd Break flour	6.3	59.5	0.80	8.8	1.07	0.39	0.67	74.7
Grader flour	1.9	50.5	0.97	8.8	1.43	0.91	0.69	74.1
3rd Break flour	12.4	55.5	1.21	10.6	1.53	0.95	1.09	72.6
1st Middlings flour	4.0	70.5	0.72	8.0	1.14	0.49	0.96	75.2
1st Middlings red. flour	1.4	63.0	0.80	8.0	1.27	0.30	0.63	75.3
2nd Middlings flour	8.0	69.0	0.91	9.8	1.79	0.82	0.94	73.6
3rd Middlings flour	7.5	58.5	1.37	11.1	1.98	0.89	1.38	70.1
4th Middlings flour	5.4	51.5	1.48	12.1	2.29	1.19	0.91	69.2
5th Middlings flour	3.8	47.0	1.66	12.3	2.45	1.85	0.96	68.6
Tailings flour	22.6	22.0	3.18	12.9	2.75	1.45	3.73	63.4
Shorts	16.0	---	4.68	10.6	2.68	1.22	9.27	58.8
Bran	5.3	---	6.49	5.5	1.21	0.62	21.64	51.2

[a] Expressed on 14% moisture basis.

Table 5. Gross Chemical Composition[a] of Milled Products from Paragon Barley

Mill stream	Yield (%)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Lipids Free (%)	Lipids Bound (%)	Crude fiber (%)	Carbohydrates (by difference) (%)
Barley	100.0	---	2.51	9.4	1.51	1.26	4.16	68.4
1st Break flour	5.5	48.5	1.07	6.0	1.02	0.64	1.07	76.8
2nd Break flour	6.1	58.5	0.80	7.6	1.05	0.44	0.63	75.9
Grader flour	2.0	55.0	0.93	7.6	1.39	0.88	0.81	75.3
3rd Break flour	12.8	48.5	1.14	8.9	1.38	1.08	0.86	74.6
1st Middlings flour	2.9	61.0	0.74	7.2	1.14	0.98	0.71	76.2
1st Middlings red. flour	1.1	53.5	0.82	7.7	1.34	0.58	0.68	76.1
2nd Middlings flour	5.9	55.5	1.00	9.2	1.98	0.50	0.73	73.1
3rd Middlings flour	7.7	41.0	1.35	9.3	1.93	1.40	1.31	72.1
4th Middlings flour	5.6	43.0	1.41	10.5	2.18	0.70	1.10	70.8
5th Middlings flour	3.5	39.0	1.67	11.3	2.48	0.83	1.64	68.9
Tailings flour	25.2	14.0	2.84	11.2	2.52	1.39	2.69	66.7
Shorts	15.1	---	4.52	9.9	2.56	0.56	8.01	61.0
Bran	6.6	---	7.20	6.0	1.45	0.73	17.9	53.4

[a] Expressed on 14% moisture basis.

Table 6. Gross Chemical Composition[a] of Milled Products from Betzes Barley

Mill stream	Yield (%)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Free lipid (%)
Barley	100.0	-	2.23	10.9	1.47
1st Break flour	3.4	26.5	1.78	8.6	1.74
2nd Break flour	4.5	39.5	1.06	9.5	1.45
Grader flour	1.6	45.0	1.19	8.9	1.77
3rd Break flour	11.5	50.0	1.33	11.1	1.62
1st Middlings flour	4.5	57.0	0.86	8.0	1.28
1st Middlings reduction flour	1.5	55.0	0.86	7.6	1.28
2nd Middlings flour	11.6	57.0	0.81	8.8	1.43
3rd Middlings flour	8.5	47.0	1.30	10.6	1.79
4th Middlings flour	6.8	52.0	1.28	10.4	1.84
5th Middlings flour	4.0	48.0	1.52	11.3	2.17
Tailings flour	23.1	26.5	2.84	12.8	2.56
Shorts	15.4	-	4.44	11.1	2.64
Bran	3.6	-	6.71	5.0	0.97

[a] Expressed on 14% moisture basis.

Table 7. Gross Composition[a] of Milled Products from Atlas Barley

Mill stream	Yield (%)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Free lipid (%)
Barley	100.0	-	1.87	7.7	1.47
1st Break flour	4.5	46.0	1.13	6.0	1.64
2nd Break flour	3.3	55.0	0.94	7.7	1.72
Grader flour	1.6	51.0	1.00	7.6	1.83
3rd Break flour	10.3	52.0	1.20	8.4	1.82
1st Middlings flour	5.3	63.5	0.73	6.7	1.41
1st Middlings reduction flour	1.7	55.5	0.77	7.0	1.33
2nd Middlings flour	11.9	62.0	0.75	7.5	1.62
3rd Middlings flour	8.1	48.5	1.18	8.3	1.91
4th Middlings flour	7.1	51.0	1.25	9.2	2.08
5th Middlings flour	4.3	45.0	1.39	10.0	2.36
Tailings flour	24.6	25.0	2.47	9.5	2.94
Shorts	11.9	-	4.02	7.2	2.61
Bran	5.4	-	5.45	2.3	0.66

[a] Expressed on 14% moisture basis.

the reduction system and are known as middlings flour. Tailings flour was separated from the mixture of break shorts, reduction shorts, and red dog milled on Alpine Pin mill; and the remaining was shorts. Bran is mainly the outer portion of barley kernel.

Several parameters were determined; they included yield, protein, ash, free lipid, bound lipid, and crude fiber. The results were shown in Fig. 4 and 5. Protein, ash, free lipid, and crude fiber generally increased in the latter fractions from reduction system. Tailings flour had the highest values of yield, protein, free, and bound lipid. Bran was richest in ash and crude fiber among those fractions.

Regression lines and correlation coefficients for the results from conventional roller milling are shown in Table 8. The data for tailings flour, bran, and shorts were not included in the calculations, except for calculations of the correlation between protein content determined by Kjeldahl method and transmission percentage by dye absorption method.

The ash content was used as an index of flour extraction. The protein content was positively correlated with ash content ($r = 0.733$). The flours from the low-protein Atlas barley had lower protein values (corresponding to ash values) than the other tested flour varieties. Also, the first break flour of all the samples had higher protein contents than expected from their ash values. When the correlation between ash and protein was calculated for samples without the first break flours, the correlation coefficient increased to $r = 0.891$.

The correlation between Agtron color and ash content was negative; the slope was curvilinear. When a relation between log Agtron color vs. ash was estimated, a negative correlation was obtained ($r = 0.633$). No Agtron color determinations were done on shorts and bran.

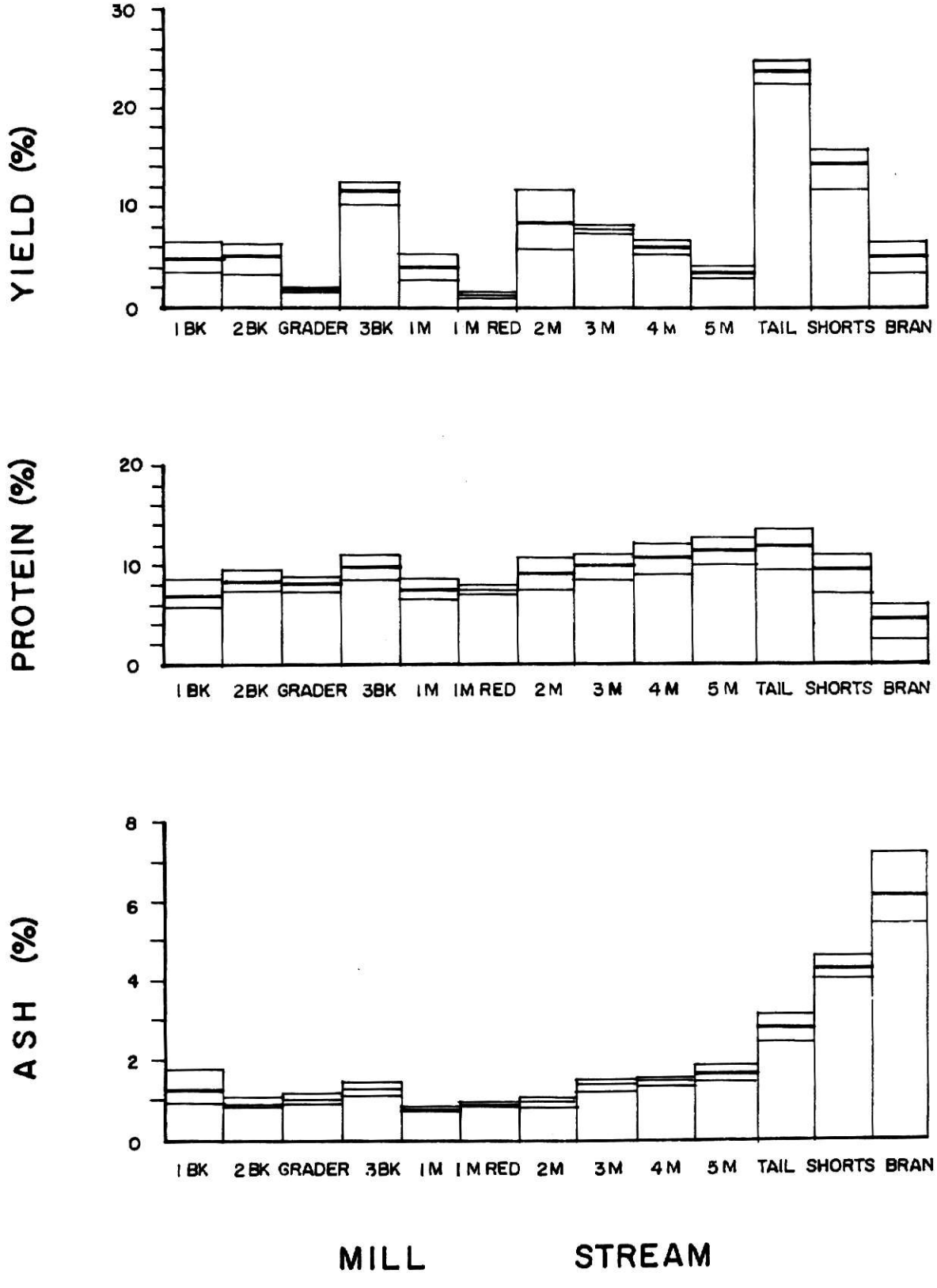

Fig. 4. Composition of mill streams from conventional roller milling. The heavy middle line in each stream represents the average value; the top and bottom lines the range for the corresponding stream in the five varieties studied.

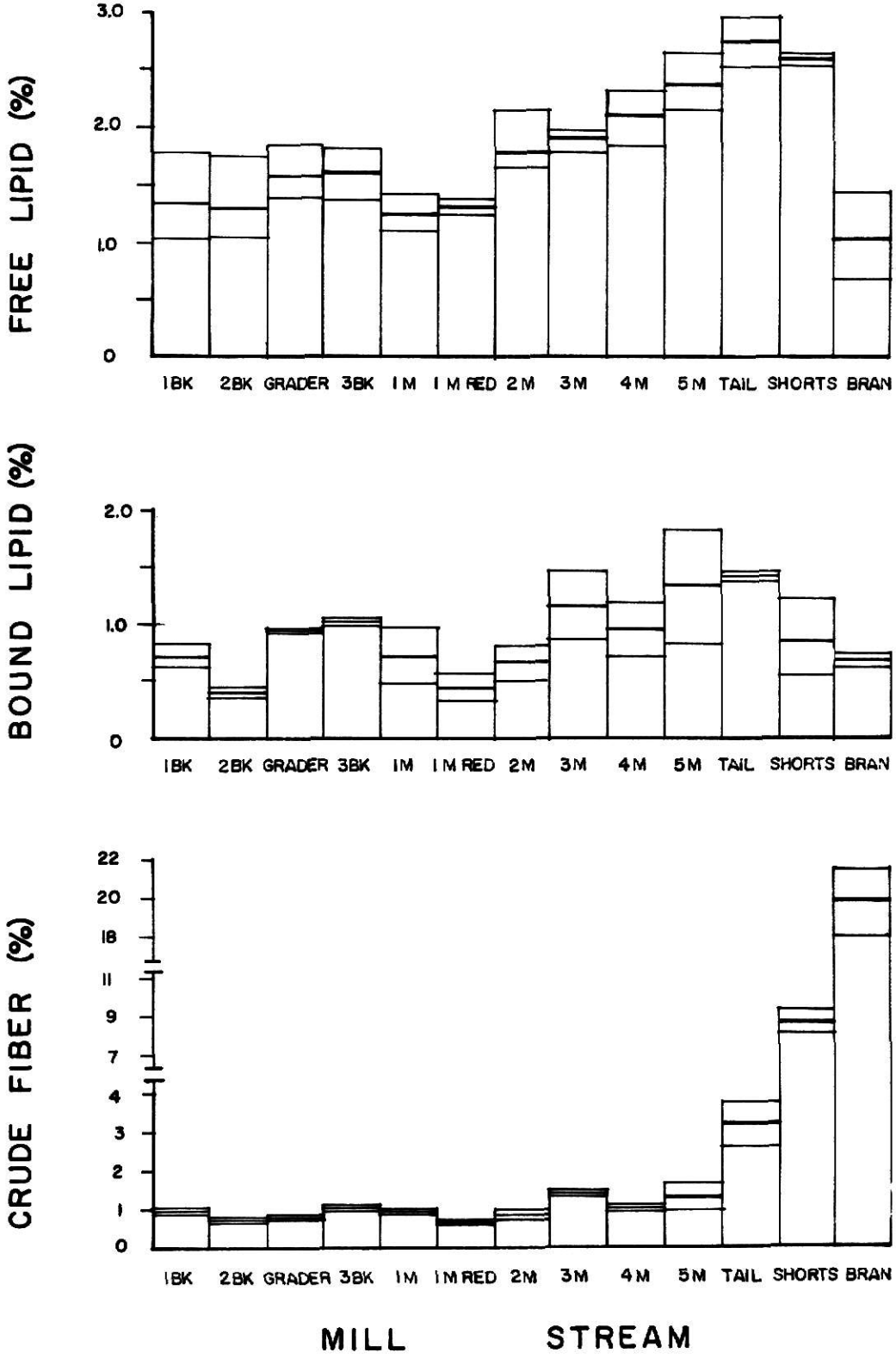

Fig. 5. Composition of mill streams from conventional roller milling. The heavy middle line in each stream represents the average value; the top and bottom lines the range for the corresponding stream in the five varieties studied.

Table 8. Regression Lines and Correlation Coefficients for Components in Barley Products from Conventional Roller Milling

Relation	Regression Line	Correlation Coefficient (r)
Protein vs. Ash		
All samples	Y = 4.18 + 4.23 X	0.733***
Samples without 1st break flour	Y = 3.94 + 4.86 X	0.891***
log Agtron color vs. Ash	Y = 1.91 - 0.17 X	-0.633***
Free lipids vs. Ash		
All samples	Y = 0.32 + 1.23 X	0.826***
Samples without 2nd middlings flour	Y = 0.17 + 1.35 X	0.854***
Crude fiber vs. Ash	Y = 0.27 + 0.60 X	0.689***
Dye-binding protein vs. Kjeldahl protein		
Below 8.7%	Y = 6.65 + 3.74 X	0.923***
Above 8.7%	Y = 11.60 X - 62.16	0.843***

Free lipid content was positively correlated with ash values. The correlation coefficient was r = 0.826. The flours from second middlings had above average free lipid contents. Calculating the data for all flour samples excluding second middlings flour increased the correlation coefficient to r = 0.854.

Ash contents increased as crude fiber contents increased; the correlation coefficient was r = 0.689. Data were calculated from milled flour fractions from two varieties: Larker and Paragon.

As expected, carbohydrate content (which was calculated by difference) increased as ash (or correlated with its protein, free lipids or crude fiber) contents decreased.

No significant relation was established between free lipids and bound lipids. With increase in extraction (as assessed by ash, Agtron color, or crude fiber determination), total lipid contents increased.

The results of thin-layer chromatography of barley lipids are illustrated in Fig. 6 and Fig. 7. Free lipids of whole barley (Fig. 7) which were fractionated by chloroform contained mostly nonpolar components, primarily triglycerides. The amount of polar components in free lipids was neglible and much smaller than in wheat (Chiu and Pomeranz, 1967). Bound lipids of whole barley (Fig. 6), which were extracted with water-saturated 1-butanol following petroleum-ether extraction, contained as in wheat mainly polar components and small amounts of nonpolar components. Unlike in wheat, glycolipids comprised a much smaller proportion than the phospholipids in bound lipids of barley.

Free nonpolar lipids in milling streams of barley were comprised mainly of triglycerides (as in free lipids of whole barley) and smaller amounts of hydrocarbons, sterol esters, diglycerides, monoglycerides, and fatty acids. No consistent differences in lipids extracted from milling streams were observed, except that tailings flour, shorts, and bran had less sterol esters, and more

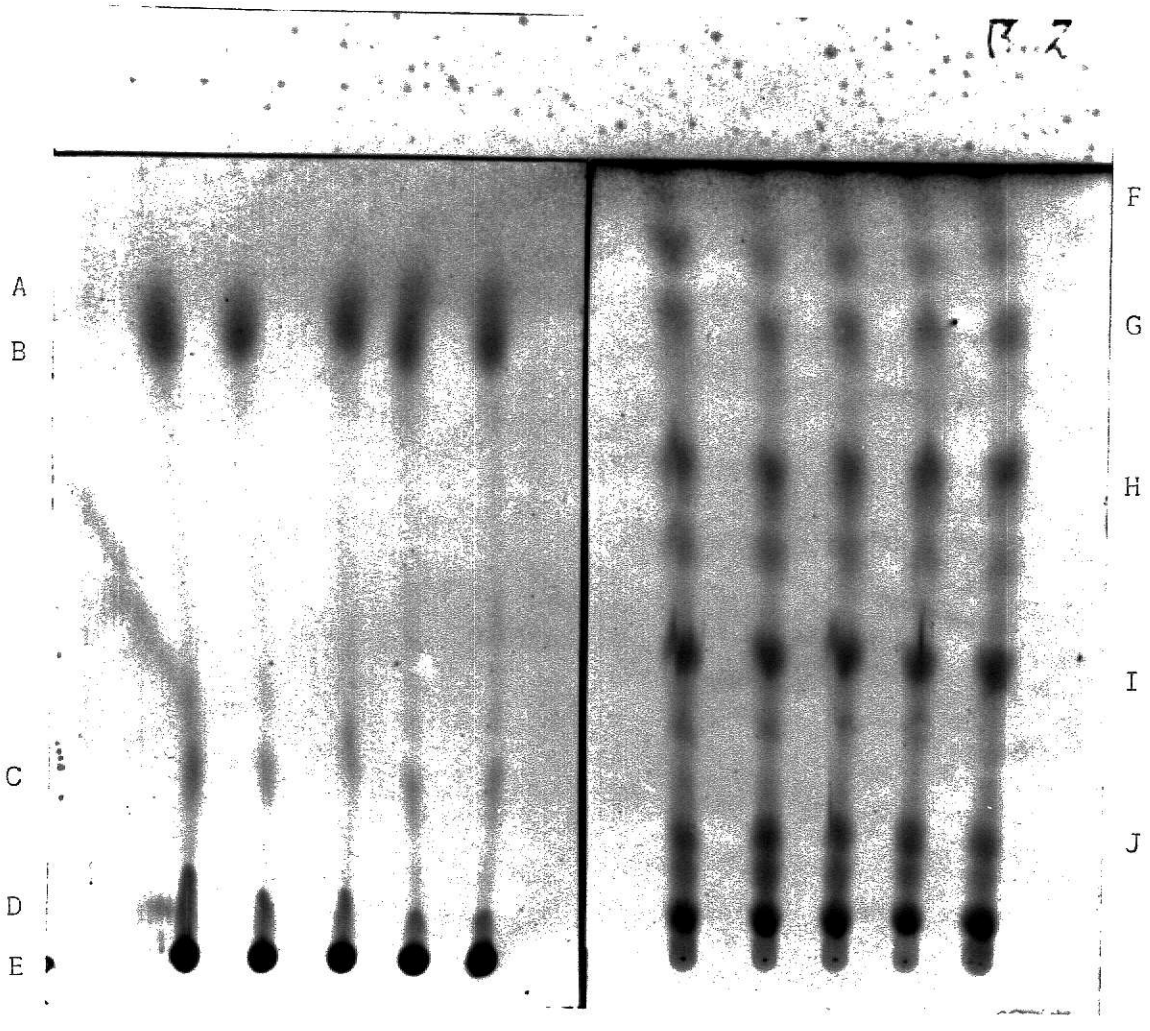

Fig. 6. TLC of 100 γ bound (water-saturated butanol following petroleum-ether) lipids from five barley varieties. From left to right: spots 1 and 6 from Primus, 2 and 7 Larker, 3 and 8 Paragon, 4 and 9 Betzes, and 5 and 10 Atlas. Spots 1 to 5 separated with chloroform, 6 to 10 with chloroform : methanol : water (65:25:4 by volume). Tentatively identified as : (A) sterol esters, (B) triglycerides, (C) diglycerides, (D) free fatty acids, (E) monoglycerides and unfractionated polar lipids, (F) unfractionated nonpolar lipids, (G) phosphatidyl ethanolamine and (H) digalactosyl diglyceride, (I) phosphatidyl choline and (J) phosphatidyl serine.

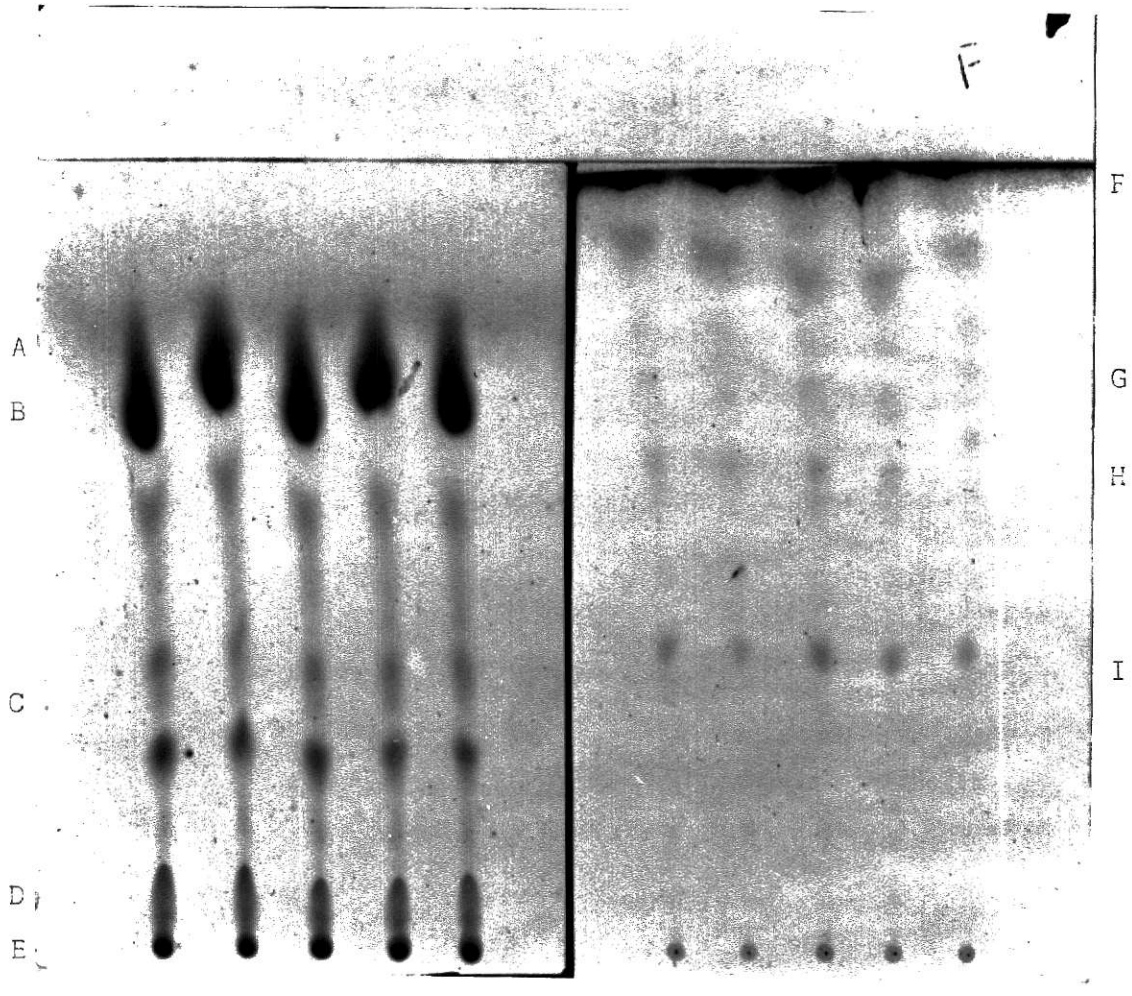

Fig. 7. TLC of 100 γ free (petroleum-ether extractable) lipids from five barley varieties. Lengend as in Fig. 6.

free fatty acids and diglycerides than the low-ash milling fractions. Free lipids in milling streams were low in polar components; the smallest amounts were in tailings flour, shorts, and bran. Bound lipids in bran and shorts were higher in triglycerides and lower in polar components than bound lipids in low-ash milling streams.

A comparison between protein determinations in all milling streams by the Kjeldahl and dye binding methods is given in Fig. 8. The data indicated that two groups of samples (with Kjeldahl protein content below and above 8.7%) had two regression lines. The correlation coefficient of the low-protein group was $r = 0.923$ and of the high-protein group was $r = 0.843$. This is in agreement with the results on wheat and milled wheat products reported by Banasik and Gilles (1962).

Some physical and chemical characteristics of air fractionated barley flours are summarized in Tables 9 and 10. In all five varieties, the protein-rich fraction B comprised over 5% of the total straight flour. Fraction B contained approximately two to three times as much protein as the original flour A. At the same time, two major fractions (D and E) were relatively low in protein. The residual flour EE and fraction C were consistently higher in protein than the original flour (A). A shift in protein contents was accompanied by shifts in ash and free lipids as shown in Fig. 9 and 10. The correlation coefficient between ash and protein for all air-separated samples was $r = 0.713$ and increased to 0.869 if EE fractions were not included in the calculations (table 11).

Protein content was positively correlated with free lipid content in all tested flours. The correlation coefficient was $r = 0.831$ and increased to 0.970 if EE samples were excluded. Free and bound lipids in air-fractionated

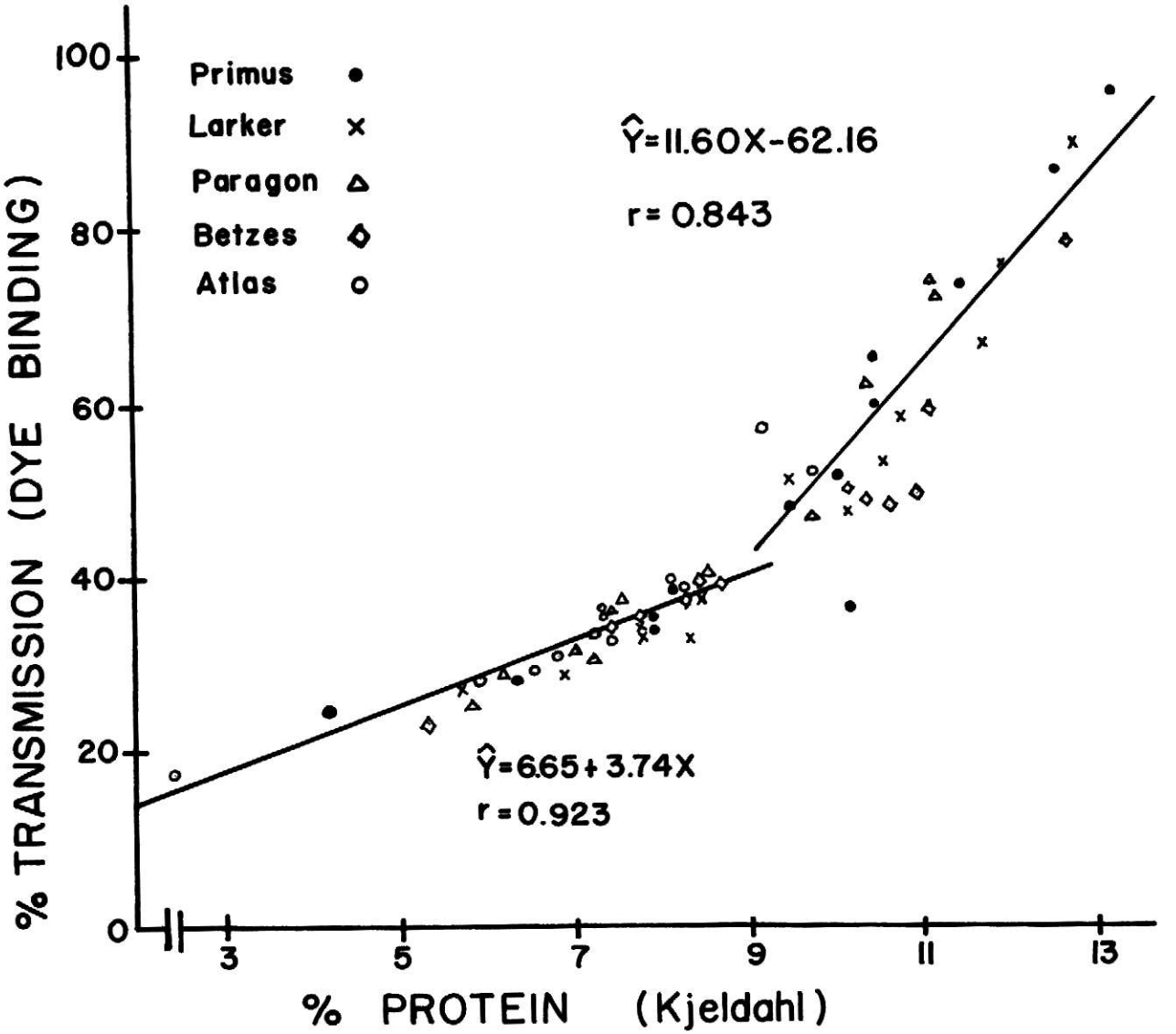

Fig. 8. Scatter diagram and regression lines for correlation between Kjeldahl protein and transmission by the dye-binding method of milled barleys.

Table 9. Some Physical and Chemical Characteristics [a] of Air-Fractionated Barley Flours

Variety and Fraction	Yield (%)	Fisher particle size (μ)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Lipids		Crude fiber (%)	Carbohydrates (by difference) (%)
						Free (%)	Bound (%)		
Larker									
A	100.0	9.0	46.0	1.39	10.7	1.83	1.13	1.29	70.8
B	5.3	2.8	45.0	2.50	24.0	3.35	2.13	0.45	55.7
C	17.2	5.2	47.0	1.70	15.8	2.13	1.66	0.73	65.6
D	28.3	10.5	54.5	0.84	6.5	1.04	0.83	0.75	76.9
E	23.8	11.7	46.5	0.85	6.7	1.14	0.67	0.94	76.4
EE	25.4	16.2	29.0	2.19	12.7	2.73	1.26	3.48	64.9
Paragon									
A	100.0	8.9	42.0	1.61	9.6	1.89	1.07	1.28	71.6
B	5.5	2.8	39.5	2.96	25.3	3.56	2.26	0.49	53.7
C	18.5	5.5	41.0	1.80	13.7	1.98	1.39	0.57	66.6
D	35.6	9.2	45.0	0.93	5.4	0.99	0.67	0.40	78.3
E	18.2	11.7	41.5	1.00	6.7	1.37	0.85	1.09	75.8
EE	22.2	16.4	--	2.74	11.7	3.13	1.46	4.31	64.1

[a] Expressed on a 14% moisture basis.

Table 10. Some Physical and Chemical Characteristics [a] of Air-Fractionated Barley Flours

Variety and Fraction	Yield (%)	Fisher particle size (μ)	Agtron color	Ash (%)	Protein (N x 6.25) (%)	Free lipid (%)
Primus						
A	100.0	8.6	46.0	1.48	10.6	2.01
B	5.2	2.7	45.0	2.72	27.8	3.72
C	18.7	5.6	43.5	1.67	14.8	2.09
D	34.7	10.4	49.0	0.81	5.9	1.03
E	17.7	10.6	44.0	0.87	6.7	1.22
EE	23.7	17.6	6.5	2.40	14.5	3.49
Betzes						
A	100.0	9.0	47.0	1.43	10.2	1.78
B	5.6	2.9	44.5	2.49	21.3	3.00
C	18.7	5.3	48.0	1.63	13.0	1.92
D	27.9	9.8	52.0	0.86	6.7	1.03
E	15.6	11.6	44.5	0.96	8.2	1.24
EE	32.2	16.8	28.5	1.80	11.0	2.22
Atlas						
A	100.0	9.7	53.0	1.22	8.1	1.99
B	5.3	2.9	44.0	2.22	19.8	3.11
C	18.7	6.0	45.0	1.30	9.9	1.90
D	27.9	9.5	50.0	0.76	5.0	1.17
E	14.4	11.5	47.5	0.74	5.6	1.38
EE	33.7	20.4	32.0	1.49	9.0	2.62

[a] Expressed on a 14% moisture basis.

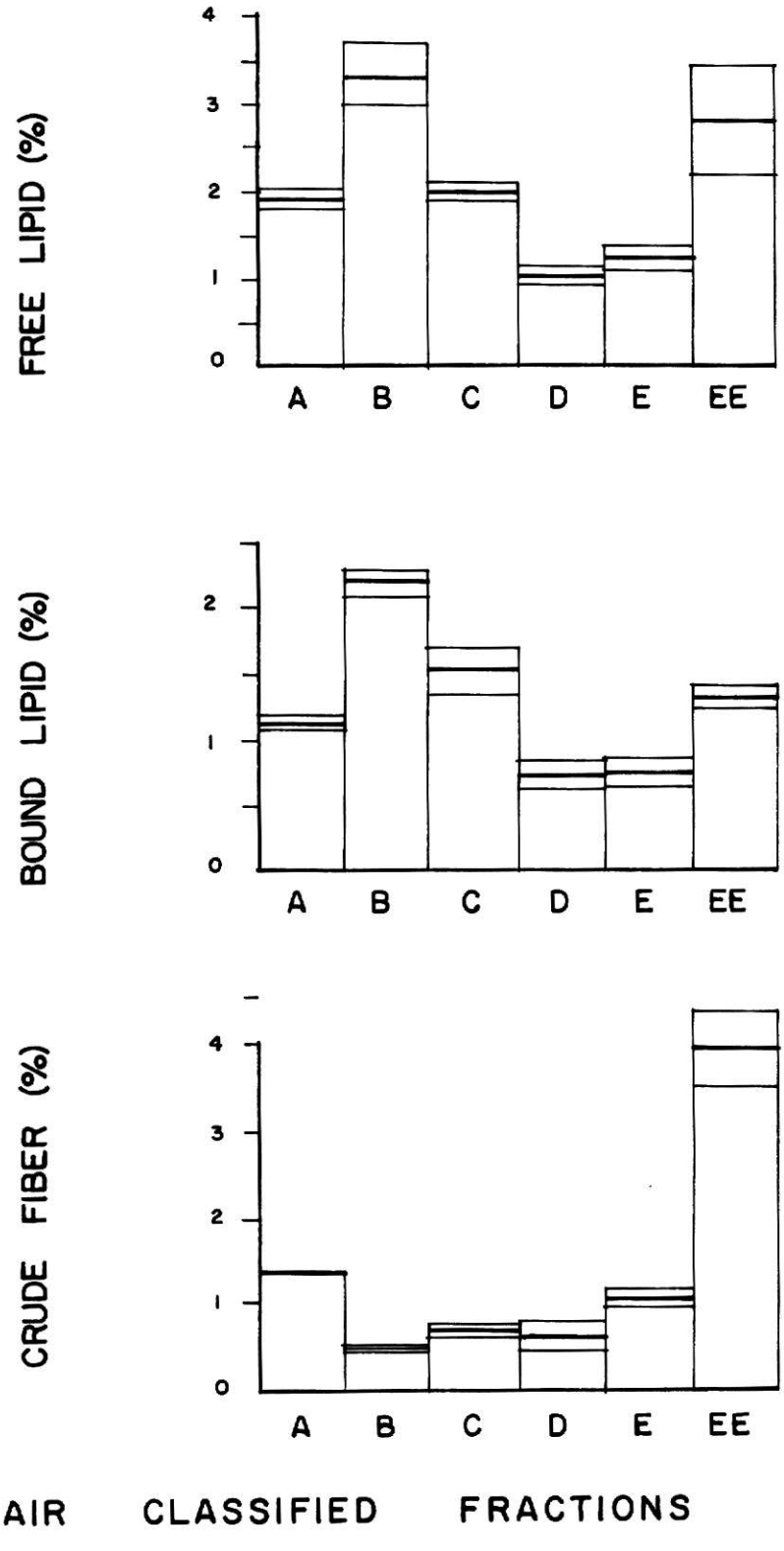

Fig. 9. Composition of air-classified flour fractions. The heavy middle line in each fraction represents the average value; the top and bottom lines the range for the corresponding fraction in the five varieties studied.

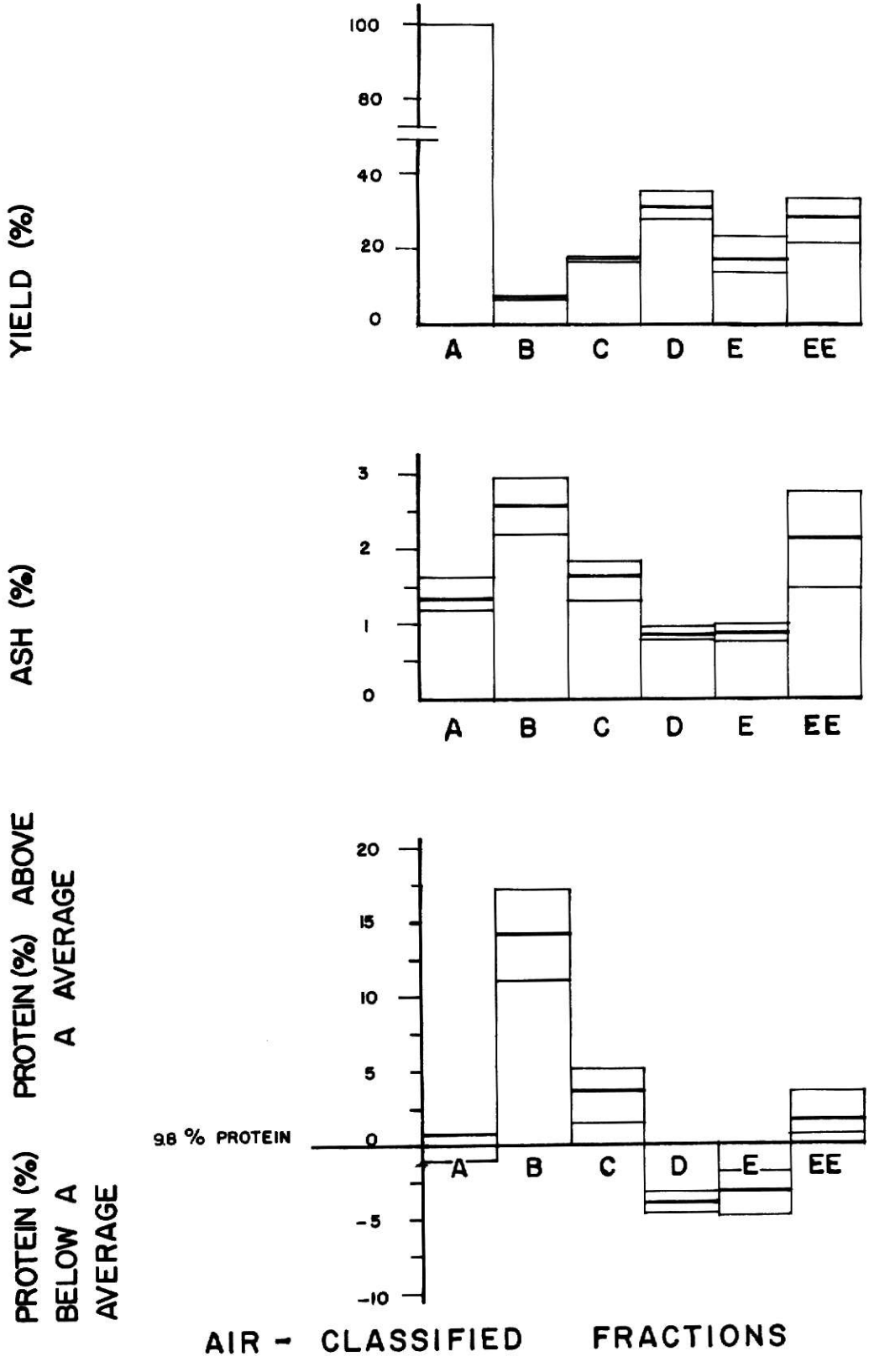

Fig. 10. Composition of air-classified flour fractions. The heavy middle line in each fraction represents the average value; the top and bottom lines the range for the corresponding fraction in the five varieties studied.

Table 11. Regression Lines and Correlation Coefficients for Components in Barley Products From Air Classification

Relation	Regression line	Correlation coefficient (r)
Ash vs. Protein		
All samples	Y = 0.63 + 0.08 X	0.713***
Samples without EE	Y = 0.40 + 0.09 X	0.868***
Free lipids vs. Protein		
All samples	Y = 0.71 + 0.11 X	0.831***
Samples without EE	Y = 0.49 + 0.12 X	0.970***
Bound lipid vs. Free lipid	Y = 0.10 + 1.56 X	0.907***
Crude fiber vs. Ash	Y = 0.21 + 0.65 X	0.390
Dye-binding protein vs. Kjeldahl protein		
All samples	Y = 8.16 + 1.16 X	0.816***
Samples with B fractions	Y = 3.12 + 0.65 X	0.899***

samples of two varieties were highly correlated (r = 0.907). The correlation between ash and crude fiber for samples of two varieties was statistically insignificant.

The correlation coefficient of Kjeldahl protein and transmission of dye binding method for all air-classified samples was r = 0.816. If the high-protein B fractions were not included, the correlation coefficient increased to 0.899 (Table 11).

Free and bound polar lipids in air-fractionated flours were similar in composition (as assessed by thin-layer chromatography) to nonpolar components in conventional roller-milled fractions (Figs. 11 and 12). In the bound polar components, fractions E and EE contained less (than other fractions) compounds with high R_f values (mainly digalactosyl diglycerides and phosphatidyl ethanolamine) and slightly more slow-moving components (presumably lysophosphatidyl choline and phosphatidyl serine).

The large shift during air fractionation in protein indicates new possibilities of utilizing milled barley products. The high-protein fraction would be a useful raw material in production of nutritional preparations low in carbohydrates but rich in protein, minerals, and lipids. The low-protein fractions with high carbohydrate contents could be particularly useful as an adjunct in the brewing industry or possibly in the manufacture of starch.

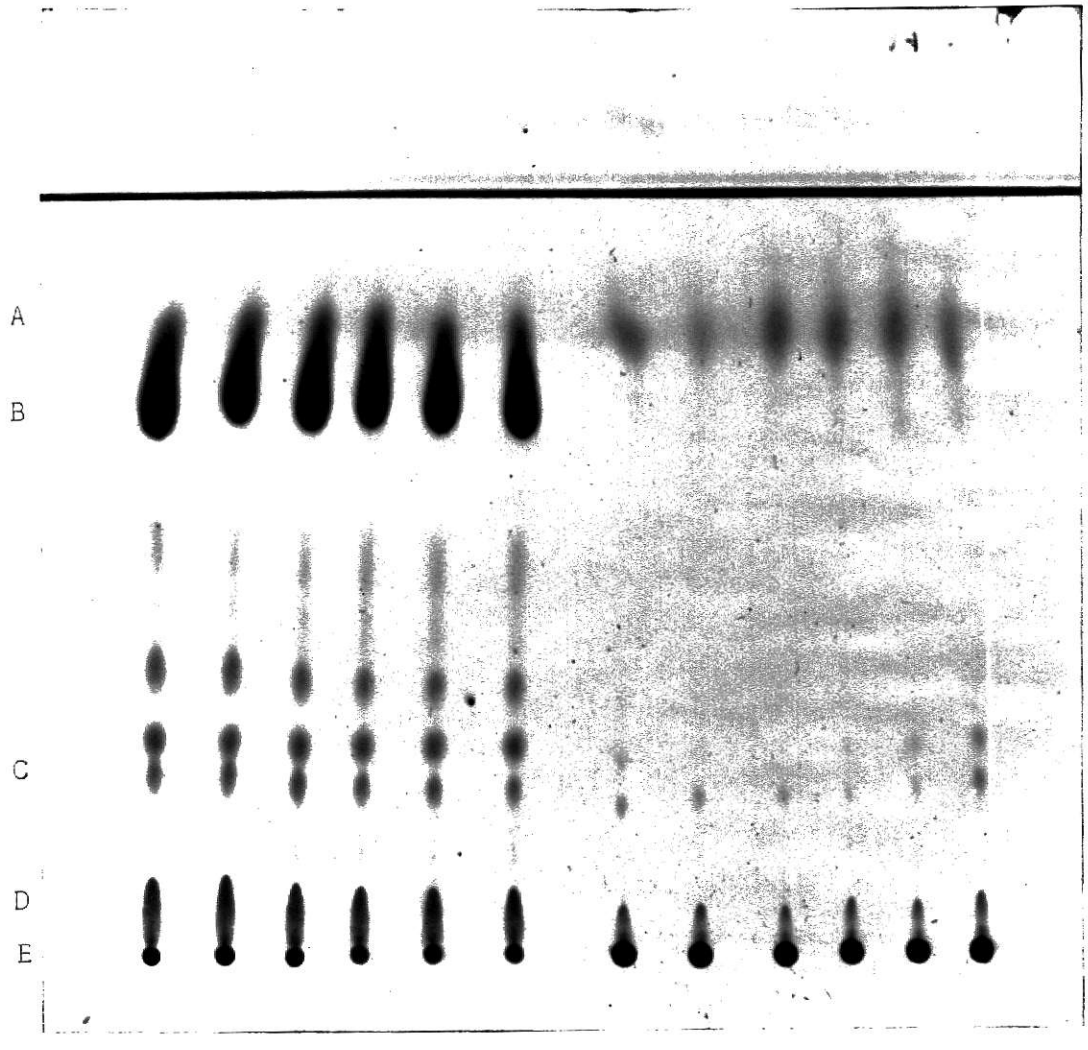

Fig. 11. TLC of 100 γ free and bound lipids extracted with butanol following petroleum-ether from air-classified flour fractions. Spots 1 and 7 from original flour fraction A, 2 and 8 fraction B, 3 and 9 fraction C, 4 and 10 fraction D, 5 and 11 fraction E, 6 and 12 residue flour fraction EE. Developed with chloroform. From left to right: spots 1 to 6 are free lipids, 7 to 12 are bound lipids, tentatively identified as in Fig. 6.

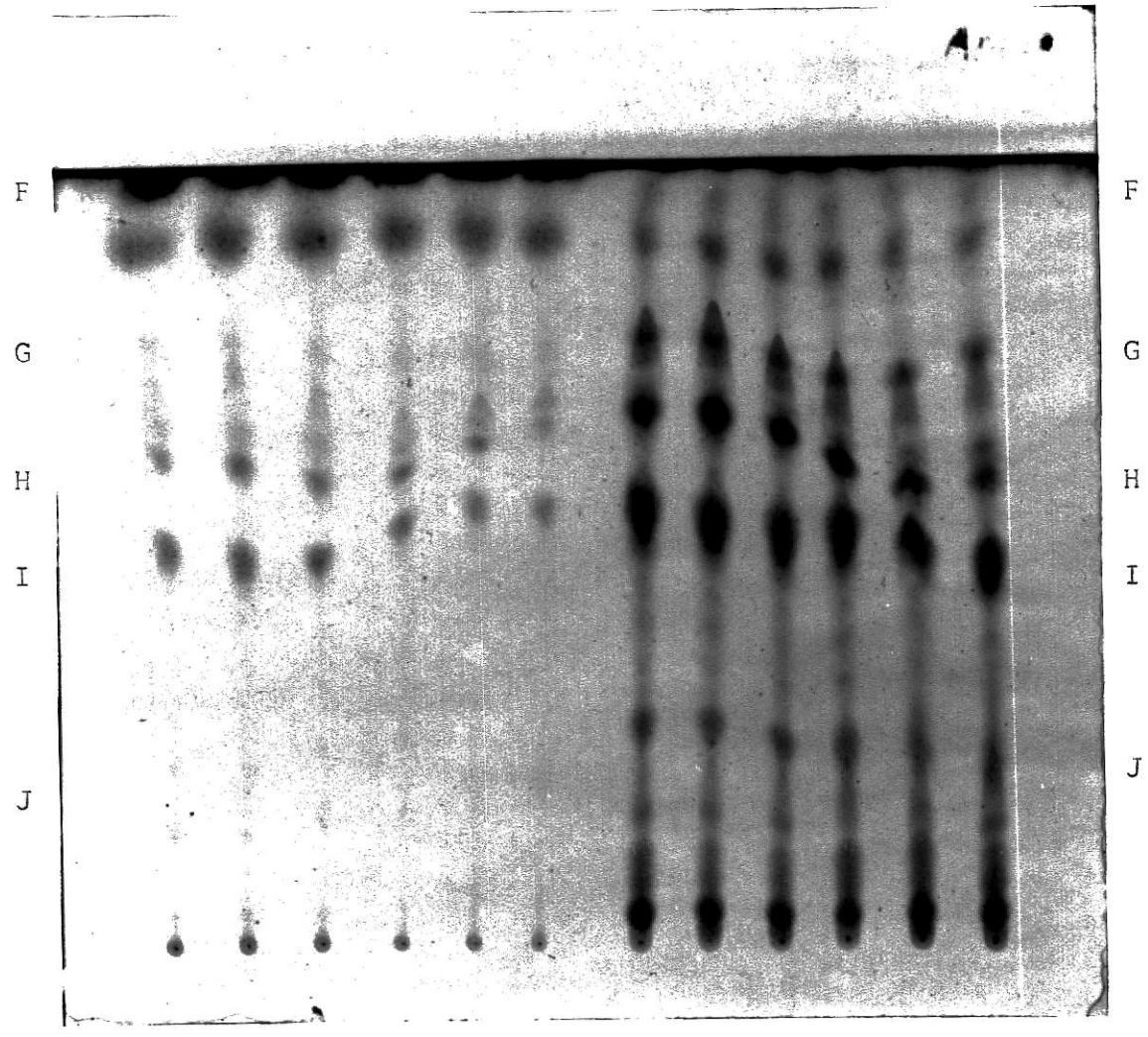

Fig. 12. TLC of 100 γ free and bound lipids extracted with butanol following petroleum-ether in samples described as in Fig. 11. Developed with chloroform : methanol : water (65:25:4 by volume) and tentatively identified as in Fig. 6.

SUMMARY

Five varieties of barley grown in four different locations (including four six-row malting varieties and one two-row malting variety) were used in this study. The barleys were milled by conventional roller milling, and mixed with tailings flour prepared by pin-milling on an Alpine Mill feed streams to make a straight 65%-extraction flour. The straight flour was separated by air-classification into five fractions varying in particle size and composition.

Analytical determinations of flour milling streams showed high correlations between ash and protein or free lipids; the correlation between ash and crude fiber was lower. The ash vs. log Agtron color was negatively correlated; and correlation between free and bound lipids was insignificant. Two methods were used to evaluate protein contents in milling fractions, the Kjeldahl and the dye binding procedures. A comparison between results obtained by the two methods showed two regression lines, for samples below and above 8.7% protein, respectively.

Thin-layer chromatography of free lipids from whole barley or from milling streams indicated predominately nonpolar components (mainly triglycerides). In bound lipids, the polar lipids comprised most of the components.

Air-classification yielded fractions (about 5% of total) that contained two to three times as much protein as the original flour. During air-classification, a protein shift was accompanied by ash and lipid shifts. Thin-layer chromatography showed the composition of free and bound nonpolar lipids was similar to that of lipids from roller milling streams.

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A STUDY ON THE COMPOSITION OF MILLED BARLEY PRODUCTS

by

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Barleys of five varieties grown in four different locations (including four six-row malting varieties and one two-row malting variety) were used. The barleys were milled by conventional roller milling, and mixed with tailings flour (prepared by pin milling feed streams on an Alpine Pin Mill) to make a straight 65% extraction flour. The straight flour was separated by air-classification into five fractions varying in particle size and composition.

Analytical determinations of flour milling streams showed high correlations between ash and protein or free lipids; the correlation between ash and crude fiber was lower. The ash vs. log Agtron color was negatively correlated; and correlation between free and bound lipids was insignificant. Two methods were used to evaluate protein contents in milling fractions, the Kjeldahl and the dye binding procedures. A comparison between results obtained by the two methods showed two regression lines for samples below and above 8.7% protein, respectively.

Thin-layer chromatography of free lipids in whole barley or in milling streams indicated predominately nonpolar components (mainly triglycerides). In bound lipids, the polar lipids comprised most of the components.

Air-classification yielded fractions (about 5% of total) that contained two to three times the protein content of the original flour. During air-classification, a protein shift was accompanied by ash and lipid shifts. Thin-layer chromatography showed the composition of free and bound nonpolar lipids was similar to that of lipids from roller milling streams.