PAPERS UPON INDUSTRIAL CHEMISTRY.

By Albert R. Leeds.

II. THE PHYSICAL AND CHEMICAL ANALYSIS OF FLOUR.

The great attention paid during five years past in the United States to the subject of the adulteration of foods, has already resulted in a voluminous literature. Unfortunately, much of this literature is crude and erroneous, and especially that referring to the topic of the present article. Originally appearing with specious pretenses of scientific merit in certain medical journals, these misstatements have gained credence with many reputable medical practitioners. Finally, having permeated downward into the columns of the daily press and into the popular mind, they have become so firmly lodged as to make their eradication difficult.

Foremost in this direction was a paper published in Gaillard's *Medical Journal*, Jan. 1882, with the following alarming title: "Highly important and extensively advertised cereal foods under the microscope. The genuine; the spurious; the worthless and the fraudulent. Therapeutic as well as dietetic facts of great value to physicians and their patients. By Ephraim Cutter, A.M., M.D., Harvard, etc." The only means of research employed by the author of this paper was the microscope, which he styles an "unerring teacher," "an infallable detector of fraudulent claims in regard to cereal foods," etc. The results of microscopic examinations are termed analyses, and as such are expressed in *figures denoting the relative percentages of gluten and starch*. The principal feature of the article, and the one most calculated to awe and convince the popular mind, was the woodcuts with which it was profusely illustrated, and which purported to be impartial representations of what might be seen of these foods when looked at through an objective, magnifying 800 diameters. Some of these cuts or slides represent flour entirely made up of unruptured gluten cells, while specimens of other flour, quite as remarkable, but for a different reason, are represented as composed of starch cells and fibrous tissue only. The two flours which are more especially singled out, the one for unstinted praise, the other for condemnation, are the Franklin Mills Entire Wheat Flour, and the Gluten Flour of the New York Health Food Co. Besides the woodcuts which represent the former as composed almost entirely of gluten cells, the excellence of the former flour incites the microscopist to add that,

"So long as the makers maintain such a proportion of gluten cells, they confer a blessing on mankind." On the other hand, the gluten flour is stigmatized as "a meal and not a flour. The circulars are travesties, and show an ignorance which, if it did not affect human life, would be ridiculous." In all this so-called gluten flour which the microscopist examined, he states that in repeated examinations only seventy gluten cells were found.

Chemical analyses show the falsity of these statements, as I will explain later on. But even without the aid of positive knowledge, founded on analytical data, these statements should have deceived no one accustomed to the use of the microscope, as is evident from the following considerations. In the first place, in making a chemical analysis of flour as much as ten grammes should be used. The analysis of so large a quantity affords a guarantee that the figures obtained represent the average composition of a mechanical mixture of the constituents of a non-homogeneous product like flour. But the weight of flour exhibited on a microscopic slide does not exceed the $\frac{1}{100000}$th part of the quantity taken for chemical analysis, and could not fairly represent an average. Neither can one determine by counting upon one slide or upon a hundred slides, the relative number of starch cells and *unruptured* gluten cells, what is the percentage of starch and gluten. It could not be done even were the gluten cells unruptured. But in the process of grinding they are largely broken and their contents commingled with the starch. When so commingled, it is difficult even with the aid of chemical reagents to discriminate between the albuminoid and amyloid constituents of flour. To estimate their percentage amounts by counting is impossible.

So different in appearance are the various parts of one slide, and so different are various slides prepared from one and the same flour, that the use of pictures of such slides to substantiate statements as to differences in composition in the flours examined, is misleading. Lest any microscopist should regard this protest as unnecessary, since it is but insisting upon a matter with which he is already familiar, let him call to mind how successfully the public is deceived by the claim that microscopic research necessarily implies research of great scientific exactitude. In the present instance certainly, authorities in medical science were led astray. Had they but reflected for a moment, that Dr. Cutter's assertion that many of the cereal foods examined contained no gluten whatsoever, was in itself a sufficient

proof of the falsity of his statements, insomuch that there is no flour but what necessarily contains some gluten, they would not have indorsed his publications without independent examinations. But in an address delivered before the New York Medical Society, Feb. 8th, 1882, its President, Dr. A. Jacobi, says of these microscopic examinations of Dr. Cutter : "I shall refer to his statements, desiring to give them the greatest possible publicity. I wish the brief article of his would be distributed in a hundred thousand copies, reprinted in every secular paper, read from every platform and pulpit of the land. For it is time that fraud should be stopped, and a nefarious trade suppressed."

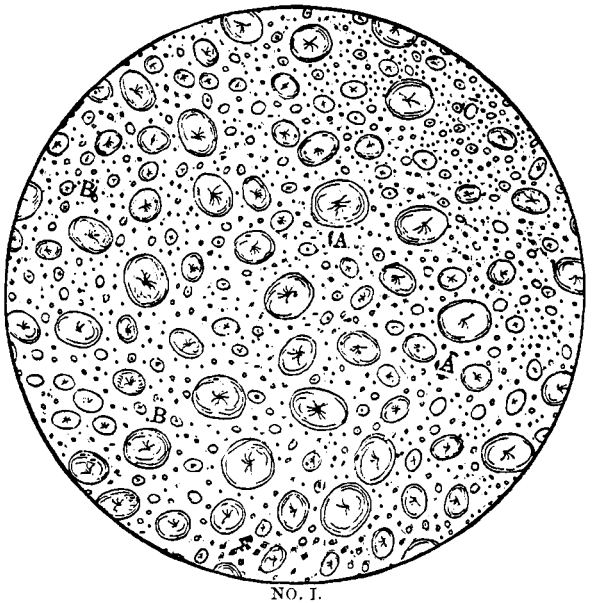
Then follows a résumé of Dr. Cutter's labors, in which without any assurance on the part of the presiding officer of a distinguished medical society, that he has submitted statements involving the reputation and business fortunes of more than two-score of the manufacturers of cereal foods in the United States to any independent critical research, he bestows indiscriminate and exaggerated commendation upon them all.

In a paper published in the Transactions of the College of Physicians of Philadelphia, III. series, VI., 377, I have given the results of the examinations of many of the foods referred to, the examinations being conducted with the aid of the microscope and mechanical manipulations, but controlled by chemical analysis. Without quoting in detail, I shall insert in this place only the table of analyses as showing how incorrect was the statement made by Dr. Cutter and approved by Dr. Jacobi, of the absence of gluten in Blair's Wheat Food, "Imperial Granum, Ridge's Food, Savory & Moore's Food, Farwell & Rhine's Gluten Flour, Hubbell's Prepared Wheat," and other cereal preparations.

ANALYSES OF HEALTH AND INFANT FOODS.

FOOD ANALYZED.	Water.	Fat.	Grape Sugar.	Cane Sugar.	Starch.	Soluble Carbohydrates.	Albuminoids.	Gum, Cellulose, etc.	Insoluble Residue.	Total.	Reaction.
Baby Sup, No. 1	5.54	1.28	2.20	11.70	61.99	14.35	9.75	7.09	....	100.00	Neutral.
Baby Sup, No. 2	11.48	0.62	2.44	2.48	51.95	22.79	7.92	5.24	....	100.00	Slightly alkaline.
Gerber & Co.'s Milk Food	6.78	2.21	6.06	30.50	38.48	44.76	9.56	....	....	101.79	Slightly acid.
Ridge's Food for Infants	9.23	0.63	2.40	2.20	77.96	5.19	9.24	....	....	102.25	Neutral.
Victor Baby Food	7.49	1.62	0.62	19.92	63.45	20.54	8.87	....	....	101.97	Slightly acid.
Anglo-Swiss Milk Food	6.54	2.72	23.29	21.40	34.55	46.43	10.26	....	....	100.50	Slightly acid.
Horlick's Food for Infants	3.39	0.08	34.99	12.45	none.	87.20	6.71	....	2.62	100.00	Slightly acid.
K. & M. Infants' Food	27.95	.....	36.75	7.58	none.	71.50	none.	....	0.55	100.00	Neutral.
Nestlé's Milk Food	4.72	1.91	6.02	32.93	40.16	44.88	8.23	....	0.08	100.00	Slightly alkaline.
Hawley's Liebig's Food	6.60	0.61	40.57	3.44	10.97	76.54	5.38	....	....	100.10	Slightly acid.
Hazard's Graham Farina	9.12	0.81	2.19	2.49	69.68	6.35	8.48	5.56	....	100.00	Neutral.
Cereal Milk	9.33	1.01	4.60	15.40	58.42	20.00	11.08	....	....	100.16	Slightly acid.
Mellin's Food	5.00	0.15	44.69	3.51	none.	85.44	5.95	....	3.46	100.00	Slightly alkaline.
Blair's Prepared Wheat Food	9.85	1.56	1.75	1.71	64.80	13.69	7.16	3.94	....	100.00	Neutral.
Savory & Moore's Infants' Food	8.34	0.40	20.41	9.08	36.36	44.83	9.63	0.44	....	100.00	Neutral.
Hubbell's Prepared Wheat Food	7.78	0.41	7.56	4.87	67.60	14.29	10.13	undet.	....	100.21	Neutral.
American-Swiss Milk Product Co.	5.68	6.81	5.78	36.43	30.85	45.35	10.54	0.77	....	100.00	Acid.
Wheat Flour for Hubbell's Wheat Food	9.02	1.01	2.34	2.46	76.07	5.66	6.40	....	....	.....	Neutral.
Imperial Granum	5.49	1.01	trace.	trace.	78.93	3.56	10.51	0.50	....	100.00	Neutral.
Robinson's Patent Barley	10.10	0.97	3.08	0.90	77.76	4.11	5.13	1.93	....	100.00	Neutral.
Farwell & Rhine's Gluten Flour	12.67	0.84	2.23	1.42	68.36	7.23	10.39	0.51	....	100.00	Neutral.

The microscopic drawings alluded to were made, it is stated, by Dr. A. T. Cuzner. Recently, in what purported to be a summary of certain information contained in a book entitled "What we Eat and What we Drink," this gentleman has published, under the title of "Food Analyses—Flour," a paper in the *Scientific-American* Supplement No. 414, in which statements are made similar to those advanced by Dr. Cutter, but nominally supported by proofs of quite different character. The fact that the microscope gives incorrect notions of the relative amounts of gluten and starch is admitted, and the attempt to make a quantitative analysis of flour by the use of this instrument is abandoned. At the same time the pictures of flour as seen under the microscope are quite different from flour as it actually appears when thus examined.

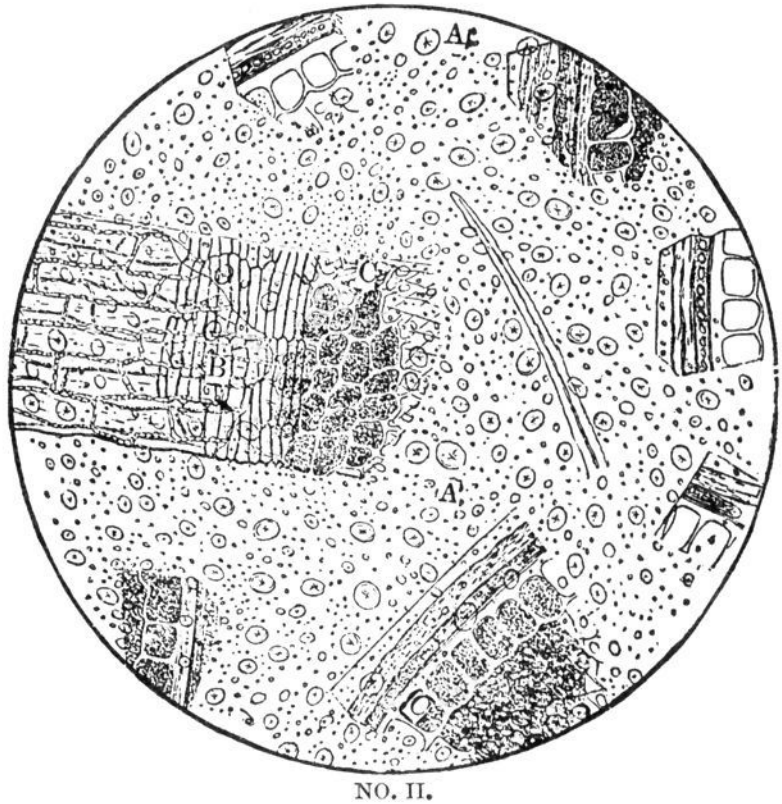


NO. I.

In the accompanying reproduction of the drawing made by Dr. Cuzner, No. 1—fig. and purporting to be a micrograph of the Pillsbury best flour, the large circles with rays or crosses at their centers, and marked A, are stated to be pictures of giant starch cells. B to stand for smaller starch cells, and C for granular gluten. Whether C is a mere dot or a very small circle without a dot in its center, is not clear from the drawing. But that the smaller starch cells are circles with dots in their centers, appears to have been certainly intended.

This drawing presumably represents the appearance in ordinary light, but in that light starch granules do not exhibit crosses, nor in any light crosses of the character depicted in the drawing. Moreover, gluten cannot be distinguished from starch in the manner stated.

The next photo-engraving No. II is a reproduction which (to quote the language of the original article) "represents the appearance of the Franklin Mills flour and equally well represents the Health Food Company's Flour, with the exception that in this flour is seen a *much larger proportion* of the bran coat with gluten cells attached, as well as unattached, together with some *hairs of wheat*, than is found in the Franklin Mills flour. In the figure, A, are seen giant starch cells; B, portion of bran coat; C, portion of bran coat with gluten cells unruptured."



NO. II.

The microscopist will be struck by the remarkable fact that this cut, which is entitled "Health food gluten flour as seen under the microscope," represents a number of thin sections of the wheat-berry. Most of these sections are transverse, and represent very beautifully the epicarp, endocarp, testa and secundine of the berry, precisely as they appear when viewed in their natural positions to one another. The largest section is a diagram illustrating the relative position of the three outer coats of the berry as viewed from without. It is a matter of nicety to prepare such excellent sections

of the berry, when it is sliced with a knife, and in the crushed fragments of the berry that remain after it has been ground into flour I had never seen anything of the character above figured. But, thinking other observers might have been more fortunate, I looked through the literature of the subject, and found precisely the same diagrams as those figured by Dr. Cuzner. In the latter case they appear in company with giant starch cells (granules?), whose magnitude is comparable with the unruptured gluten cells. In the former, they appear as illustrations of thin sections of the berry, Fig. 3 in the above cut No. III showing "the relative position of the

Fig. 3.

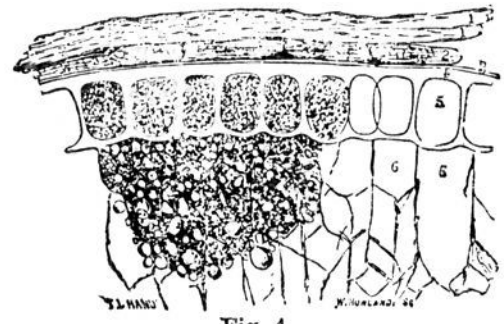


Fig. 4.

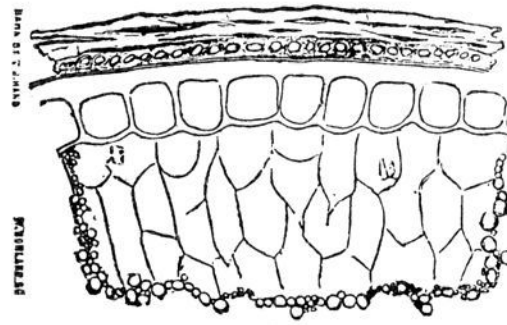


Fig. 5.

NO. III.

several layers of the investing coats of the berry, as seen from without; Fig. 4, as viewed in a section transverse to the greater length of the berry; Fig. 5, as presented in longitudinal section." In the latter case, as reproduced by Dr. Cuzner, they are portions of what he saw when Health Food Gluten Flour was examined under the microscope; in the former, they form the cut which can be found on page 4, Report on Vienna Bread, printed in 1875 at the Government Printing Office in Washington, this report having been written by E. N. Horsford, U. S. Commissioner to the Vienna International Exhibition, 1873. I have had this last cut, No. III, reproduced by photo-engraving to compare with Dr. Cuzner's No. II.

It is needless further to insist upon the worthlessness of mere microscopic inspection in determining the relative composition of flour. But after the erroneous nature of the results arrived at by Dr. Cutter by means of microscopic inspection only had been pointed out, one of his critics, Prof. J. G. Richardson, in an article entiled "A Serious Microscopic Blunder" (Phila. Med. News, June, 1882), called attention to the fact that he or any other physician, without the employment of the more exact methods of chemical analysis, could obtain sufficiently approximate results by simple manipulation of flour, to prevent them from being led astray in the matter. He says:

"Dr. Cutter asserts that the opaque, oval or rounded cells (constituting the fourth coat of the wheat grain, according to Prof. Parkes) afford most of the gluten, and hence on their presence the chief strength of the food depends." He therefore declares that a large number (fourteen) of the food-stuffs he examined, and found under his microscope to display none of these so-called "gluten-cells," "contain no gluten" (page 9), and broadly intimates that they are consequently frauds upon the public. But the fact is, these so-called "gluten cells" (denominated by Payen, *oleiferes*) probably include in their substance starch, phosphates, fatty matters and coloring materials, containing only parts, perhaps, but a small part, less than one-seventh, of the gluten which exists in wheat. Thus, Peligot, as a mean of fourteen analyses, gives the percentage of gluten in flour (whence "gluten-cells" are removed) at 12.8, while in bran (containing nearly all the "gluten cells") it is only 10.84, and other observers confirm his statements. If my friend, Dr. Cutter, or any of his disciples, would like to satisfy himself that he has made a lamentable mistake in this matter, let him take say ten grams of one of the fine flours he asserts "contain no gluten," mix it with water into a dough, let it stand for half an hour, and then stir it in a porcelain capsule, with successive portions of water, until the starch is washed away, and the adhesive fibrillated gluten is left nearly pure, in the proportion, after drying, of from seven to twelve per cent. (*Vide* Parkes' Practical Hygiene, fifth edition, 1878, p. 224.) The small starch-corpuscles and granules, left by this process entangled among the threads of gluten, can be beautifully differentiated by adding a drop of iodine solution, which affords the usual deep-blue reaction with the starch, but dyes the gluten filaments of a yellowish-brown tint."

The chemist will recognize in the above extract from Parkes' Hygiene, the result of the investigation of v. Bibra, Millon, Rivot, Ritthausen and others upon the gluten contained in cereals. The method elaborated by these chemists appears to have been adopted after the above suggestions, by Dr. Cuzner, but unfortunately with a modification which robs it at the same time of its simplicity and its value. For instead of manipulating the gluten-dough in a very small flow of water directly between the fingers (which is best), Dr. Cuzner proposes to tie it up in a muslin bag and manipulate this in a jar of water until all the starch is washed out.

His language is: "Having an occasion to make an analysis of certain flours as to the relative amount of starch and gluten they each contained, I thought that the process adopted, being simple and one easily carried out by persons of ordinary intelligence, it would interest your readers and give them the means of ascertaining for themselves the food value of any flour they might at any time be using as food in their families."

Then follows more in detail the process, which is as follows: "A certain portion (2,000 gr.) of each of these flours was mixed with water, separately from the rest, and inclosed in a piece of muslin, as we inclose a pudding. This inclosed dough was then kneaded in a certain amount of water in order to separate the starch from the rest of the flour. During this kneading process, water readily passed through the cloth to the dough, and back again to the remainder of the water, carrying with it on its return the starch cells, albumen and sugar. By continuing this kneading process, the starch, sugar, albumen and gum were finally separated from the gluten, which remained a soft, tenacious, elastic substance, insoluble in water, inside the cloth. The gluten was then removed from the inside of the cloth, moulded, dried, and weighed. The water containing the starch, gum, albumen, and sugar, was placed in a vessel and allowed to stand for some hours, in order that time might be allowed for the starch-cells to settle to the bottom.

At the end of this time the water was poured off and the starch moulded into a cake, dried, and weighed. In the examination of the Franklin Mills' and Health Food Co.'s flour, an additional process was required. During the kneading process, described above, the fine bran with adherent gluten cells was forced through the cloth and became mixed with the starch-cells in the water. This

water had to be filtered through very fine lawn muslin. The starch-cells readily passed through this cloth, but the bran was detained on the muslin, and afterward collected, dried, and weighed. As the purpose of this analysis was not to ascertain the amount of albumen, gum, and sugar contained in the flours, but rather the amount of gluten and starch, the examination was continued no further. But if the reader should desire to ascertain how much albumen, gum, and sugar a certain amount of flour contains, the following process may be adopted. Take the water poured off from the settled starch, and boil it. This will coagulate the contained albumen, which can be collected on a filter, dried, and weighed. The water that passes through the filter can afterwards be evaporated over boiling water, and the gum and sugar collected, dried, and weighed."

An entirely original and quite surprising use of pictorial illustration in connection with analysis is now given. Three rectangular blocks are depicted with sharp angles and perfectly plane sides as representing the dried gluten obtained from the three flours. They are all equally symmetrical, and distinguishable only by the fact that the Franklin Mills gluten cake is large, the Health Food Co. gluten small, and the Pillsbury gluten cake of intermediate size. The striking angularity and symmetry of these gluten blocks which appear as if cut out of steel, is puzzling. Crude gluten, as I have encountered it, is an extremely tough substance of a leather-like consistency, which, on drying, is puffed out by the imprisoned moisture in globular and more or less fantastic shapes.

Three equally symmetrical blocks also in all respects similar, except in size, represent the starch. They have a monumental character, and more closely represent grave-stones than anything in the nature of starch. Difficult as the task would be of moulding crude gluten into rectangular blocks, yet it would be easy of execution compared with that of compacting starch granules into similar masses—an achievement thus far to me quite inexplicable. Two more solid blocks represent bran, that from the Franklin Mills flour small, that from the Health Food Co. large. How bran which has been separated from both starch and gluten can be built up, compacted, dried and weighed in coherent rectangular blocks is not explained.

The author then gives his results, which I have reduced to per

cents. and supplemented with the percentages unaccounted for.

	Bran.	Gluten.	Starch.	Unaccounted for.
Pillsbury Best Flour....		13.25 p. c.	80 p. c.	6.75 p. c.
Franklin Mills Flour..	2.5 p. c.	15.75 "	50 "	31.75 "
Health Food Co. Flour.	7.5 "	7.00 "	45 "	41.5 "

The absurdity of styling that an analysis, in which 30–40 per cent. of the constituents of the flour are to be put down as albumen, gum, sugar, moisture, salts, etc., is manifest.

The process was then tried on some "Pillsbury Best" flour and some Health Food Co. Whole Wheat Flour. 150 grames of each flour were taken, made into a dough, and inclosed in pieces of muslin cloth. These pieces of muslin cloth were previously washed, dried and weighed. They were then kneaded in water until the washings were no longer milky, an operation requiring many hours, when the wash-waters amounting in one case to eight, in the other to ten liters, were collected and allowed to stand. At the end of a week the precipitation of starch was still incomplete, the supernatant liquid appearing milky. The liquid was then syphoned off, great care being requisite to prevent disturbance in the easily moved starch granules at the bottom. How to get rid of the last portions of water, without losing some of the starch, and in case this were successfully accomplished, how the starch could be detached from the vessels and moulded into cakes without loss, these difficulties I saw no way of overcoming. Instead the starch was filtered off upon tared filters, and after drying at 110° in the ordinary manner, was weighed.

The gluten was detached as far as was practicable from the muslin cloth, and after drying the latter, the weight of the gluten which could not be detached was added to the weight of the main portion after drying at 110°. Great care must be exercised to obtain constant weights on drying, owing to the slowness with which the gluten gives up its last portions of moisture. If so many hours and such large amounts of water were requisite to wash the starch through ordinary muslin, the further separation of all this starch from bran by passing it a second time in a state of suspension in water through very fine lawn muslin, appeared impracticable.

The total soluble matter in the filtrates from the starch was determined, and also the crude gluten in the same. This was neces-

sary inasmuch as some of the gluten remained behind, and some passed through the muslin. Microscopic examination showed that the so-called starch contained cellular tissue and gluten, the so-called gluten contained cellulose and starch. Weighings performed on impure products of this kind of course have no real value.

PHYSICAL ANALYSES BY WASHING IN BAGS.

	Pillsbury Best.	Health Food Flour.
Starch	69.25 per cent.	52.92 per cent.
Gluten remaining in bag	3.40 "	21.41 "
Gluten in wash-waters	8.56 "	5.94 "
Soluble matters in wash-water	4.94 "	4.96 "
Water in flour	11.10 "	11.32 "
Total found	97.25 "	96.55 "
Unaccounted for	2.75 "	3.45 "

CHEMICAL ANALYSES OF SAME SPECIMENS OF FLOUR.

Starch	67.86 per cent.	65.19 per cent.
Soluble Alb.	2.84 "	2.30 "
Insoluble Alb.	8.62 "	11.21 "
Total Alb.	11.45 "	13.51 "
Sugar	2.83 "	2.67 "
Gum	5.02 "	3.84 "
Fat	1.31 "	1.63 "
Cellulose	0.81 "	2.35 "
Saline, etc	0.42 "	1.38 "
Phosphoric Acid	0.17 "	0.39 "
Water	11.10 "	11.32 "
Total	100.81	101.89 "

The result of the latter analysis coming out differently from what I anticipated, former analyses having shown that the Health Food Flour contained much the largest amount of albuminoids of any of the many samples of flour analyzed, determinations were made of two more samples of the same flour. They agree better with the former figures, although still falling short in percentage of albuminoids of the results obtained on other samples.

	Lab. No. 1,129.	Lab. No. 1,130.
Starch	58.67	58.35
Gum	2.53	undet.
Sugar	5.39	"
Soluble Albuminoids	2.35	"
Insoluble "	12.16	"
Total "	14.51	13.74
Ash	1.30	1.58
Phosphoric Acid	0.37	0.35
Cellulose	4.06	2.47

During the course of the chemical analyses detailed above, trial was made of the various methods for the analysis of flour heretofore proposed. Attempts were made to substitute direct for indirect determination of several constituents, and at the same time to effect a gain in rapidity of working and in accuracy of results. These attempts have been only in part successful. And inasmuch as the difficulties to be overcome can be best explained in connection with the trials of previous methods, the results of these trials will be stated first.

A. Cairns' Method (Quantitative Analysis, p. 255).

" Digest 5 grms. of the flour in 100 c. c. cold water for one or two hours, with frequent stirring, filter through a filter previously exhausted with hydrochloric acid, washed, dried and weighed, wash with about 100 c. c. cold water. The solution contains: (1) *albumen*, (2) *gum*, (3) *sugar* and a portion of the soluble salts. The residue contains : (4) *celluloses* (5) *starch*, *gluten* and *fat*."

" *Solution.*—1. Boil and then filter ; the precipitate consists of albumen. Dry at 100°, and weigh.

The treatment with water, filtration and precipitation of albumen should be completed on the same day. By keeping the solution hot it may be continued through two days, but this is not advisable."

[NOTE.—These are tedious operations, and of questionable accuracy. Granting that the solution of gum, soluble albumen and sugar is perfect, nevertheless, complete washing by this method is troublesome to effect.]

The precipitation of the albumen on boiling so dilute a solution is also imperfect. In an actual trial, even after concentrating the solution to one-half and boiling, only 37 per cent. of the soluble albumen was precipitated. On evaporating to 40 c. c. an additional precipitate of 52 per cent. was obtained, and to 15 c. c. a third precipitate of .4 per cent. It is not only necessary to collect these precipitates of albumen on weighed filters, but to boil down in tared beaker glasses, because the coagulated albumen attaches itself to the sides of the beaker and cannot be perfectly detached. These 3 determinations of soluble albumen required therefore 9 weighings in all, and the final result was incorrect, falling short of the correct result by 7 per cent. of the amount actually present.

The albumen so obtained should be ignited, and its amount of ash deducted.

"2. Evaporate the filtrate from the albumen nearly to dryness, add a large excess of alcohol, warm and then allow it to cool, filter on a weighed filter; wash with alcohol. Dry at 100° C, and weigh the gum thus obtained."

[NOTE.—The gum thus precipitated carries down with it some saline matters, and it should be ignited and the weight of ash deducted.]

"3. Evaporate the alcoholic filtrate from the gum to small bulk, add water, and boil out the alcohol. Concentrate the solution to 50 c. c. and divide into halves. In the first half determine the dextrose directly by copper sulphate solution. In the second half add a few drops of dilute sulphuric acid, boil, neutralize with potassium hydrate and determine dextrose as before. The excess of dextrose found in the second solution is due to cane sugar."

"*Residue.*—Wash with a jet from the wash-bottle into a beaker. Then dry the filter with what adheres to it and weigh. This weight, less that of the filter found at the beginning, gives the weight of adhering substance, which must be taken into account in the subsequent determinations."

[NOTE.—Although the amount left behind is small—in a trial only 0.0815 grm. out of an original weight of 5.3005 grm.—yet this filtration is very tedious and requires two additional weighings.]

"4. Add to the substance in the beaker 50 times its weight of water, containing one per cent. of sulphuric acid, and heat for several hours, until the starch goes into solution, and only light flocculent cellulose is left. Filter and wash until all sulphuric acid is removed, dry at 100° and weigh."

[NOTE.—All the albuminoids and starch are not carried into solution by this method, and the weight of cellulose so obtained much exceeds the true amount. The excess, as determined in one trial, was 5 times. The so-called cellulose when submitted to chemical treatment under the microscope was found to have unruptured cells, containing starch granules and undissolved albuminoid bodies.

"5. To the filtrate from the cellulose, diluted to 400c.c., add about 30c.c. concentrated sulphuric acid, and heat on a water-bath at about 95° for several hours, adding water from time to time to keep it up to the original bulk. Digest this until a drop of the solution shows no coloration when heated with diluted iodine solution, and also gives no precipitate with alcohol. When the conversion of the starch into dextrose is complete, neutralize the excess of acid by sodium or potassium hydrate, and determine the glucose with copper sulphate as before."

[NOTE.—It has been shown by Allihn (J. pr. Chem. xxii,50), and by Salomon that the conversion effected by means of sulphuric acid is only partial, the former authority stating that under the conditions which usually prevail in starch analyses only 95 per cent. of the starch is converted into dextrose. In an experiment in which the results obtained with sulphuric acid were compared with those obtained by means of hydrochloric acid, when used according to Sachse's method (Chem. Centr. 1877, 732), to be given later, I obtained a less discrepant result. Sulphuric acid yielded 98 per cent. of the amount as determined by hydrochloric acid.]

"The starch can also be determined in a separate portion, by washing a weighed quantity with water, then with ether, and again with water, drying and then making an elementary analysis for carbon. The carbon found is from both starch and cellulose. Deduct the carbon due to cellulose found as above (formula, $C_{12}H_{10}O_{10}$ the same as that of starch), and calculate the rest to starch (44 parts carbon = 100 parts starch)."

[NOTE.—This method is tedious, difficult of execution, and inaccurate. After washing with water and ether, and drying as directed, a tough horny cake was left in the filter paper, consisting, in addition to the starch and cellulose, of the insoluble gluten, etc. The physical qualities of this mass were such that it could not be detached from the filter. Even had it been possible to do so, the percentage of one ingredient (the starch) could not have been estimated from a carbon determination, when besides the cellulose, a mixture of at least four substances, such as are present in varying amounts in crude gluten, were undetermined.]

"*Albuminoids.*—Determine the total nitrogen in 1 grm. by combustion with soda-lime, and from this calculate the albuminoids; 15.5 parts N = 100 parts albumenoids. From this deduct the albumen found as above; the difference is gluten."

[NOTE.—It has already been shown that the percentage of albumen found as above, is only a fraction of the amount actually present. The ratio of 15.5 : 100, gives for the nitrogen multiplier 6.45. This is certainly too high. Ritthausen concluded from the results of his elaborate analyses of the wheat albuminoids, that the correct multiplier is 6. Others place it at 6.33, and this number has been employed in making the calculations contained in the present article, except where otherwise stated.]

"*Fat.*—Weigh out 2 or 3 grms. treat with ether, boiling it gently over the water-bath, decant the ether through a filter into a weighed dish, repeat this two or three times, evaporate off the ether, and weigh the fat."

[NOTE.—This method of fat extraction gives results much too low. As thus determined, the Pillsbury flour contained only 1.02 per cent. of fat. When the treatment with ether was continued until no more fat could be extracted, the sample of flour (undried) yielded 1.35 per cent. of fat; dried, it yielded 1.31 per cent. These latter results are in accordance with those stated by König (Nahrungsmittel, p. 559), who recommends that the extraction with ether should be performed on the dried substance rather than on the undried, the latter method usually yielding the higher results. This he attributes, not so much to a decomposition and alteration of the fat, but to a solution in water-holding ether of substances other than fat—such as resin, wax, chlorophyll, etc.]

"*Ash.*—Burn 40 or 50 grms. of the flour in a weighed dish. If there is any difficulty in burning off the carbon, cool and weigh the dish and contents; then extract with hot water, filter through a small filter, avoiding any transfer of the carbonaceous substance to the filter. Dry the dish, and weigh again. The loss represents mineral salts dissolved out. Moisten with nitric acid, add the filter-paper and contents, burn again, cool, and weigh. The weight, less that of the dish, represents the remainder of the ash. The weight of the ash of the small filter-paper may be ignored. The ash may be dissolved in water with a little nitric acid, and analyzed as required."

[NOTE.—This amount of flour appears enormously greater than needed, except it be desired to make an extended analysis of the ash. In an actual trial 22.9435 grm. flour was taken, and burned in a platinum capsule, with a small flame placed somewhat to one side of the dish. The platinum was raised to a red heat only, and contact of a current of heated air with the carbonized mass was favored by this disposition of the flame. At the end of 8 hours I was surprised to find that the ash was burned perfectly, no carbon remaining.]

An accurate method is given by König (Nahrungs- und Genuss-mittel, p. 316 & 561). Ten to twenty grammes of the flour is burned, at first over a small flame, until a black coal remains, and no more smoke is given off. Then the flame is removed, the coal pulverized, and allowed to stand for some hours in the air. On now strongly igniting, a very white ash is quickly obtained, or, if necessary, the exposure to air and ignition may be repeated. This gives the raw ash, which may contain coal, sand, and carbonic anhydride, and is not to be set down as the pure ash or "ash."

To correct for carbonic anhydride, the entire raw ash (from 10 to 20 grms. of substance) is dissolved by hydrochloric acid in a CO_2 apparatus, and its amount subtracted.

The solution so obtained is filtered upon a dried tared filter, repeatedly washed with hot HCl, then with hot Na_2CO_3 and NaHO, in order to remove separated silica, then again with hot water, dried at 100 to 110°, and weighed. This residue of coal and sand is then to be deducted from the raw ash.

König says truly that CO_2 in the ash of pure cereals is too inconsiderable to necessitate a correction for it. If the ash does evolve notable CO_2, it indicates an addition of chalk, magnesite, etc.

In nice questions, involving falsifications of the flour, the above method is advisable. But for ordinary determinations a very simple and rapid method, which is given later, will be found accurate.

(*To be continued.*)