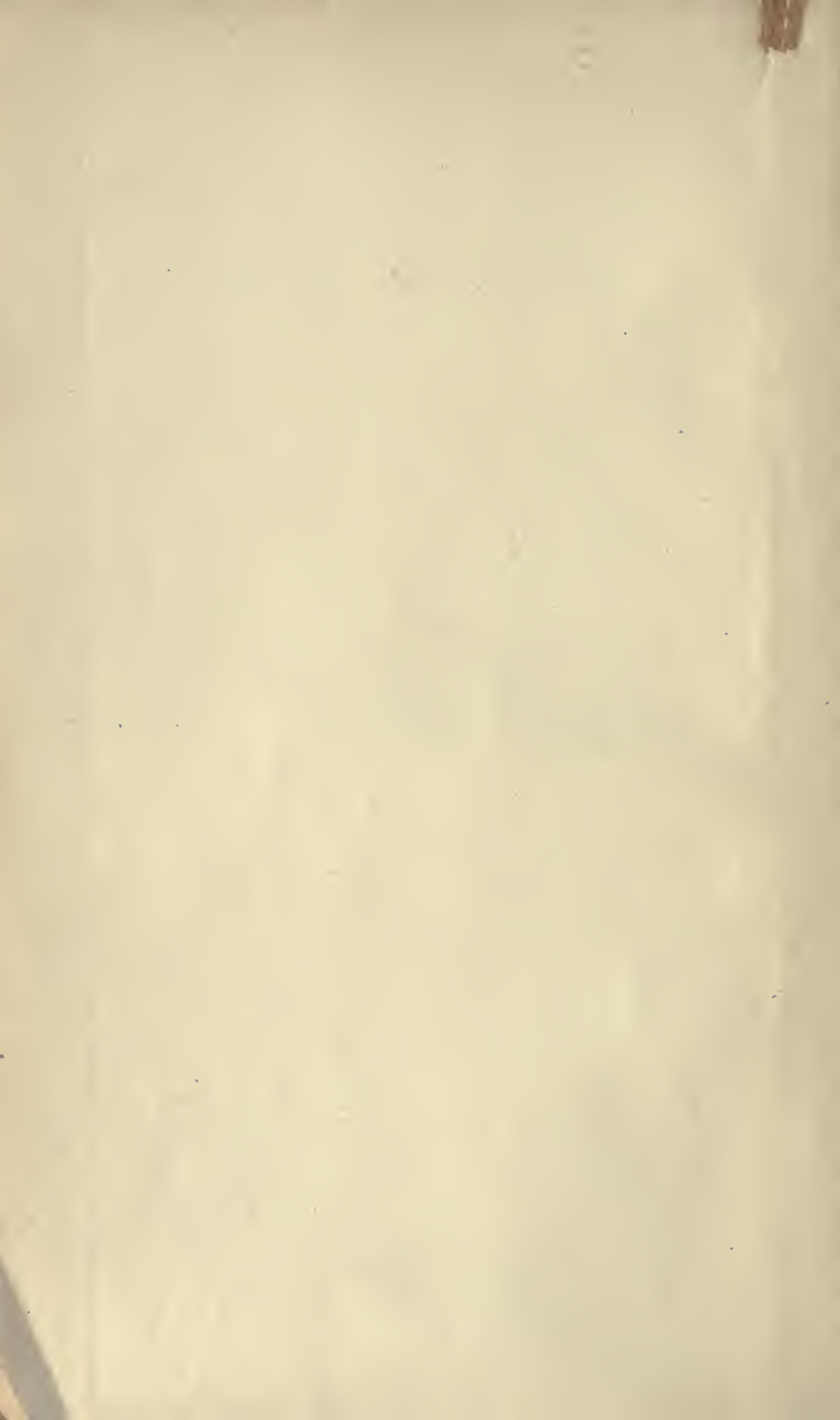




U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

INVESTIGATIONS MADE UNDER DIRECTION OF

H. W. WILEY,

CHIEF CHEMIST.

PART SEVENTH.

TEA, COFFEE, AND COCOA PREPARATIONS.

BY

GUILFORD L. SPENCER, Assistant Chemist,

WITH THE COLLABORATION OF MR. ERVIN E. EWELL.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

WASHINGTON:

GOVERNMENT PRINTING OFFICE.

1892.

AGRICULTURAL
LIBRARY,
UNIVERSITY
—OF—
CALIFORNIA

U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

INVESTIGATIONS MADE UNDER DIRECTION OF

H. W. WILEY,

CHIEF CHEMIST.

PART SEVENTH.

TEA, COFFEE, AND COCOA PREPARATIONS.

BY

GUILFORD L. SPENCER, Assistant Chemist,

WITH THE COLLABORATION OF MR. ERVIN E. EWELL.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

WASHINGTON:

GOVERNMENT PRINTING OFFICE,

1892.

TABLE OF CONTENTS.

	Page.
Letter of transmittal.....	V
Letter of submittal	VII
Tea.....	875
Statistics of tea consumption.....	875
General classification	875
Methods of manufacture	876
Black teas	876
Green tea	878
Adulteration—definition	879
Adulteration—methods.....	880
Detection of facing.....	881
Spent or exhausted leaves	882
Foreign leaves	883
Foreign astringents	885
Added mineral matter.....	885
Lie tea	886
General remarks on tea adulterants.....	886
General statements concerning the constituents of teas.....	887
Analytical methods	889
General remarks to analysts.....	892
Report of the examination of teas bought in the open market	892
Conclusion.....	898
Coffee	899
Statistics of consumption.....	899
General statements.....	900
Chemical composition.....	901
Methods of analysis	907
Adulteration—definition	908
Adulterants and their detection.....	909
Substitutes for coffee	914
Imitation coffees	915
Detection of imitation coffees	920
Report of examination of samples bought in the open market	921
Roasted coffees.....	926
Green coffees.....	927
Ground coffees	927
Extracts.....	929
Substitutes	932
Conclusion	932
Cocoa preparations	933
Nature, source, commercial importance, etc., of the cocoa bean	933
Chemical constituents	935
The commercial preparations of the cocoa bean, their nature, methods of preparation, and adulteration	941
Microscopical examination	946

	Page.
Cocoa preparations—Continued.	
Methods of analyses reported by various chemists.....	949
Summary of results obtained by various chemists.....	959
Analyses of cocoa beans made by various chemists.....	960
Analyses of the husks from cocoa beans reported by various chemists..	962
Analyses of commercial preparations of cocoa beans reported by various chemists.....	963
Ash analyses reported by various chemists.....	969
Results of the examination of cocoa preparations in the laboratory of the U. S. Department of Agriculture.....	970
Description of samples of cocoa preparations examined in the laboratory of the U. S. Department of Agriculture.....	971
Analyses of cocoa preparations made in the laboratory of the U. S. Department of Agriculture.....	980
Discussion of results obtained.....	987
Appendix A.—Bibliography of the literature of tea, coffee, and cocoa preparations.....	991
Appendix B.—United States tea adulteration law.....	1011
Appendix C.—Abstracts from the Italian law relating to the adulteration of foods.....	1013
List of leaves, illustrated by Plates XXXIX–XL.....	1014

LIST OF ILLUSTRATIONS.

	Page.
Plate XXXIX. Genuine tea leaves and possible adulterants.....	1014
XL. Genuine tea leaves and possible adulterants.....	1014
XLI. Tea leaf, upper and lower surface of epidermis.....	1014
XLII. Stone cell of tea leaf; seed coat of coffee.....	1014
XLIII. Raw coffee; wheat bran, outer layer.....	1014
XLIV. Wheat bran, second and gluten layers.....	1014
XLV. Roasted chicory: parenchyma and milk vessels; pitted cells.....	1014
XLVI. Cocoa, epidermis of husk; cocoa husks, thick walled cells..	1014
XLVII. Cocoa husk, thin membrane; section of cotyledon of cocoa..	1014

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., March 1, 1892.

SIR: I beg to transmit herewith for your inspection and approval the manuscript of Part Seventh of Bulletin No 13, relating to the adulteration of coffee, tea, and chocolate.

Respectfully,

H. W. WILEY,
Chemist.

Hon. J. M. RUSK,
Secretary of Agriculture.

LETTER OF SUBMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., February 29, 1892.

SIR: I herewith submit to you the report which has been prepared by Mr. E. E. Ewell and myself upon investigations of tea, coffee, and cocoa preparations. That part of the report and bibliographic work upon cocoa preparations is entirely due to Mr. Ewell, who has also assisted in the general analytical work. I also acknowledge the assistance of Messrs. McElroy, Trescott, Krug, and Sanborn.

This report has been unavoidably and materially delayed in its preparation, but this time has not been lost, since much of the foreign and other work on artificial coffees has been accomplished since it was begun.

Respectfully,

G. L. SPENCER,
Assistant Chemist.

Dr. H. W. WILEY,
Chief Chemist.



FOODS AND FOOD ADULTERANTS.

PART VII.—TEA, COFFEE, AND COCOA PREPARATIONS.

TEA.

Tea is prepared from the various species of *Thea*. This plant is a hardy evergreen shrub.

STATISTICS OF TEA CONSUMPTION.

The extent of the consumption of teas is shown by the following statements obtained from the United States Treasury:

Imports of tea less the amount exported.

1890.	1889.	1888.	1887.
<i>Pounds.</i>	<i>Pounds.</i>	<i>Pounds.</i>	<i>Pounds.</i>
83,494,956	79,192,253	83,944,547	87,481,186

The present per capita consumption of tea in the United States is approximately 1.33 pounds.

We derive about 51 per cent of our tea from China, 42 per cent from Japan, and all but a small fraction of a per cent of the remainder from the British possessions.

GENERAL CLASSIFICATION.

Teas are usually designated as black or green, the name depending upon the color of the prepared leaf, due to the methods of curing and not, as formerly supposed, to differences in species.

Col. Money,¹ in his work on tea cultivation, gives an illustration, from which the following is taken, indicating the leaves which constitute

¹ Tea cultivation, 3d ed., page 102 *et seq.*, Lieut. Col. Money; London, W. B. Whittingham & Co.

each of the different kinds of tea as classified according to the age of the leaf.



FIG. 27.—*a*, Flowery Pekoe; *b*, Orange Pekoe; *c*, Pekoe; *d*, Souchong 1st; *e*, Souchong 2nd; *f*, Congou.—*a*, *b* (when mixed together), Pekoe; *a*, *b*, *c*, *d*, *e* (when mixed together), Pekoe Souchong.

If there be another leaf below *f*, and it be taken, it is named and would make Bohea.

Each of these leaves was first a flowery Pekoe leaf (*a*), it then became *b*, then *c*, and so on.

At the base of the leaves *c*, *d*, *e*, *f*, exist buds 1, 2, 3, 4, from which new shoots spring.

METHODS OF MANUFACTURE.

The methods of preparing teas differ in the different countries in which this commodity is grown. In India the manufacturing processes are very much simplified, and the greater portion, if not all the work, is accomplished by machinery; thus the leaves only come in contact with the hands of the laborers in picking.

BLACK TEAS.

¹ The methods of the manufacture of black teas in Japan is essentially as follows:

The leaves are withered by exposure to the sun, fire being used only

¹ Abstracted from Bulletin No. 7, Imperial College of Agriculture, Tokyo, Komaba, Japan. Y. Kozai.

in cloudy or rainy weather. An hour's sunning is usually sufficient. After withering, the leaves are rolled and twisted. Black teas are usually rolled in an apparatus made especially for this purpose. The rolled leaves are now subjected to a fermentation. This is a very important operation since its influence on the quality of the tea is considerable. During this operation the leaves lose their disagreeable raw odor and acquire a fine flavor and the desired tint. One method of fermenting the leaves is to make them up into small balls, which are placed in shallow bamboo trays, covered with a white cloth, and set aside in a sunny place. A second method is to spread the leaves in a tray, press them together, cover and place them in a sunny place as above. The progress of the fermentation is determined by the appearance of the leaves, the full time required being about an hour. After fermentation the leaves are exposed in a thin layer to the sun. During this sunning the green color of the leaves gradually changes to a black. The next process is termed the "firing." The leaves are placed in a tray over a charcoal fire. The temperature gradually increases as the moisture is driven off. The leaves are constantly turned to insure uniform drying. The leaves are transferred to another tray carefully mixed, and the drying repeated until they are dry. The final operation consists in passing the leaves through sieves of different meshes and packing. The tea is divided into three classes depending upon the size of the leaves, viz, Pekoe (the leaf buds), Souchong, and Bohea.

As already stated, these manipulations are very much simplified in India. According to Col. Money¹ the operations practiced in the older tea countries have been reduced from twelve to five. Col. Money states that the brokers in India judge of the quality of the teas by the following examination:

They judge from three things, (1) the tea (*i. e.*, the prepared leaf), (2) the liquor, (3) the out-turn.²

The tea.—The color should be black, but not a dead black, rather a grayish black with a gloss on it. No red leaf should be mixed with it, it should be all one color. The tea should be regular: that is, each leaf should be about the same length, and should have a uniform twist, in all but "broken teas." (These latter are called "broken," because the leaf is more or less open and broken.) The tea should also be regular of its kind, that is, if Pekoe all Pekoe, if Congou all Congou; for any stray leaves in a tea, even if of a better class, will reduce its value. In the high class of teas, viz, Pekoe and broken Pekoes, the more Pekoe tips that are present the higher, in consequence, will its price be.

The liquor.—In taste this should be strong, rasping, and pungent, in case of Pekoes a "Pekoe flavor." There are other words used in the trade to particularize certain tastes, but the words themselves would teach nothing. Tea-tasting can not be learned from books. If the liquor is well flavored, as a rule, the darker it is in the cup the better. But to judge of teas by the color of the liquor alone is impossible, for some high-class teas have naturally a very pale liquor.

¹ Tea cultivation, Lieut. Col. Edward Money. W. B. Whittingham & Co., London.

² The out-turn consists of the tea leaves after infusion.

The out-turn.—A good out-turn is generally indicative of a good tea. It should be all, or nearly all, one color. No black (burned) leaves should appear in it. A greenish tinge in some of the leaves is not objectionable, and is generally indicative of pungent liquor, but the prevailing color should be that of a bright new penny.

GREEN TEA.

The leaves are first steamed until they lose their elasticity. The operations of rolling and drying follow the steaming. The leaves are finally sorted by means of a series of sieves. In China, instead of steaming the leaves as practiced in Japan, they are heated in a pan over a charcoal fire.

The Japanese sometimes prepare a tea which they term "flat tea." In this tea the leaves, as the name indicates, are not rolled. They are obtained from plants which are kept in darkness for a week or two before picking; keeping the plants in darkness is said to produce a fine aroma.

The operations of tea manufacture require considerable skill and experience. At certain stages of the work an error may ruin the quality of the product.

In general the quality of a tea depends upon the age of the leaf, also upon the time of picking. The leaves gathered after the first and second picking (as high as twenty-five pickings are made in India) gradually become tougher and less juicy as the season advances.

The India teas are usually very much stronger than those from China and Japan, one part giving an infusion as strong as three parts of that from the latter countries. To those persons who have acquired a taste for the Chinese and Japanese teas, those from India at first seem too strong and the flavor is not as agreeable as would be expected from the high price of these teas. It is an excellent plan to add a certain proportion of India tea to the Chinese or Japanese product, the strength and usually the flavor of the latter being considerably improved by this mixture.

As has been stated the leaves of the India teas only come in contact with the hands of the workmen at the time of picking; this may also be said to a certain extent in regard to the Japanese teas, whereas those of Chinese origin are manipulated almost entirely by hand, and even the feet are sometimes used in rolling some of the cheaper grades.

The black teas have grown in favor to such an extent in England that now but a small proportion of the teas consumed are green. This is largely due to the supposition that the black teas contain less astringent matter and also act to a less extent upon the nerves. The following analyses from the valuable bulletin of Mr. Y. Kozai (*loc. cit.*, p. 24) show in parallel columns the percentage composition of black and green teas made from the same leaves. In this experiment Mr. Kozai took special precautions in sampling the leaves in order that the teas might have identically the same composition provided the chemical

changes were the same and of the same extent. These analyses are of especial value on account of Mr. Kozai's knowledge of tea manufacture and his residence in a tea-producing country. The percentages are referred to the dry matter.

	Original leaves. ¹	Green tea.	Black tea.
Crude protein..... Per cent..	37.33	37.43	38.90
Crude fiber..... do	10.44	10.06	10.07
Ethereal extract..... do	6.49	5.52	5.82
Other nitrogen-free extract..... do	27.86	31.43	35.39
Ash..... do	4.97	4.92	4.93
Theine..... do	3.30	3.20	3.30
Tannin ² do	12.91	10.64	4.89
Soluble in hot water..... do	50.97	53.74	47.23
Total nitrogen..... do	5.97	5.99	6.22
Albuminoid nitrogen..... do	4.11	3.94	4.11
Theine nitrogen..... do	0.96	0.93	0.96
Amido-nitrogen..... do	0.91	1.13	1.16

¹ Portion of original sample of leaves dried at 85° C.

² Calculated as gallotanic acid.

This table shows the marked difference between the composition of the green and the black teas. In the black tea there is a very considerable increase in the "other nitrogen-free extract." This increase has evidently taken place at the expense of the tannin, a part of which has been decomposed during the fermentation. This reduction in the tannin is probably one of the reasons why black is considered more wholesome than green tea by the English, but the change in the tannin alone will not account for this preference. There are not sufficient data concerning the "other nitrogen-free extract" and other constituents for a further discussion of this question or for a statement as to whether this preference for black tea by the English is not merely a prejudice.

ADULTERATION—DEFINITION.

The law of the State of New York (see Part 2, Bulletin 13, p. 239) defines adulteration of food as follows:

The term "food," as used in this act, shall include every article used for food or drink by man. The term "drug," as used in this act, shall include all medicines for internal and external use.

b. In the case of food or drink.

- (1) If any substance or substances has or have been mixed with it so as to reduce or lower or injuriously affect its quality or strength.
- (2) If any inferior or cheaper substance or substances have been substituted wholly or in part for the article.
- (3) If any valuable constituent of the article has been wholly or in part abstracted.
- (4) If it be an imitation of, or be sold under the name of, another article.
- (5) If it consists wholly or in part of a diseased, or decomposed, or putrid, or rotten animal or vegetable substance, whether manufactured or not, or, in the case of milk, if it is the produce of a diseased animal.

b. In the case of food or drink.—Continued.

- (6) If it be colored, or coated, or polished, or powdered, whereby damage is concealed or it is made to appear better than it really is or of greater value.
- (7) If it contain any added poisonous ingredient or any ingredient which may render such article injurious to the health of the person consuming it: *Provided*, That the State board of health may, with the approval of the governor, from time to time declare certain articles or preparations to be exempt from the provisions of this act: *And provided further*, That the provisions of this act shall not apply to mixtures or compounds recognized as ordinary articles of food, provided that the same are not injurious to health and that the articles are distinctly labeled as a mixture, stating the components of the mixture.

Under this definition the following may be classed as the adulterations of teas:

- (1) Facing (6).
- (2) The addition of spent or partially exhausted leaves (3).
- (3) The addition of foreign leaves (1).
- (4) The addition of foreign astringents and substances designed to affect the apparent quality or strength (1).

ADULTERATION—METHODS.

Facing.—The treatment of teas with various coloring matters, a process termed facing, comes properly under the head of adulterants. Facing consists in treating the prepared leaves with mixtures containing Prussian blue, turmeric, indigo, or plumbago to impart some favorite color or gloss to the leaf and always has a fraudulent intent. Leaves which have been damaged in the manufacture or which from their age or certain imperfection are inferior are faced to improve their appearance and price. The teas consumed by the Chinese and Japanese themselves are not faced, while those for export seldom escape this treatment. The Chinese and Japanese black teas are usually treated with plumbago (black lead). There is no evidence that these facing agents are deleterious to the health in the quantities in which they are employed, but inasmuch as they add a useless foreign matter to the teas for the purpose of deception their use should be discouraged. "Prussian blue is insoluble in water and alcohol. * * * It is deemed a tonic, febrifuge, and alterative, but is at present rarely used. * * * The dose is from 0.2 to 0.33 gram repeated several times per day and gradually increased until some effect is produced."¹

In order to take the amount of Prussian blue stated above as a single dose in the form of tea-facing, one would have to consume nearly a pound of tea. It would require a long time under these conditions for even an inveterate tea-drinker to consume this amount of Prussian blue.

Hassall² includes Prussian blue in his list of substance "more or less injurious."

¹ See U. S. Dispensatory, 14th ed., p. 1171.

² Food, by A. H. Hassall, p. 254.

The remarks on Prussian blue apply to other facing materials, especially in regard to the large quantity of tea that must be consumed in order to take even the smallest medicinal dose of the coloring matter. The amount of coloring and inert matter (the latter often soapstone) usually amounts to a very small percentage of the weight of the tea, though statements have been made that the facing sometimes amounts to as much as from 1 to 3 per cent.¹ According to Y. Kozai² the maximum amount of facing in the green teas of Japan is about 0.4 per cent. Excessive facing is evidently a fraud, as it increases the weight and price of tea without giving the purchaser a fair return for his money.

DETECTION OF FACING.

Facing is usually easily detected by the microscope. A portion of the leaf is mounted as an opaque object, the coloring matter appearing in small dots.

Prussian blue.—This substance is easily detected by means of the microscope. Shake the leaves in a glass cylinder with water and examine the detached particles with the microscope. If the coloring matter sought is present, transparent particles of a brilliant blue may be seen. Prussian blue may often be identified by the microscope on the leaf mounted as an opaque object. The particles detached as above may be examined chemically as follows: Treat with hot sodium hydroxide solution, acidulate with acetic acid, and add ferric chloride. If Prussian blue was present in the facing the characteristic blue precipitate will be formed. The powdered tea leaf may be examined by the chemical method, but it is advisable to remove the tannin by precipitation with gelatin³ solution and filtration through powdered kaolin, after acidulating with acetic acid. The color of Prussian blue is discharged by sodium or potassium hydroxide.

Indigo.—Under the microscope indigo appears of a greenish blue. Its color is not discharged by sodium hydroxide, a distinction from Prussian blue. Indigo forms a deep blue solution with sulphuric acid.

Turmeric.—Turmeric is identified by means of the microscope. According to Hassell (*op. cit.*, 143) turmeric consists of characteristic yellow cells of a rounded form which are filled with peculiar shaped starch granules. On the addition of an alkali the cells turn brown, swell up, and the outlines of the starch granules become visible.

Plumbago.—The microscope is employed in the detection of plumbago. A thin slice of the tea leaf will exhibit numerous bright particles if plumbago facing has been used.

Gypsum, soapstone, etc.—These substances, employed with the coloring matter in facing teas, may be separated by shaking the leaves in a cylinder with water. The sediment is examined by the usual qualitative methods for these substances.

¹ Food, A. H. Hassall, pp. 122 to 129.

² Bulletin No. 7, Imperial College of Agriculture, p. 28.

³ See method for estimation of tannin, p. 890.

SPENT OR EXHAUSTED LEAVES.

The detection of spent or partially exhausted leaves in teas is not a simple problem. The chemical methods all depend upon the proportion of certain of the soluble constituents which remains in the tea leaves. A large number of analyses of genuine teas made by different chemists show a very wide range in the percentage composition of teas; hence the difficulty in obtaining a basis for comparison. A genuine tea may contain as little as 10 per cent tannin or even less, and as high as 25 per cent. It is evident from this that the percentage of tannin can not be depended upon for the detection of the admixture of spent leaves. A large quantity of such exhausted leaves would be required to reduce a tea containing a high percentage of tannin to the average percentage. The same may be said, in a measure, of each of the soluble constituents of the leaves. Hence, we see the difficulty in securing data of value on this point from a determination of the soluble matters. The only estimation of this kind that is of value, and its value it must be said is but relative, is the percentage of extract matter. A large number of analyses by different authorities shows a range in the extract matter of from 26 to 50 per cent of the air-dried tea. This extract is not the total soluble matter of the prepared leaf, but is the soluble matter extracted under certain conditions which are fully described on page 892.

One of the most valuable series of analyses of genuine teas that have been made is that of Mr. Joseph F. Geisler.¹ This chemist obtained the following averages of extract matter:

	Moist- ure.	Extract— half hour boiling in 100 parts water.	Total extract.
Indian tea:	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Maxima	6.19	39.66	45.64
Minima	5.56	37.80	41.32
Average	5.81	38.77	42.94
Average for tea dried at 100° C.		41.13	45.58
Oolong tea:			
Maxima	6.88	44.02	48.87
Minima	5.09	34.10	40.60
Average	5.89	37.88	43.32
Average for tea dried at 100° C.		40.22	46.03
Cougon tea:			
Maxima	9.15	32.14	37.06
Minima	7.65	23.48	27.48
Average	8.37	28.40	34.35
Average for tea dried at 100° C.		30.98	37.48

Formulae have been given, notably by Allen, for the calculation of the proportion of spent leaves from the amount of extract matter. Little

¹ Joseph F. Geisler in *Am. Grocer*, October 23, 1884; also in Prescott's *Organic Analysis*, pp. 505 to 512.

reliance can be placed on the results of such calculations, owing to the sources of error already named. If a tea contains less extract matter than the minima given by Geisler, the kind of tea being taken into account, it may be placed on the doubtful list, and if all other conditions agree it may be considered to have been adulterated by the addition of spent leaves. In investigating this form of adulteration the appearance of the leaf must largely determine the chemist's decision. If the leaves are very much broken, frayed, or partly unrolled, there is evidence of spent tea. For the examination, the suspected sample is soaked in water and the softened leaves are unrolled and examined on a glass plate. The following averages of the soluble constituents of genuine teas are given for comparative purposes. The analyses of Geisler are given above, so far as they relate to the extract.

Table showing the maxima, minima, and mean percentages of the principal soluble constituents of tea.

		Indian teas, Jos. F. Geisler.	Indian and Ceylon teas, David Hooper.	Black teas, A. H. Hassall.	Oolong teas, Jos. F. Geisler.	Congou teas, Jos. F. Geisler.	Green teas, A. H. Hassall.	Oolong teas, Jesse P. Batter-shall.	Japan teas, Jesse P. Batter-shall.
Tannin.	Maxima ..	18.86	21.22		20.07	13.89			
	Minima ..	13.04	10.14		11.93	8.44			
	Mean	14.87	16.62		16.38	11.54			
Theine.	Maxima ..	3.30		3.04	3.50	2.87	2.79		
	Minima ..	1.80		1.36	1.15	1.70	1.08		
	Mean	2.70		2.08	2.32	2.37	2.17		
Soluble ash.	Maxima ..	3.68			3.17	3.52			
	Minima ..	3.24			2.60	2.28			
	Mean	3.52			3.20	3.06		3.44	3.60

The differences between the highest and lowest percentages of each constituent are so marked that little possibility of detecting other than exceedingly gross adulteration, by the admixture of spent leaves, is afforded by these analytical data.

The total ash of genuine teas, according to various authorities, ranges from a little below 5 to nearly 7 per cent, the iron from .08 to .17 per cent, and the silica from .14 to .80.

FOREIGN LEAVES.

The addition of foreign leaves is best detected by means of the microscope. The leaf of the tea plant is quite characteristic in its venation, serration, and stomata. The veins recurve before reaching the border of the leaf and each forms a loop with its neighbor. The serrations are almost lacking in very delicate leaf buds, but are very distinct in the older leaves. Plates XXXIV and XL have been prepared to illustrate the leaf of the tea plant and other leaves which are said to have been

used in the adulteration of teas. These illustrations were prepared from photographic prints made by the following simple method. The natural leaf was used in making an ordinary silver print, precisely as the photographer would employ a negative. The finished print was copied by a photoengraving process. Many of these illustrations show even the delicate veins of the leaves; the tea leaf, however, is quite fleshy, and did not yield a photographic print as distinct as those from the other plants. The lower epidermis of the leaf contains most of the stomata, which are surrounded by curved cells. There are few stomata in the upper epidermis. The stomata are shown in Plate XLI. Hairs are very numerous on the younger tea leaves, but sometimes entirely wanting in old leaves. They always contain theine. Dr. Thomas Taylor,¹ in a report to the Department, mentions the presence of stone cells in tea leaves and states that his observations confirm those of Blyth in regard to the absence of these formations in certain leaves, viz, those of the willow, sloe, beech, Paraguay tea, ash, black currants, two species of hawthorn, and raspberry. Dr. Taylor also reports the presence of stone cells in the leaves of the *Camellia Japonica*, a plant related to the tea. Dr. Taylor prepares the sample of the leaf for examination by boiling three minutes with a strong caustic soda or potash solution. After the boiling a fragment of the leaf is placed on a slide under a cover glass and the latter is pressed down firmly with a sliding motion until the specimen is thin enough for microscopic examination. The stone cells appear as shown in Plate XLII.

In the general study of the serration and venation of a tea leaf the specimen should be steeped in hot water, and, after softening, the leaves should be unrolled and spread upon a glass plate for examination by transmitted light. Even small fragments of tea leaves will usually show some distinctive characteristic. In general it may be stated that a microscopic examination is only necessary in exceptional cases. In doubtful samples the stomata should be examined, and a search should be made for stone cells; the epidermis of both the upper and lower leaf should be examined. Even in the case of dust the microscope will furnish conclusive evidence as to whether it is from tea or some other plant.

According to Blyth² every part of a theine-producing plant, even the minute hairs, contain this alkaloid. The writer cited employs the following method in examining a leaf fragment for theine:

The leaf or fragment of a leaf is boiled for a minute in a watch glass with a very little water, a portion of burnt magnesia of equal bulk is added, and the whole heated to boiling and rapidly evaporated down to a large-sized drop. This drop is transferred to a subliming cell, * * * and, if no crystalline sublimate be obtained when heated to 110° (a temperature far above the subliming point of theine), the fragment can not be that of a tea leaf. On the other hand, if a sublimate of theine is obtained it is not conclusive evidence of the presence of a tea leaf, since other plants of the camellia tribe contain the alkaloid.

¹ Annual Report of the Secretary, 1889, p. 192.

² Foods: Their Composition and Analysis, A. W. Blyth, p. 322.

Theine is detected under the microscope by the appearance of the crystals.

The ash of suspected leaves should be examined for manganese and potassium, since both these substances are always present in the tea leaf.

A low proportion of soluble ash is an indication of foreign leaves, since the ash of leaves suitable for use as an adulterant usually contains a low percentage of soluble matter as compared with that from tea. Facing renders dependence upon the proportion of insoluble ash rather uncertain, as this form of adulteration, if excessive, may increase the amount of insoluble mineral matter to a considerable extent.

A careful review of the methods of detecting foreign leaves shows the microscopic to be the only methods to be relied upon in all cases.

FOREIGN ASTRINGENTS.

Catechu.—Teas are sometimes treated with this substance to increase their astringency and indicate a greater strength than they naturally possess.¹ Hager's method for the detection of catechu gave very satisfactory results in the Department's investigations. Small quantities of the substance can not be detected with certainty. The following is a general description of Hager's method:

Boil an extract of tea (1 gram per 100 cc. water) with an excess of litharge; filter; the filtrate should be clear. To a portion of the filtrate add a solution of nitrate of silver. In the presence of catechu a yellow flocculent precipitate is formed, which rapidly becomes dark. Under the same conditions pure tea gives a slight grayish precipitate of metallic silver. The writer prefers a modification of this test, using ferric chloride instead of the silver. Rinse a small porcelain dish with a dilute solution of ferric chloride; a sufficient quantity of the reagent will adhere to the dish. Add the suspected solution, prepared according to Hager. If catechu is present a characteristic green precipitate is formed.

ADDED MINERAL MATTER.

Soapstone, gypsum, etc.—These substances, as has been stated, are detected by the ordinary methods of qualitative analysis, the particles to be examined being separated from the leaves by shaking in a cylinder with water.

Iron salts.—Sulphate of iron is said to be occasionally added to a tea to deepen the color of the infusion. Iron salts may be separated from the leaves or powder by cold dilute acetic acid. This solution is tested for iron by the usual qualitative methods.

Iron.—Metallic iron has been reported as sometimes present as an adulterant of teas. Iron may be separated from the finely powdered

¹Pharm. Centralhalle, 1879, p. 258.

sample by a magnet. It may be distinguished from magnetic iron oxide by the separation of metallic copper from cupric salts.

Magnetic oxide of iron.—Blyth¹ states that ferruginous particles are sometimes found as adulterants of teas. These particles may be separated by a magnet. The author quoted (*loc. cit.*) states that he has found over 1 per cent of this ferruginous sand in teas, and that it must have been an adulterant.

Sand, particles of brick, etc.—A small amount of sand in a tea may be due to accidental causes while gathering the leaves, but any considerable quantity and particles of brick and similar matter can only be considered as having been added with fraudulent intent. These substances may be separated by shaking the leaves with water and collecting the sediment.

Copper.—The green color of some teas is popularly attributed to copper. There is little evidence to prove that copper has ever been employed for this purpose. Hassall² made a large number of examinations of teas without detecting copper in a single sample. If copper is present in a tea a portion of the powdered sample added to ammonia water will impart a blue tint to the latter, the depth of the tint depending upon the amount of copper present.

LIE TEA.

This substance, as its name implies, is an imitation of tea, usually containing fragments or dust of the genuine leaves, foreign leaves, and mineral matters, held together by means of a starch solution and colored by one of the facing preparations. It is stated that gunpowder and imperial teas are more subject to this form of adulteration. Of the samples examined by the Department of Agriculture all were free from lie tea.

According to Hassall³ the percentage of ash in lie tea ranges from 13.05 to 52.92 for black teas and 13.13 to 56.34 for green teas. The same authority also found black teas containing from 6 to 17.7 per cent lie tea, and green teas containing 1.38 to 48.46 per cent of this adulterant.

To detect lie tea treat the suspected sample with boiling water; if it contain this adulterant portions will break up into dust and leaf fragments.

GENERAL REMARKS ON TEA ADULTERANTS.

The adulterants of teas, as a rule, are not such as may be considered prejudicial to the health. The mineral matters employed in facing, etc., with the exception of salts of iron or copper, are insoluble in water, hence would not be present in the infusion. Copper, which is

¹ Foods: Their Composition and Analysis, A. W. Blyth, 322.

² Food, Its Adulterations and the Methods for Their Detection, A. H. Hassall, p. 131.

³ Hassall, *op. cit.*, 117.

probably very rarely present, would be very objectionable even in very small quantities. Sulphate of iron is a powerful astringent. In the small quantities which would be used in increasing the astringency of teas it would possibly not be seriously objectionable. It may also be said of sulphate of iron that this substance was not detected in a very large number of samples examined by Hassall nor in the Department's samples.

Facing, if excessive, increases the weight of the tea, but there is no evidence of its being prejudicial to the health.

From these statements we see that the adulterations of teas are intended more especially to enhance the value of inferior grades, except in the case of lie tea or the addition of foreign or exhausted leaves. In the latter cases a spurious article, which is not justified by any quality or principle which it contains, is foisted upon the market.

The evidence of authorities upon food adulteration confirms the statement that the addition of foreign leaves is now but little practiced. The general freedom of the teas of the markets of the United States from adulteration is largely due to the enforcement of the United States tea-adulteration law. Dr. Jesse P. Battershall,¹ under whose direction a very large number of samples of teas were examined in connection with this law, notes a very perceptible improvement in the quality of teas imported under its provisions.

GENERAL STATEMENTS CONCERNING THE CONSTITUENTS OF TEAS.

The analyses of teas give little or no evidence bearing upon the market value of this commodity. A tea may be very rich in theine or tannin, and yet bring the same price as one poor in these substances. The same may be said in a general way of the other constituents. This is largely due, undoubtedly, to differences in soil, climate, the age of the leaf, and methods of manufacture. The flavor, strength, and appearance of a tea in general determine its market value. The flavor is due to the volatile oil developed during the manufacture, the strength to the proportion of tannin, and the appearance of the leaf to its age and the care taken in the rolling and other manipulations. A tea may be deficient in tannin and still bring a high price on account of the delicacy of its flavor, or, by the admixture of leaves rich in tannin, an operation termed blending, its strength may be increased to meet the demands of the market. In genuine unfaced teas the value is usually in a direct ratio with the soluble matter in the ash. According to Geisler, the finer the quality of the tea the more theine, soluble ash, and extractive matter it will contain, though this is not uniformly true. It has been stated that the relatively high price of Indian teas is largely due to their percentage of tannin. This permits the use of a very much smaller quantity of tea in preparing the infusion for the table. The writer found,

¹ Food Adulteration and Its Detection, E. & F. Spon, New York, p. 20.

among the samples which he submitted to analysis, 50-cent teas (retail price) containing as much tannin as many of those whose selling price was nearly double this amount.

An attempt was made in classifying teas to utilize the property which high grades have of quickly giving up a portion of their soluble matter when steeped in water. These experiments were carefully made, the conditions being the same in each case, but unfortunately the results were far from satisfactory. This problem must be left until more thorough studies of the chemistry of teas have been made, and until then we must continue as heretofore to depend upon the skill of the professional tea-taster and the honesty of the dealers in regulating the relative prices.

Since the above and other remarks on the valuation of teas were written, a Russian chemist¹ has made an interesting series of investigations bearing upon this question.

According to this chemist the quality of the tea depends upon the care with which the fermentation is conducted. The fermentation of the leaves destroys a portion of the tannin, thus decreasing the astringent action of the tea. Mr. Doorkovitch has devised a process for the estimation of the products of the fermentation. In the article cited he presents the table of analyses given below, and on a basis of these analyses makes the following deductions:

The greater the ratio of theine to the total amount of tannin and products of fermentation, the dearer the tea. The more regular the fermentation, the better the tea.

The regularity of the fermentation is recognized by the relative amount of products of fermentation in tea.

Analyses of first-crop China teas of the season of 1890.

[Tea dried at 100° C.]

Number of tea.	Water.	Theine.	Tannin.	Products of fermentation.	Extractive substances.	Total amount of theine, tannin, and products of fermentation.*	Theine, per cent A.*	Tannin, per cent A.*	Products of fermentation, per cent A.*
1	7.44	2.14	9.44	1.80	33.43	13.38	16.00	70.55	13.45
2	7.79	2.50	9.87	1.61	33.33	13.98	17.89	70.60	11.51
3	8.29	2.53	9.27	1.68	32.11	13.48	18.78	68.76	12.46
4	-----	2.68	10.05	1.44	37.26	14.17	18.92	70.92	10.16
5	7.97	2.66	9.77	1.55	34.55	13.98	19.03	69.89	11.08
6	8.16	2.65	9.76	1.45	31.20	13.86	19.13	70.41	10.46
7	7.66	2.72	9.59	1.78	30.70	14.09	19.21	68.06	12.63
8	7.90	2.73	-----	-----	-----	-----	-----	-----	-----
9	7.91	2.86	-----	-----	-----	-----	-----	-----	-----
10	-----	2.91	10.38	1.52	34.88	14.81	19.65	70.09	10.26
11	7.60	3.00	10.55	1.67	34.00	15.22	19.79	69.31	10.90

* Total amount of theine, tannin, and products of fermentation is represented by A for brevity.

¹Examination of China Teas, P. Doorkovitch, Jour. anal. and appl. Chem., 5, 6, 345.

Analyses of first-crop China teas of the season of 1890—Continued.

Number of tea.	Water.	Theine.	Tannin.	Products of fermentation.	Extractive substances.	Total amount of theine, tannin, and products of fermentation.*	Theine, per cent A.*	Tannin, per cent A.*	Products of fermentation, per cent A.*
12	-----	2.87	10.05	1.74	33.90	14.66	19.92	68.50	11.68
13	-----	2.83	10.07	1.35	33.15	14.25	19.87	70.66	9.47
14	8.07	2.88	9.65	1.65	30.92	14.17	20.33	68.10	11.57
15	-----	2.82	9.36	1.59	33.00	13.77	20.55	67.90	11.55
16	-----	3.11	10.03	1.70	32.21	14.84	20.96	67.59	11.45
17	8.10	3.00	9.36	1.88	34.12	14.24	20.02	65.73	13.25
18	-----	3.10	10.60	1.50	34.10	14.60	21.23	68.50	10.27
19	-----	3.16	9.80	1.75	33.66	14.71	21.48	66.62	11.90
20	-----	3.02	9.37	1.50	32.40	13.89	21.74	67.46	10.80
21	9.08	3.00	9.45	1.18	33.80	13.63	22.02	69.33	8.65
22	7.84	3.00	8.84	1.18	32.20	13.02	23.05	67.89	9.06
23	8.85	3.02	9.05	.90	33.00	12.97	23.29	69.77	6.94
24	8.20	3.27	9.21	1.44	34.95	13.92	23.50	66.16	10.34
25	8.24	3.25	9.14	1.25	32.93	13.64	23.84	67.00	9.16
26	9.13	3.41	9.32	1.44	33.26	14.17	24.07	65.77	10.16
27	9.78	3.33	9.22	1.27	32.00	13.82	24.11	66.71	9.18
28	8.42	3.45	9.42	1.38	34.80	14.25	24.22	66.10	9.68
29	7.83	3.21	9.00	1.17	33.46	13.38	24.52	67.26	8.22

* Total amount of theine, tannin, and products of fermentation is represented by A for brevity.

ANALYTICAL METHODS.

In the following paragraphs are presented the methods adopted for the estimation of the various constituents of teas.

Caffeine or theine, $C_8H_{10}N_4O_2$.—After experiments with a number of methods for the estimation of caffeine, the following was adopted on account of its simplicity and the accuracy of the results:

¹Transfer 3 gs of powdered tea into a 300 cc flask, add about 250 cc of water; heat gradually to the boiling point, using a small fragment of tallow to prevent frothing; boil gently 30 minutes, washing down any particles of tea which may collect on the flask above the liquid. After the liquid begins to boil the flask should be filled almost to the neck and water should be occasionally added to keep its level at this point. Several samples may easily be extracted in this way at the same time, since they require but little attention after regulating the flame of the lamp. After boiling 30 minutes, cool, add a strong solution of basic acetate of lead in sufficient quantity for the removal of precipitable substances; usually about 3 cc are required; complete the volume to 300 cc, mix thoroughly, and filter, rejecting the precipitate unwashed. Treat 50 cc of the filtrate with H_2S for the removal of the lead; boil off the excess of H_2S ; filter into a separatory funnel, washing the precipitate with hot water, or an aliquot part of the filtrate may be taken and washing of the precipitate may thus be avoided. The error from the volume of the precipitate is very slight.

Extract the water solution in the separatory funnel seven times with chloroform; collect the chloroform solution of caffeine in a small tared flask, and remove the solvent by distillation; dry the caffeine at $75^\circ C$. two hours, weigh, and calculate the percentage.

¹ Jour. anal. Chem. 4, 4, p. 390.

Nitrogen.—Tea contains a very high proportion of nitrogen. The analyses of Mr. Kozai, tabulated on page 879, will show how this nitrogen is distributed in the prepared leaf.

The nitrogen of teas should be determined by the absolute method, or Kjeldahl method, modified for alkaloids. These methods are fully described in the text-books on quantitative analysis, and in Bulletin 24, page 217, of the Chemical Division of this Department.

Albuminoid nitrogen.—*Stutzer's*¹ *method*.—Prepare cupric hydrate as follows: Dissolve 100 grams of pure cupric sulphate in 5 liters of water, and add 2.5 cubic centimeters of glycerin; add dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cubic centimeters of glycerin per liter, and then wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerin, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture. To 1 gram of this substance add 100 cubic centimeters of water in a beaker; heat to boiling, or, in the case of substances rich in starch, heat on the water bath ten minutes; add a quantity of cupric hydrate mixture containing 0.7 to 0.8 grams of the hydrate; stir thoroughly; filter when cold; wash with cold water, and put the filter and its contents into the concentrated sulphuric acid for the determination of nitrogen after Kjeldahl. For the above filtration use Schleicher and Schüll's No. 589 paper, or Swedish paper, either of which contains so little nitrogen that it can be left out of account.

Tannin.—*Löwenthal's*² *method as improved by Conneler and Schroeder, and Procter*.—In the estimation of tannin by this method the following standard solutions and reagents are required:

(1) Potassium permanganate solution containing, approximately, 1.33 grams of the salt per liter.

(2) Tenth-normal $\left(\frac{N}{10}\right)$ oxalic acid solution, for use in standardizing the permanganate solution.

(3) Indigo-carmin solution, containing 6 grams of this indicator and 50 cc concentrated sulphuric acid per liter.

(4) Gelatin solution, prepared by swelling 25 grams of gelatin for one hour in a saturated solution of common salt, then heating until the solution is complete, and finally, after cooling, making up to one liter (W. H. Krig's method of preparing this solution).

(5) Salt-acid solution, prepared by mixing 975 cc saturated common salt solution and 25 cc concentrated sulphuric acid.

(6) Powdered kaolin.

The potassium permanganate solution is standardized in the usual manner by oxalic acid. It is obvious, in the analytical manipulations which follow, that the end reaction (golden yellow or pink tinge) which is adopted in the first process must also be employed in the second. The indigo-carmin should be very pure and especially free from indigo-blue.

(a) Five grams of finely powdered tea are placed in a flask of approximately 500 cc capacity and boiled thirty minutes with 400 cc distilled water. The water should be cold when added to the sample. After the completion of the boiling, the flask is cooled and the solution and residue are transferred to a half-liter flask and the volume made up to 500 cc. The solution required for analysis is filtered off if necessary. To 10 cc of the tea infusion, 25 cc indigo-carmin solution are added, and

¹ Bulletin 31, Div. Chem., U. S. Dept. Agric., p. 189.

² Conneler and Schroeder, *Ztsch. anal. Chem.* **25**, 121. Procter, *Journ. Soc. Chem. Ind.*, **3**, 82.

approximately 750 cc distilled water. The permanganate solution is now added, a cubic centimeter at a time, the liquid being vigorously stirred after each addition, until the color changes to a light green; the addition of permanganate is continued more slowly, drop by drop, until the whole liquid takes on a bright golden-yellow color (Couneler and Schroeder), or, if preferred, until the pure yellow liquid shows a faint pinkish rim (Procter). The burette reading is now taken, and furnishes the value (*a*) of the formula. It is absolutely necessary to vigorously stir the liquid during the whole operation. It is best to repeat this titration, as well as that which follows in the next step of the analysis, and take a mean of several readings. (*b*) 100 cc of the tea-infusion (filtered if not sufficiently clear after decantation) are mixed with 50 cc of the gelatine solution in an Erlenmeyer flask, then 100 cc of the salt-acid solution and 10 grams of kaolin are added, and the whole vigorously shaken in the well-corked flask. Several minutes' shaking is necessary. If these directions are carefully followed the precipitate will settle very rapidly, leaving a clear, supernatant liquid which filters with great ease. The use of kaolin, as recommended by Procter, is an important modification of the original method, without which it is often impossible to separate the precipitate. The whole liquid is filtered and 25 cc of the filtrate (=10 cc of the original infusion) are mixed with 25 cc of indigo-carmin solution and about 750 cc water, and a titration made as under (*a*). The burette reading gives the value *b* of the formula.

The value *a* is the amount of permanganate solution necessary to oxidize all oxidizable substances present; *b*, the amount required to oxidize the substances other than tannin; hence $a - b = c$ = permanganate solution required by the tannin. According to Neubauer, .04157 grams of gallo-tannic acid is equivalent to .063 grams oxalic acid; therefore, knowing the amount of oxalic acid equivalent to the permanganate required to oxidize the tannin, we can easily calculate the amount of this latter substance present.

The above method for tannin was selected after experiments with several other methods. The general advocacy of its use by a number of very able analysts and the satisfactory results obtained in this Department led to its adoption for this work. Unfortunately, owing to the great variations in the results obtained by different methods and the acknowledged inaccuracy of many of these methods, the value of a large number of tannin determinations, by various authorities, is doubtful. For work of this kind the method of analysis should be clearly stated, and as far as possible, for the sake of uniformity, analysts should all adopt the same method in order that their work may be comparable with that of others.

Water.—The moisture may be determined in the usual manner by drying 1 or 2 grams of the powdered tea three hours at 100° C. in a flat dish, and calculating the water from the loss in weight. This method probably entails a slight loss of theine, which is credited to the moisture, but the error is very small and is negligible.

Ash, total.—Two grams of the powdered tea are incinerated, at as low a temperature as practicable, and the percentage calculated as usual.

Ash, soluble and insoluble.—The total ash is treated on a filter with hot water until all the soluble matter is dissolved; the solution is evaporated to dryness, and the residue ignited at a moderate temperature. The per cent soluble ash is calculated from the weight of this residue; the insoluble ash is determined by difference.

Ash insoluble in acid.—The water insoluble residue from the soluble ash determination is treated with hydrochloric acid. The undissolved portion is washed with water, dried and weighed, and its percentage calculated.

Ash, alkalinity.—The soluble matter obtained in determining the soluble ash is dissolved in a little water and titrated with $\frac{N}{10}$ acid. The alkalinity is calculated as potassio oxide (K_2O).

Extract, total, and insoluble leaf.—Two grams of finely powdered tea are successively extracted with seven portions of 50 cc each of boiling water. The extract is

decanted each time and the fractions united. The decanted solution is now boiled and passed through a tared filter. The insoluble residue is finally transferred to this filter, thoroughly washed with boiling water, dried, and weighed. The total weight, less the tare of the filter, is the insoluble residue from which the per cent insoluble leaf is calculated. The total extract is determined by difference, deducting the per cent of insoluble leaf from 100.

A weighing tube should be used in weighing filters and residues on filters.

Half-hour extracts.—This extract is of doubtful value for comparative purposes unless certain conditions are adopted and strictly adhered to. Slight variations may cause very perceptible errors in the results. I have followed essentially the conditions indicated by Geisler, which are evidently based on Wanklyn's method.

Place 1 gram of leaf tea in a 300 cc flask, add 100 cc cold distilled water and a small fragment of paraffin, to prevent foaming. Immerse the flask a minute or two in water heated to 90°C. in order to raise the temperature of its contents quickly to within a few degrees of the boiling point. Having dried the outside of the flask, boil the mixture thirty minutes. The flask must be fitted with a reflux condenser. After boiling, cool quickly in a stream of water and filter off the extract. Transfer an aliquot part of the extract to a tared dish and dry. Calculate the per cent of matter from the weight of the residue.

GENERAL REMARKS TO ANALYSTS.

The analyst must be guided almost entirely by comparisons of his work on the suspected teas with the records of analyses of pure samples. The microscopic are almost the sole methods of detecting many of the adulterants of teas. Questions in regard to quality, where this depends upon aroma only, must be answered by a professional tea-taster.

REPORT OF THE EXAMINATION OF SAMPLES OF TEAS BOUGHT IN THE OPEN MARKET.

The samples examined in the course of the investigations were purchased in stores of all grades. The analyses of these teas and general statements concerning them are given in the following tables:

Description of samples.

Date of purchase.	Serial number	Retail dealer.	Sold as—	Retail price per pound.
1887. Feb. 15	4850	Metzger, Seventh between D and E streets.	Cheap green tea	\$0.50
	4851	do	Cheap black tea	.50
	4852	G. G. Cornwall & Sons.....	Gunpowder.....	1.30
	4853	do	Oolong tea.....	.88
Jan. 18	4860	B. W. Reed's Sons	do	.60
	4863	Pacific Tea Store, Seventh street NW.	Black tea.....	.50
	4864	do	Green tea.....	.50
	4865	Brought from China by T. C. Trescot, esq.	From a Formosa garden (black)	
Apr. 8	4940	Metzgers, Seventh street	Imperial tea	1.00
	4941	do	Formosa Oolong	.75
	4942	Atlantic and Pacific Tea Co., Seventh street.	English breakfast	.80

Description of samples—Continued.

Date of purchase.	Serial number	Retail dealer.	Sold as—	Retail price per pound.
1887.				
Apr. 8	4943	Atlantic and Pacific Tea Co., Seventh street.	English breakfast	\$0.60
	4944	do	do	.40
July 27	5139	N. M. Burchell, 1325 F street.....	Russian tea (black).....	1.25
	5140	do	English breakfast	1.00
	5141	do	Gunpowder	.75
	5142	527 Tenth street NW.....	Black tea	.60
	5143	Jas. Rutherford, 906 D street NW	do	.50
	5144	W. H. Combs, 920 Pennsylvania avenue	Gunpowder	.50
	5145	do	Uncolored Japan	.50
	5146	Wm. Orme & Son, 1013 Pennsylvania avenue.	English breakfast	.90
	5147	M. Goddard, 620 Twelfth streets NW	Uncolored Japan.....	.75
Aug. 19	5152	1200 Thirteenth street NW	Oolong.....	.60
	5153	1130 Thirteenth street	Japan	.50
	5154	700 Thirteenth street	Gunpowder.....	.90
	5155	Corner New York avenue and Thirteenth streets NW.	Oolong.....	.50
	5156	F. N. Lanckton, 209 Four-and-a-half street.	Fine imperial	.50
	5157	J. H. Grimes, 235 Pennsylvania avenue	Basket fired	.60
	5158	B. Maranghi, 101 B street SE.....	Black tea	.45
	5159	M. McCormack, 227 Pennsylvania avenue.	Japan	.50
	5160	G. Riani, 135 B SE	Black tea.....	.50
	5161	Enterprise Tea Co., Fourth street and Pennsylvania avenue SE.	Mixed	.40
Aug. 23	5167	Pekin Tea and Coffee Co., 1308 Seventh street.	Old Hyson tea	.60
	5168	Great Atlantic and Pacific Tea Co., 1620 Fourteenth street.	do	.50
	5169	United States Tea Co., 628 Pennsylvania avenue.	do	.50
	5170	Chicago Grocery Co., 806 Seventh street.	Young Hyson tea	.50
	5171	China and Japan Tea Co., 714 Seventh street.	do	.60
	5172	1500 Seventh street	Moyune Gunpowder.....	.80
	5173	J. C. Ergood, 915 Louisiana avenue	Moyune Gunpowder, third picking.	.75
	5174	212 Thirteenth street SW	Cheap Moyune Gunpowder.....	.50
	5175	Geo. E. Kennedy, 1209 F street	Gunpowder	1.00
	5176	Jackson & Co., 626 Pennsylvania avenue.	do	.75
	5177	F. Bell Tea Co., 824 Seventh street	Japan Basket	.50
	5178	F. Bell Tea Co., 824 Seventh street.....	Mixed teas.....	.50
	5179	1367 C street SW	Oolong.....	.50
	5180	301 Fourteenth street NW.....	Black, cheap.....	.50
	5181	G. G. Cornwall & Son, 1416 Pennsylvania avenue.	Pekoe.....	.85
1881.				
June 1	5940	Park & Tilford, Broadway, New York	Best Formosa Oolong.....	.75
	5944	do	Best English Breakfast.....	.90
	5945	do	Best Oolong	.80
	5946	do	Best Japan	1.00

Description of samples—Continued.

Date of purchase.	Serial number	Retail dealer.	Sold as—	Retail price per pound.
1881.				
June 1	5947	Park & Tilford, Broadway, New York.	Best Young Hyson	\$1.00
	5948	do	Best Gunpowder	1.00
	5949	Not known, New York	Black tea ¹	.47
13	5960	Martin, Gillet & Co., Baltimore, Md.	Standard Heno Tea	
	5961	Presented by the Chinese minister to the ladies' committee, Garfield Hospital.		
19	5963	Abbott Branch Store, H and Eleventh streets NW.	Imperial cheap green tea	.50
1890.				
Feb. 3	6659	McGruder, New York avenue NW....	Best Oolong	1.00
	6660	do	Best English Breakfast	1.00
Mar. 8	6679	C. D. Kenny, corner Seventh and Ninth streets NW.	Gunpowder	.50
10	6680	Redstrake, New York avenue and Sixth streets.	Johnson's Spring Leaf Blended Tea ²	.55
Apr. 26	6803	The Boston Store	Tetley's India and Ceylon Tea No. 1.	.70
	6804	do	Tetley's India and Ceylon Tea No. 2.	.50

¹ Sold only in 5-pound packages.² Sold in 5-cent packages; 11 packages = 15½ ounces.*Analyses of teas.*

Date of purchase.	Serial number.	Moisture.	Total ash.	Soluble ash.	Insoluble ash.	Soluble ash, per cent total ash.	Total extract.	Half-hour extract.	Tannin.	Nitrogen.	Theine.
1887.		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Feb. 15	4850	5.52	5.86	3.93	2.03	65.36	52.75		13.17	3.83	2.50
	4851	5.38	6.00	3.54	2.46	59.00	48.98		13.17	3.60	1.09
	4852	5.72	6.21	4.01	2.20	64.58	50.11	44.02	14.11	4.58	3.01
	4853	5.40	6.63	3.31	3.32	49.92	49.47		12.30	4.06	1.50
Jan. 18	4860	3.96	5.88	3.37	2.51	57.31	51.06		15.41	4.12	2.13
	4863	5.65	5.76	2.57	3.19	44.62	46.84		11.17	3.46	2.69
	4864	5.31	7.40	2.87	4.53	38.78	47.27	34.92	10.74	3.27	1.12
	4865	7.05	5.69	3.24	2.45	56.94		31.72		4.01	
Apr. 8	4940	6.99	5.76	3.67	2.09	63.72	48.28	36.58	12.57	4.03	2.13
	4941	6.45	6.83	3.47	3.36	50.81	48.22	35.88	7.44	4.00	1.92
	4942	7.05	6.48	4.19	2.29	64.66	44.60	32.12	5.07	3.98	3.11
	4943	8.43	6.13	3.21	2.92	52.37	44.50	31.56	9.88	3.70	1.58
	4944	7.76	6.66	3.79	2.87	56.91	42.84	30.08	5.29	3.99	2.89
July 27	5139	7.78	5.83	3.65	2.18	62.61	51.26	39.70	10.71	4.09	3.43
	5140	8.79	5.85	3.55	2.30	60.61	47.73	22.00	7.63	4.59	3.43
	5141	6.39	5.93	3.54	2.39	59.70	48.28	28.26	6.93	4.37	2.60
	5142	6.67	6.41	3.04	3.37	47.41	46.75	34.20	8.13	3.45	2.42
	5143	8.24	5.83	2.78	3.05	47.69	48.33	32.40	8.51	3.23	1.06
	5144	6.35	6.87	3.29	3.58	47.89	48.25	34.50	9.05	3.36	1.62
	5145	7.89	6.46	3.16	3.30	48.92	47.83	38.10	8.25	3.97	2.07
	5146	8.57	6.45	2.59	3.86	40.16	47.86	36.12	11.02	3.93	2.53
	5147	7.18	7.15	3.55	3.60	49.66	50.91	40.56	12.51		1.00
Aug. 19	5152	8.74	6.08	3.59	2.49	59.15	47.93	39.04	8.00	3.12	1.61
	5153	8.12	7.69	2.87	4.82	37.33	46.18	34.14	7.03	3.09	2.20

Analyses of teas—Continued.

Date of purchase.	Serial number.	Moisture.	Total ash.	Soluble ash.	Insoluble ash.	Soluble ash, per cent total ash.	Total extract.	Half-hour extract.	Tannin.	Nitrogen.	Theine.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
1887.											
Aug. 19	5154	6.32	6.69	3.41	3.28	50.87	53.76	42.68	8.61	3.24	1.87
	5155	7.40	6.13	2.99	3.14	48.85	49.09	39.42	9.92	3.34	1.55
	5156	6.59	5.99	3.28	2.71	54.67	50.40	39.60	9.79	3.83	1.55
	5157	4.91	6.55	4.34	2.21	66.26	49.31	39.24	8.65	3.86	1.49
	5158	8.58	6.11	3.21	2.90	52.87	48.98	31.66	10.10	3.25	1.04
	5159	9.58	5.89	3.50	2.39	59.42	49.27	37.54	7.61	4.11	2.31
	5160	8.46	5.92	1.78	4.14	30.03	45.41	34.44	8.76	3.16	2.14
	5161	7.82	6.83	2.61	4.22	38.21	48.26	38.42	7.70	3.30	1.57
23	5167	8.71	6.21	3.24	2.97	52.18	43.40	33.98	6.71	3.18	1.93
	5168	9.90	6.05	2.58	3.47	42.58	47.08	34.52	8.25	3.33	2.00
	5169	8.61	5.93	2.67	3.26	45.01	48.40	29.78	4.77	3.19	2.53
	5170	9.72	5.36	1.66	3.70	31.02	46.44	36.12	10.75	2.91	1.98
	5171	8.75	6.43	4.21	2.18	66.10	48.98	39.06	11.62	3.53	2.27
	5172	8.28	6.02	2.78	3.24	46.14	50.52	39.30	14.09	3.80	2.01
	5173	8.40	6.45	4.25	2.20	65.89	50.76	40.96	11.48	4.15	2.00
	5174	9.64	6.65	1.99	4.66	34.37	47.70	36.68	7.23	3.33	1.26
	5175	5.05	6.09	4.41	2.68	55.95	52.93	43.54	12.60	4.16	2.22
	5176	7.54	6.65	4.40	2.25	66.09	47.56	38.30	8.26	3.93	2.29
	5177	8.70	5.59	3.04	2.55	55.35	49.97	38.74	14.76	3.76	2.05
	5178	9.15	6.20	2.41	3.79	38.91	47.88	34.84	6.13	3.37	2.98
	5179	8.64	5.98	3.09	2.89	51.67	48.51	37.02	10.06	3.38	2.09
	5180	8.60	5.66	2.88	2.78	50.87	49.65	37.98	12.39	3.32	1.43
	5181	8.49	5.38	2.71	2.67	50.39	53.32	39.98	15.51	3.82	2.06
1888.											
June 1	5943	5.82	6.53	4.18	2.40	63.53	52.07	39.90	14.58		2.17
	5944	8.22	6.36	4.22	2.14	66.36	48.79	39.82	5.87		2.73
	5945	4.95	6.33	3.83	2.50	60.51	50.16	38.55	12.34		1.34
	5946	4.87	6.17	3.63	2.54	58.39	51.19	45.28	8.38		2.54
	5947	5.54	6.10	4.22	1.88	69.18	49.53	41.98	11.48		1.73
	5948	5.32	6.50	4.61	1.89	70.92	54.36	40.94	12.01		1.64
	5949	6.03	6.75	4.23	2.52	62.67	50.41	38.32	10.19		1.03
June 13	5960	4.85	6.40	3.84	2.56	60.00	42.68	40.24	11.08		2.15
	5961	3.59	6.98	3.86	3.12	55.30	48.03	38.31	14.76		1.93
June 19	5963	4.61	6.00	3.66	2.34	61.00	41.38	39.20	9.01		1.28
1890.											
Feb. 3	6659	5.08	6.21	3.45	2.76	55.55	50.11	41.00	12.85		3.09
	6660	3.65	5.29	3.24	2.05	61.06	49.46	40.40	15.21		2.66
Mar. 8	6679	4.14	7.40	3.00	4.40	40.54	47.38	35.18	8.91		1.49
Mar. 10	6680	3.70	7.12	2.87	4.25	40.31	47.86	32.70	12.35		1.43
Apr. 26	6803	4.84	5.61	3.48	2.13	62.03	47.26		13.67		2.15
	6804	4.50	5.90	3.51	2.39	59.48	45.56		11.99		1.92

Table showing the condition of the leaves and the foreign matters present.

Serial No.	Color, etc.	Retail price per pound.	Qualitative examination.
4850	Green	\$0.50	Largely composed of broken leaves. Faced.
4851	Black.....	.50	Leaves large.
4852	do	1.30	Leaves medium size, very much broken. Faced with black lead.
4853	do	.88	Leaves very much broken.
4860	do	.60	Composed entirely of broken leaves. Faced.
4863	do	.50	Leaves small, mostly broken.
4864	Green	.50	Largely composed of fragments. Faced.
4865	Black		Do.
4940	Green	1.00	Large leaves, very much broken. Faced.
4941	Black	.75	Fragments of medium and small sized leaves.
4942	do	.80	Largely composed of fragments of medium leaves.
4943	do	.60	Do.
4844	do	.40	Consists largely of fragments and frayed leaves. Black lead facing; contains magnetic iron oxide.
5139	do	1.25	Largely composed of fragments.
5140	do	1.00	Do.
5141	Green	.75	Composed of large leaves. Faced.
5142	Black	.60	Largely composed of fragments.
5143	do	.50	Leaves large.
5144	Green	.50	Leaves frayed and largely fragments. Faced.
5145	Uncolored Japan.	.50	Fragments of large and small leaves; frayed. Faced.
5146	Black	.90	Fragments of medium sized leaves; frayed. Black lead facing.
5147	Uncolored Japan.	.75	Medium and small leaves, and a large proportion of stems.
5152	Black	.60	Medium and small leaves.
5153	Uncolored Japan.	.50	Composed of fragments of leaves. Faced.
5154	Green	.90	Fragments. Faced.
5155	Black	.50	Fragments. Contains paddy husks and rice seed. Black lead facing. Contains magnetic iron oxide.
5156	Green	.50	Medium and small leaves. Faced.
5157	Black	.60	Large leaves, frayed. Contains paddy husks. Black lead facing. Contains catechu.
5158	do	.45	Large leaves, frayed. Contained paddy husks.
5159	do	.50	Large leaves, frayed. Black lead facing.
5160	do	.50	Fragments. Contains paddy husks. Black lead facing.
5161	Black and green.	.40	Fragments of large leaves. Black lead and Prussian blue facing.
5167	Green	.60	Largely composed of fragments. Indigo facing.
5168	do	.50	Fragments of large leaves. Faced.
5169	Black	.50	Fragments of leaves. Contained pieces of gypsum, wood, and fragments apparently nut shells.
5170	Green	.50	Fragments. Faced.
5171	do	.60	Fragments. Faced.
5172	do	.80	Large leaves. Faced.
5173	do	.75	Large leaves. Faced.
5174	do	.50	Large leaves. Faced.
5175	do	1.00	Leaves small. Many fragments. Faced.
5176	do	.75	Leaves large. About 20 per cent tea-dust. Faced.
5177	Japan basket.	.50	Leaves small. Fragments few. Contains paddy husks.

¹ Facing unless otherwise stated is a mixture containing Prussian blue.

Table showing the condition of the leaves and the foreign matters present—Continued.

Serial No.	Color, etc.	Retail price per pound.	Qualitative examination.
5178	Black and green.	\$0. 50	Fragments of large leaves. Prussian blue and black lead facing. Contains magnetic iron oxide.
5179	Black.....	. 50	Fragments of medium-sized leaves. Black-lead facing contains magnetic iron oxide.
5180	do	. 50	Fragments of large leaves. Contains paddy husks.
5181	do	. 85	Small leaves and fragments. Black-lead facing.
5943	do	. 75	Small leaves and fragments. Contains paddy husks. Black-lead facing.
5944	do	. 90	Small leaf fragments.
5945	do	. 80	Small leaf fragments. Black-lead facing.
5946	Japan	1. 00	Small leaves, fragments, and pekoe tips.
5947	Green	1. 00	Small leaves. Indigo facing.
5948	do	1. 00	Small leaves and fragments. Indigo facing.
*5949	Black.....	. 47	Small leaf fragments.
5960	Black and green.		All fragments. Contains paddy husks.
5961	Green		All stems and leaf buds.
5963	do	. 50	A large proportion of fragments of leaves.
6659	Black.....	1. 00	Very few fragments.
6660	do	1. 00	Largely composed of fragments.
6679	Green.....	. 30	Fragments of leaves. Contains fragments of brick and lead. Faced.
6680	Green and black.	. 55	Fragments of leaves. Contains paddy husks and a few grains of rice. Black lead and Prussian blue facing.

* Sold in \$5 packages only.

The prices of a few samples are omitted, since these teas were not purchased by the Department. As nearly as possible all the grades of teas on the market are represented in this work.

Many of these samples are of very inferior quality, but neither the analytical nor microscopical data give positive evidence of the addition of spent or foreign leaves. That this latter form of adulteration is still practiced is evidenced by the work of a Canadian official chemist who found two samples containing foreign leaves.¹ Dr. Jesse P. Battershall² examined nearly 2,000 suspected samples of teas under the United States tea adulteration act, and states that he found foreign leaves present in only a few instances.

Some of the higher-priced teas contained frayed and unrolled leaves, but not in sufficient quantities to justify considering the sample to have been adulterated with spent leaves, especially as the relative proportions of the soluble constituents of the teas varied little from the average for genuine, unadulterated samples.

A large number of the samples examined by the writer were faced. With the present ideas in regard to this practice, it can not be considered a form of adulteration, but facing should be condemned on account of its use in making inferior teas appear to be of a superior quality. This

¹ Report on adulteration of food. Supplement III to the report of the Department of Inland Revenue, 1886. Ottawa.

² Food adulteration. Jesse P. Battershall, page 20.

practice also enables the admixture of spent leaves with little fear of detection. Faced teas can not be excluded from this country under the United States tea adulteration act, since this law specifies that the addition of chemical and other deleterious substances must be in sufficient quantities to render the tea unfit for use. This wording admits of excessive facing, since it has never been shown that the substances usually employed for this purpose are prejudicial to health, even when taken in greater quantities than could be employed in treating teas.

A few of the samples examined contained magnetic-iron oxide, probably derived from the black-lead facing. Serial number 6679 contained a few fragments of brick and lead. These substances were probably added to increase the weight of the tea and were not accidental.

A general qualitative examination was made of each sample. The foreign matters detected are given in the table, p. 896. Very few objectionable substances were found. A statement of the condition of the leaves is also given in this table. The teas of all grades were largely composed of fragments of leaves.

CONCLUSION.

The analytical and other work in connection with this report indicates that there are few if any spurious teas on the market. The range in quality is undoubtedly very great, many samples deserving to be termed "tea" simply because they are composed of the leaves of the *Thea*, and not through the many pleasant qualities which we usually associate with the beverage of this name.

With the strict enforcement of the United States adulteration act, the consumer is reasonably well protected, so far as securing the genuine leaf is concerned, but of course has no protection from the sale of inferior teas.

COFFEE.

The seeds of the *Coffea arabica*, after roasting at a temperature approximating 200° C., are ground and employed in preparing the popular beverage termed coffee. Various substitutes have been prepared by manufacturers for the purpose of cheapening the cost of this beverage and defrauding the consumers. In the manufacture of these so-called substitutes and in the adulteration of genuine coffees, chicory, cereals, etc., occupy a prominent place. These substances have little, if anything, in common with coffee and possess none of the latter's valuable properties. It is the purpose of this report to deal with methods for the detection of these substances in considerable detail, but first certain statistical and other data will be considered.

STATISTICS OF COFFEE CONSUMPTION.

The following statistical statements are based on the Report of the United States Treasury Department:¹

Imports of coffees, less the amount exported.

	Pounds.
1887	500,819,587
1888	408,562,775
1889	561,132,100
1890	490,181,755

The per capita consumption is approximately 7.8 pounds.

In addition to the consumption of pure coffee, there is a very large quantity of so-called substitutes, chicory, etc., sold and consumed as coffee. It is impossible to obtain statistics in regard to these substitutes and adulterants. The high prices of coffee prevailing the past year or more have induced certain unscrupulous dealers to impose spurious coffee beans upon the public and to adulterate ground and other coffees to such an extent that often samples may be purchased having little in common with the genuine.

The fluctuations in the prices of coffees are shown in the accompanying table. The values of the coffees are those which they bore in the markets of the countries from which imported. This does not include the cost of transportation, etc.

¹ Commerce and Navigation of the United States.

Average price of coffee from 1878 to 1890, inclusive.

Year.	Value per pound.	Year.	Value per pound.
1878	\$0. 168	1885.....	\$0. 082
1879.....	. 125	1886.....	. 076
1880.....	. 135	1887.....	. 107
1881.....	. 125	1888.....	. 140
1882.....	. 100	1889.....	. 129
1883.....	. 082	1890.....	. 160
1884.....	. 093		

The coffee consumed in the United States is principally supplied by Brazil, Venezuela, the Central American States, México, and Colombia. The quantities supplied by these countries are respectively 63, 12, 6½, 4, and 3½ per cent of the total imported. Very little coffee is imported directly from Africa.

GENERAL STATEMENTS.

Of the following statements those in quotation marks are from the observations of Lascelles:¹

"West India coffee is for the most part even-sized, pale, and yellowish, firm and heavy, with fine aroma, losing little in weight by the roasting process."

"Brazil coffee is larger, less solid, greenish or white, usually styled by the brokers 'low' or 'low middling.'"

"Java coffee is smaller, slightly elongated, pale in color, deficient in aroma and essential oil, and light."

"Ceylon produces coffee of all descriptions, but the ordinary plantation coffees are even-colored, slightly canoe-shaped, strong in aroma and flavor, of considerable gravity, and admit better of adulteration than most other kinds."

Mocha is usually considered the best coffee of commerce. It is stated that East India coffees are sometimes shipped to Arabia and exported from this latter country as genuine Mocha coffee. The seeds of the Mocha are small and dark yellow.

Java coffee when new is a pale yellow and is then cheaper than when old and brown. This color is partly a result of the method of curing in addition to the effects of age.

The high price of Java has led to the coloring of cheaper grades with mineral pigments or otherwise, in imitation of this favorite coffee. It may be well to state that this practice can not be general, since no foreign coloring matters were found in the Javas examined in the course of the investigations treated of in this work, though it is probable that coffees colored by exposure to a high, moist heat may have escaped detection.

¹ The Nature and Cultivation of Coffee, Arthur R. W. Lascelles. London: Sampson Low, Son & Marston.

The following table, by Thorpe,¹ indicates the variations in the size of coffee beans:

Number of seeds in a measure holding 50 grams of water.

Fine brown Java	187
Fine Mysore	198
Fine Neilgherry	203
Costa Rica	203
Good ordinary Guatemala	207
Good La Guayra	210
Good average Santos	213
Fine long-berry Mocha	217
Good ordinary Java	223
Fine Ceylon plantation	225
Good average Rio	236
Medium plantation (Ceylon)	238
Manilla	248
Ordinary Mocha	270
West African	313

Rio coffees form a very large proportion of those consumed in the United States. Judging from the above table, the Rio coffee bean is considerably smaller than the Java and is approximately the size of the Mocha.

CHEMICAL COMPOSITION.

In preparing the tables on the following pages, showing the composition of coffees, the better known authorities have been consulted and analyses have been selected which give the principal constituents of the grades usually found in our markets.

The estimation of the sucrose in sample No. 8712 was accidentally omitted. Judging from other analyses made in the Division of Chemistry the average per cent sugars given by König is rather high. Considerable quantities of pure sucrose have been separated from coffees in the course of these investigations. It has been definitely determined that the soluble carbohydrates of coffee consist very largely of sucrose.

The caffetannic acid in No. 8712 was estimated by a method described on page 908. Many of the statements in regard to this constituent are very indefinite; no description of the methods for its estimation could be found in the literature accessible.

¹ Dictionary of Applied Chemistry, p. 578.

Table showing the chemical composition of unroasted coffees.

Description.	Moisture.	Est.	Crude fiber.	Total ash.	Total nitrogen.	Alkaloidal nitrogen.	Albuminoid nitrogen.	Caffeine.	Albuminoids.	Sugar.	Dextrin.	Caffeotannic acid.	Remarks.
	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per cent.	Per cent.	Per cent.	Per cent.	
Java coffee, serial No. 8712.	6.55	13.48	31.50	4.18	2.15	.46	1.69	1.58	10.56			10.88	U. S. Department of Agriculture.
Do.....		14.42	33.71	4.47	2.30	.49	1.81	1.69	11.31			11.64	Calculated to dry matter.
Brazil coffee (old).....	11.22	14.27		3.51				1.18	6.96				Church. ¹
Do.....	12.07	14.06		3.75				1.75	12.19	6.36		7.01	Ludwig. ²
Brazil coffee (new).....	11.65	14.10		3.55				1.16	13.92	5.96		5.84	Do.
Not given.....	8.26	11.42	42.36	3.97				1.10	10.68	8.18		14.03	Hassall. ⁴
Mocha.....	8.98	12.60	(⁶)37.95	3.94				1.08	9.87	9.55	.87		James Bell. ⁷
East India.....	9.26	11.81	(⁶)38.60	3.98				1.11	11.23	8.90	.84		Do.
Java coffee.....	13.81	12.17	16.61	3.98				1.48	13.68	7.40		62.35	W. Kisch. ³
	11.23	13.27	18.17	3.92				1.21	12.07	8.55		63.79	König's averages. ⁷

¹ From Zusammensetzung der menschlichen Nahrungsmittel, J. König.⁴ Food, Its Adulterations and Methods for its Detection, A. H. Hassall, p. 146.⁵ Includes cellulose and insoluble coloring matter.⁶ Nitrogen, free extract matter.⁷ König op. cit., p. 1002.² Loc. cit.³ Includes caramel gum, tannin, etc.

The chemical composition of roasted coffees.

Description	Moisture.	Fat.	Crude fiber.	Total ash.	Caffeine.	Albuminoids.	Other nitrogen-free extract matter.	Sugar, gum, and dextrine.	Dextrine.	Remarks.
Not given	<i>Per ct.</i> .36	<i>Per ct.</i> 8.30	<i>Per ct.</i> 44.96	<i>Per ct.</i> 5.17	<i>Per ct.</i> 1.06	<i>Per ct.</i> 12.03	<i>Per ct.</i> 26.28	<i>Per ct.</i> 1.84	<i>Per ct.</i>	Hassall. ²
Do.....		16.14	25.07	3.87	1.42	12.31	39.84	1.35		Blyth. To dry substance. ³
Do.....	3.19	15.63	24.27	3.75	1.38	12.05	38.41	1.32		Blyth. To original substance. ³
Mocha.....	.63	13.59	48.62	4.56	.82	11.23		.43	1.24	James Bell. ⁴
East India.	1.13	13.41	47.42	4.88	1.05	13.13		.41	1.38	Do.
Java.....	1.92	16.51	18.42	4.91	1.44	17.18	38.61	2.45		W. Kisch. ⁵
	1.15	14.48	19.89	4.75	1.24	13.98	45.09	.60		König's averages. ⁶

¹ Reported as cane sugar.² Hassall, *op cit.*, 146.³ Foods, their composition and analysis, A. W. Blyth, 346.⁴ König, *op cit.*, 1002.⁵ *Loc. cit.**Composition of some pure coffees.*

[A. Smetham, Analyst, 1882. 73.]

	1.	2.	3.	4.	5.	6.	7.
Water per cent.	3.89	3.49	1.84	3.54	1.59		
Fat do..	12.13	11.40	10.13	10.63	10.13	11.75	10.80
Cellulose do..	26.33	27.50	34.40	30.26	27.90		
Ash do..	4.63	4.29	4.40	4.08	4.19	4.25	4.20
Soluble ash do..	3.34	3.50	3.60	3.14	3.40	3.25	3.35

No. 1, Ceylon coffee; No. 2, Costa Rica; No. 3, Ceylon; No. 4, East Indian coffee; No. 5, Jamaica; Nos. 6 and 7, best and purest kinds of coffee bought from a wholesale dealer.

Analyses of various coffees.

[O. Levesie, Arch. Pharm. (3) 8 294.]

	1.	2.	3.	4.	5.	6.	7.
Caffeine per cent.	1.43	.64	1.53	1.14	1.18	.88	1.01
Fat do..	14.76	21.79	14.87	15.95	21.12	18.80	17.00
Gum do..	25.30	22.60	23.80	27.40	20.60	25.80	24.40
Caffeic and tannic acid do..	22.70	23.10	20.90	20.90	21.10	20.70	19.50
Cellulose do..	33.80	29.90	36.00	32.50	33.00	31.90	36.40
Ash do..	3.80	4.10	4.00	4.50	4.90	4.30	
Potash do..	1.87	2.13					
Phosphoric acid do..	.31	.42	.27	.51	.46	.60	

No. 1, best Jamaica; No. 2, best green Mocha; No. 3, pearl Ceylon; No. 4, washed Rio; No. 5, Costa Rica; No. 6, Malabar; No. 7, East Indian coffee.

*The ash of coffees.**

Constituents.	Serial No. 8874, Mocha, 38 c. per lb.	Serial No. 8875, Maracai- bo 33 c. per lb.	Serial No. 8876, Java 38 c. per lb.	Serial No. 8877, Rio, 30 c. per lb.
Sand per cent †..	1.44	0.72	0.74	1.34
Silica (SiO ₂) ‡do....	0.88	0.88	0.91	0.69
Ferric Oxide (Fe ₂ O ₃)do....	0.89	0.89	1.16	1.77
Lime (CaO)do....	7.18	5.06	4.84	4.94
Magnesia (MgO)do....	10.68	11.30	11.35	10.60
Potash (K ₂ O)do....	59.84	61.82	62.08	63.60
Soda (Na ₂ O)do....	0.48	0.44		0.17
Phosphoric acid (P ₂ O ₅)do....	12.93	13.20	14.09	11.53
Sulphuric acid (SO ₃)do....	4.43	5.10	4.10	4.88
Chlorine (Cl)do....	1.25	0.59	0.73	0.48
	100.00	100.00	100.00	100.00

* The descriptions of samples are based on the dealer's statements.

† Numbers given are parts in 100 of mineral matter after deduction of carbon dioxide. Oxygen equivalent to chlorine is not deducted.

‡ Soluble in solution of sodium carbonate.

One of the most important constituents of coffee is the alkaloid caffeine. This alkaloid is identical, chemically and physiologically, with that present in tea and termed "theine." Coffee contains a much smaller proportion of this alkaloid than is found in tea leaves. Caffeine was discovered in 1820 by Runge,¹ and was isolated some time afterwards by Pelletier,² Robiquet,³ Caventon, and Garrot. This alkaloid is also found in the leaves of the coffee tree. According to Payen⁴ caffeine is present as a caffetannate. Caffeine may readily be obtained from coffee by extraction with chloroform. The alkaloid separated in this way, after purification, is obtained on evaporating the solvent in beautiful white silky, fibrous crystals. Unroasted coffees contain from a little less than 1 per cent to approximately 2 per cent of caffeine. Payen⁵ reports from 3.5 to 5 per cent caffeine plus caffetannate of potassium, the free alkaloid amounting to .8 per cent. Caffeine⁶ melts at 224° to 228° C., sublimes at 187° C., is soluble in 58 parts of water at 20° C., in 9.5 parts of water at 100° C., in 21 parts alcohol, in 545 parts of ether and in 9 parts of chloroform at 20° C. According to Lapean⁷ the solubility of caffeine in alcohol at 15.5° C. is 1 part in 150 of the solvent. ⁸Blyth cautions the analyst against drying this alka-

¹ Schweigg. Jour. Chem. Phys. **31**, 308.

² Jour. Pharm. [2], **12**, 229.

³ *Op. cit.*, 234.

⁴ Ann. chim. phys. [3] **16**, 108; Jahresb. ii. d. Fortsch. d. Chem. 1849, 486.

⁵ Ann. chim. phys. [3] **26**, 108; Jahresb. ii. d. Fortsch. d. Chem. 1849, 486.

⁶ Vierteljahreschr. pr. Pharm **16**, 167.

⁷ Pharm. J. Trans. [3], **11**, 902; Jahresb. d. Chem. 1881, 902.

⁸ Foods: Their composition and analysis. A. W. Blyth.

loid at 100° C. as is usual, and claims that it commences to sublime at 79° C. The specific gravity¹ of caffeine at 10° C. is 1.23. The following reactions are given by Luchini:² Wenzell's reagent (solution of 1 part KMnO_4 in 200 parts of hot H_2SO_4), gives an amethyst color with caffeine, which changes to a dark violet, then becomes blood red and after twenty-four hours a brown precipitate forms: 1 part caffeine in 10,000 can be detected by this reagent. With Luchini's reagent (a hot solution of $\text{K}_2\text{Cr}_2\text{O}_7$ in concentrated H_2SO_4 there is no change on standing twenty-four hours. According to Böttiger³ caffeine may be detected by evaporating an alcoholic extract of the substance to dryness, treating with hydrochloric acid, again drying, then adding water. The presence of the alkaloid is shown by a purple-red color. Schwarzenbach⁴ employs chlorine water instead of hydrochloric acid in the above test. The purple residue becomes yellow on heating and red when treated with ammonia.

Kornauth⁵ evidently overestimates the percentage of caffeine in coffees. He states that a percentage of caffeine below 1.97 is an indication of an adulterated sample. The writer's analyses and those quoted in the table (p. 902) all show percentages of caffeine considerably below the limit given by Kornauth.

The fat is a prominent constituent of coffee. Husemann⁶ separated a white, odorless fat on cooling an alcoholic extract of coffee. The melting point of the fat was 37.5° C.; it consists⁷ of the glycerides of palmitic acid and of an acid ($\text{C}_{12}\text{H}_{24}\text{O}_2$). The percentage of fat in raw coffee is approximately 13.

The caffetannic acid of coffee has been isolated by Pfaff⁸ and by Rochleder.⁹ The latter chemist also reported the presence of traces of citric acid. Caffetannic acid as isolated by W. H. Krug in connection with this Department's investigations agrees with the description given by Beilstein.¹⁰ It is a light yellow, sticky mass, with a slightly acid and astringent taste. According to Hlasiwetz¹¹ it is a yellow bitter mass of the formula $\text{C}_{15}\text{H}_{18}\text{O}_8$.

Bonssingault¹² found in a sample of coffee 2.21 per cent mannite, 8.73 per cent invert sugar, and 2.37 per cent sucrose.

The carbohydrates of the coffee berry have received very little

¹ Ann. Chem. Pharm., **1**, 17.

² Arch d. Pharm. [3 R.], **23**, 684; Ztschr. anal. Chem. **25**, 565.

³ Pol. Notizblätter, 1873, 257; Ztschr. anal. Chem. 1873, 442.

⁴ Chem. Centrbl., 1861, 989; Ztschr. anal. Chem. **1**, 229.

⁵ Mittl. a. d. Pharm. Inst. n. Lab. f. angew. Chem., Erlangen, Heft. 3, 1-56.

⁶ Pflanzenstoffe, p. 1367.

⁷ Wien. Akad. Ber., **24**, 40.

⁸ Schweigg. Journ., **62**, 31.

⁹ Wien. Acad. Ber., **7**, 815.

¹⁰ Organische Chemie, Beilstein, **3**, 343.

¹¹ Hlasiwetz, Ann. Chem. Pharm., **149**, 219.

¹² Compt. rend., **91**, 639.

thorough study until quite recently. The recent investigations and discoveries in this line by Dr. Schulze at Zürich, and Dr. Tollens at Göttingen, and their collaborators, have brought about a more careful study of the carbohydrates of coffee among a host of other vegetable materials. Thanks to their labors, the percentages of compounds included in the term "other non-nitrogenous substances" have already been materially lessened in a great many cases.

Rather indefinite statements about sugar, gum, and dextrin, make up the existing literature of the carbohydrates of coffee which are soluble in water. The coffee berry contains no starch. Mr. Walter Maxwell¹ has demonstrated the presence of an insoluble carbohydrate which yields galactose by hydrolysis, and has succeeded in obtaining a considerable portion of very pure and well crystallized galactose. R. Reiss² has reported mannose as an hydrolysis product of an insoluble carbohydrate of coffee.

By investigations made in this laboratory, cane sugar has been shown to be the principal soluble carbohydrate present. It is accompanied by a small percentage of a substance closely resembling dextrin and some reducing sugar. The latter may be due to the inversion of a small amount of the cane sugar before or during the process of extraction. A considerable amount of cane sugar was obtained in pure, well-defined crystals. For the purpose of isolating it from other soluble substances of the berry, the extract obtained by the use of 60 to 70 per cent alcohol is treated with a slight excess of lead acetate and the excess of the latter removed from the filtrate by means of hydrogen sulphide. The sugar is now converted into strontium saccharate by treatment with strontium hydroxide at the boiling point of the liquid. The precipitate of saccharate is separated by filtration, suspended in water, and decomposed by a current of carbon dioxide. The filtrate from the strontium carbonate thus formed is evaporated to a heavy sirup. This sirup is purified by repeated solution in alcohol, reëvaporation and resolution, gradually increasing the strength of the alcohol. The final solution in very strong alcohol is left to crystalize. For this method we are indebted to Schulze, Steiger, and Maxwell.³

Just as satisfactory a preparation of cane sugar was obtained by the evaporation of the filtrate from the precipitated lead sulphide and by direct treatment of the residue with alcohol without the use of strontium hydroxide. A preparation is now in progress without the use of either strontium hydroxide or lead acetate, and promises very good results, the separation being made by use of alcohol alone.

The portion of coffee insoluble in water is also being made the subject of detailed study. By distillation with hydrochloric acid an abun-

¹ Unpublished notes.

² Ber. d. d. chem. Gesell., 1889, 22, 609.

³ Untersuchung über die chemische Zusammensetzung einiger Leguminosensamen. Landwirt, Versuchs-Stat., 1891, 39, 269.

dance of furfuraldehyde was obtained, which indicates the presence of some pentose yielding substance. By treatment of a considerable portion of the water-insoluble material with a 5 per cent solution of sodium hydroxide, and by precipitation of the extract thus obtained with alcohol,¹ a gummy substance was obtained. This gum yielded mucic acid by oxidation with nitric acid, an indication of the presence of a galactose yielding carbohydrate; by distillation with hydrochloric acid furfuraldehyde was also obtained, showing the presence of a pentose yielding substance in the gum. A galactose and pentose yielding gum has been separated from barley.² Whether the gum obtained from coffee is a mixture of galactose and pentose yielding substances, or is a single substance yielding both, has not been determined.

The residue obtained by evaporation of the sulphuric acid extract of the insoluble material yields an abundance of mucic acid when oxydized with nitric acid, showing the presence of galactose, as stated by Maxwell.

Satisfactory evidence of the presence of any considerable amount of mannose in the sample of coffee used in the investigation has not yet been obtained. Parallel experiments with coffee and vegetable ivory dust were made. With the latter, an abundant precipitate of mannose hydrazone was obtained; with coffee, the precipitate was not of sufficient amount for a satisfactory determination of its properties.

Dierbach³ isolated the coloring matter of coffee and termed it "coffee-green."

Coffee also contains traces of volatile oils and about 4 per cent of mineral matter. Potassium salts form a considerable proportion of the mineral matter. Silica, when present, amounts to little more than a trace.

Kornauth⁴ states that in the ash of pure coffees the potash amounts to from 50 to 200 times the soda.

According to Bernheimer,⁵ the products obtained on roasting coffees are palmitic acid, caffeine, caffeol, acetic acid, carbonic acid, hydrochinon, methylamine, pyrol, and acetone. Caffeol is an oil, of the formula $C_2H_{10}O_2$, boiling at a temperature of from 195° to 197° C.

A large number of analyses of coffees have been made, references to which are given in the bibliography accompanying this report.

METHODS OF ANALYSIS.

In the detection of adulterants but little chemical work is necessary, hence methods will only be given for the estimation of the principal constituents of coffee.

Moisture.—Dry in a flat dish to constant weight and calculate the moisture as usual.

Caffeine.—The method of estimating this alkaloid in tea (p. 889) may be employed.

¹ A method used for the separation of xylan from wood.

² Lintner and Düll, Chem. Ztg., 1891, 266.

³ Ann. d. Chem. u. Pharm., **14**, 236.

⁴ Mittl. a. d. Pharm. Inst. u. Lab. f. angew. Chem., Erlangen. Heft 3, 1-56.

⁵ Wien. Akad. Ber. (2 Abth.), **81**, 1,032; Jahresb. d. Chem. 1880, 1069.

Fat.—Extract 2 grams of the finely powdered coffee in Soxlet's apparatus, or in one of the various modifications of this apparatus, with anhydrous alcohol-free ether. Remove the ether from the extract by distillation and weigh the residue. Estimate the caffeine in this residue and deduct. The caffeine will probably amount to a small fraction of a per cent.

The sample of whole coffee beans may be prepared for analysis by means of a file or rasp. Particles of iron in the raspings should be removed by means of a magnet.

Albuminoid nitrogen.—(See p. 890.)

Crude fiber.¹—A quantity (6 to 8 grams) of the finely ground material is weighed into a flask, and treated several times with very dilute caustic potash (0.2 per cent), in order to remove the greater part of the albuminoids. Boil several times with a somewhat stronger solution of caustic potash (1.25 per cent), and after total removal of the alkali boil repeatedly with acetic acid. The residue after thorough washing with water is dried at 105° C. and weighed.

Caffetannic acid.—Owing to the lack of a method for the estimation of caffetannic acid, the following was devised by Mr. W. H. Kring, of the Chemical Division, for this report:

Weigh out duplicates of 2 grams each. Add 10 cc. of water and digest for 36 hours. Then add 25 cc 90 per cent alcohol, and digest for 24 hours more. Filter, washing the residue on the filter with 90 per cent alcohol. The filtrate contains tannin, caffeine, and traces of coloring matter and fat. Heat it to the boiling point and add a boiling concentrated solution of lead acetate. If this is carefully done a caffetannate of lead containing 49 per cent lead will be precipitated. As soon as the precipitate has become flocculent it is collected on a filter, washed with 90 per cent alcohol until the washings show no lead with ammonium sulphide, washed with ether to remove traces of fat, dried and weighed. The composition of the precipitate is $Pb_3 (C_{15} H_{15} O_{82})$. Therefore weight of precipitate: weight of caffetannic acid :: 1263.63: 652.

Total soluble and insoluble ash.—Refer to page 891 under the analysis of teas for methods of ash determinations.

The color of the ash should be noted. A red ash is indicative of the use of venetian red or other iron compounds in coloring the sample.

ADULTERATION—DEFINITION.

Referring to the law of the State of New York (p. 879), the adulteration of coffees may be defined as follows: The addition of foreign matter of any kind to reduce the strength or affect the quality; the substitution of cheaper substances in part or wholly for the genuine coffee; facing or coloring in imitation of better grades or to conceal damage.

The use of cereals in so-called "blending" should be considered an adulteration. The cereals act simply as a diluent, increasing the weight and bulk without corresponding benefit to the purchaser. These remarks might also well be applied to chicory, were it not that many persons show a decided preference for coffee containing this substance. Notwithstanding this preference, chicory is and should be considered an adulterant, except when the package containing the mixture is distinctly branded and the proportions of pure coffee and chicory indicated. It is stated that chicory possesses medicinal properties of a diuretic and laxative character which render its excessive use objec-

¹ Unpublished work of Mr. W. Maxwell, U. S. Department of Agriculture.

tionable. There is not sufficient evidence against chicory to warrant placing it in the list of objectionable substances. Obvious adulterants of coffees are the so-called substitutes molded in imitation of the genuine beans.

ADULTERANTS AND THEIR DETECTION.

Facing or coloring.—It is not an uncommon practice to treat inferior or damaged coffees by some process for the improvement of their appearance and in imitation of superior grades. Java seems to have been especially subject to this treatment, or rather other coffees are colored in imitation of Java. E. Waller states¹ that South American coffees are often exposed to a high, moist heat, which changes their color from green to brown, thus forming imitation Java. Waller also mentions the use of pigments in coloring coffees. This chemist found one twenty-fourth grain of Scheele's green per one-half ounce of coffee. He also reports the use in the Brooklyn mills of yellow ochre, silesian blue, chrome yellow, burnt umber, venetian red, drop black, charcoal, and French black. Coffee is polished by rotation in cylinders with soap-stone.

The following² is another method of preparing imitation Javas. Raw coffee, which has been damaged by sea water, is washed, decolorized with lime water, again washed, rapidly dried, and colored by a slight roasting or by means of azo-orange. By this method Santos coffees are converted into imitation Javas. The weight lost is regained by steaming, and then coating the beans with glycerine, palm oil, or vaseline to prevent evaporation.

Coffees are sometimes faced with Prussian blue or indigo, lead chromate, etc. The following list of facing mixtures is from the published investigations of K. Sykora.³

(1) Mixture of indigo, lead chromate, coal, and clay.

(2) (Approximately) 5 parts indigo, 10 parts coal, 4.5 parts lead chromate, 65.5 parts clay, and 15 parts ultramarine.

(3) (Approximately) 5 parts indigo with some yellow dye, 3 parts coal, 8 parts lead chromate, 82 parts clay, 2 parts ultramarine.

(4) (Approximately) 12 parts indigo and some yellow dye, 5.5 parts coal, 4.5 parts lead chromate, 6.6 parts clay, and 12 parts ultramarine.

A mixture examined by G. C. Wittstein⁴ was composed of 15 parts Prussian blue (or indigo), 35 parts lead chromate, 35 parts clay and gypsum, and 15 parts water. According to Nanning, coffee beans are colored blue by shaking with finely powered iron.

Indigo and Prussian blue may be detected by the microscope or chemically (see page 881 for methods). Lead chromate should be ex-

¹ Analyst, **9**, 128.

² Bull. de la Soc. Chim. de Paris, **47**, 7; Chem. News, **56**, 24.

³ Chem. Centrbl., 1887, No. 47; Rep. f. anal. Chem., 1887, 765.

⁴ Chem. News, **33**, 194.

aminated for in the ash by the usual qualitative methods for the detection of lead and chromium. The ash should also be examined for copper, and in the case of moist preparations of coffee preserved in tin cans, both tin and copper should be searched for. Azo-colors are detected as follows:

Azo-colors are detected by treating the beans with strong alcohol, evaporating the solution to dryness, and treating the residue with water. This solution will give the characteristic reactions of these dyes.

It is preferable, when possible, to detach the facing by shaking the coffee with cold water. The sediment may be examined chemically or microscopically. Lead, tin, copper, and arsenic are the only objectionable metals liable to be present in coffee or its preparations. The ash should be examined for these metals.

Chicory.—One of the common adulterants of coffee is the prepared root of the chicory plant, *Cychorium intybus*. There are several chemical methods for the detection of chicory, depending upon positive and negative tests. Ground chicory when thrown on cold water sinks quickly, coloring the water, and is soon softened, whereas ground roasted coffee floats, imparting no color. Chicory is easily bleached by chlorinated soda (labarraque solution); coffee is but slowly affected by this bleaching agent. The coloring¹ matter of chicory is not precipitated by iron salts, while that of coffee is colored green and is partially precipitated. G. C. Wittstein² employs the following method:

Boil 30 drops of the coffee infusion in a test tube with 2 drops of concentrated hydrochloric acid; add 15 drops potassium ferrocyanide solution (1 part of the salt to 8 of water), and again boil until the liquid becomes a dark green; add 6 drops of potassium hydroxide solution and boil; if chicory is present the liquid will become brown and murky, otherwise a precipitate will separate and settle to the bottom of the tube, leaving the supernatant solution of a light-yellow color.

A. Franz³ states that copper acetate gives a greenish-brown precipitate with coffee infusions and a dark-red brown precipitate with chicory. With coffee the supernatant liquid is greenish and with chicory red brown.

Hiepe⁴ tests for chicory as follows: Ignite 25 grams of the sample and determine the amount of chlorine present in the ash. Coffee contains 0.03 per cent chlorine, and chicory as high as 0.28 per cent. Kornauth⁵ gives the maximum and minimum chlorine content of coffee as respectively 0.06 per cent and 0.15 per cent.

Chicory can be most readily and certainly identified in mixtures by means of the microscope. The microscopic appearances of coffee and chicory are shown in Plates XLII, XLIII, and XLV.

¹ Ding. Polyt. Journ., **211**, 78; Jahresh. d. Chem., 1874, 1043.

² Ding. Polyt. Journ., **215**, 84; Am. Chemist, **6**, 220.

³ Arch. Pharm. [3], **8**, 298; Jahresh. d. Chem., 1876, 1021.

⁴ Moniteur Scientifique [3], **10**, 1339; Jahresh. d. Chem., 1880, 1220.

⁵ Mitt. a. d. Pharm. Inst. u. Lab. f. Angew. Chem. Erlangen, Heft 3, 1 to 56.

The quantitative estimation of chicory or of the amount of coffee in mixtures can not be made with certainty, and in all such estimations only an approximate percentage content can be stated. The proportion of chicory in a mixture is usually calculated from the specific gravity of an extract made under definite conditions, comparison being made with a coffee extract prepared under the same conditions. In other methods a comparison is made of the depth of color of infusions under certain standard conditions.

According to A. H. Allen¹ the following method gives fairly good results:

A weighed portion of the finely ground sample is boiled with water, filtered, and the residue washed with hot water until the filtrate amounts to 10 cc. per gram of the sample taken. The specific gravity of this extract is then determined and the chicory calculated by the formula.

$$c = \frac{(1.023 - d)}{14.5} 100$$

in which c is the per cent of coffee, d the density of the extract. Allen found the mean density of the 10 per cent decoction of a large number of coffees to be 1.0085, and of chicory under the same conditions, 1.023.

The presence of cereals or other foreign matter would of course render this method unreliable.

Prenier² states that—

Chicory may be estimated by sifting a definite weight, approximately 2 grams of the ground mixture; the powder which passes the sieve is always coffee. The larger grains are macerated with cold water for some hours, then thrown on a piece of stretched cloth and rubbed with a pestle. The chicory will pass through the cloth. The residue left on the cloth is now dried and weighed with the powder.

This is evidently a very rough method.

Another method for the estimation of chicory, and as rough as the above, is that of C. Draper.³ In this method a glass similar in shape to a percolator is employed. The stem is graduated and is sealed at the lower end. This apparatus is partly filled with cold water, and a definite volume of the ground sample is slowly distributed upon its surface. The chicory sinks to the bottom of the water and its depth is noted in the graduated tube.

The writer has found pure coffee, evidently overroasted, that would sink in water. Other writers have also found coffee that would sink, after a few minutes, in water. In case the adulterant has been treated with a fat, it would be liable to float instead of falling to the bottom of the apparatus. Chicory is often so treated.

The cells, milk vessels, etc., are well illustrated in the Plate XLV. The milk vessels, *vasa lacticentia*, should be carefully studied, since their peculiarities will usually serve to distinguish chicory from other roots liable to be employed as adulterants.

Mangoldwurzel.—This is a root much used abroad for cattle feeding.

¹ Chemical examination of coffee.—Chem. News, 29, 129, 140, 167, 189, 221. *Op. cit.* 30, 2.

² Journ. Pharm. Chim. [5] 1, 222–224; Journ. of the Chem. Soc. 1880, 514.

³ Philos. Mag. 38, No. 228, 104; Zeitsch. f. anal. Chem. 7, 388.

According to Hassall,¹ the large size of the cells and the absence of milk vessels distinguish this root from chicory.

Cereals, leguminous seeds, and acorns.—Judging from these investigations the adulteration of coffees in this country with chicory is not as common as with cereals, pease, beans, etc. These latter substances, in general, are detected by the presence of starch and are finally identified by their structures as shown by the microscope.

The following method of A. H. Allen is probably the best for the detection of starches, chemically, in adulterated coffees:

Boil a portion of the powdered sample with water and filter; cool the filtrate, acidulate with sulphuric acid; add a strong solution of permanganate of potassium, small quantities at a time, until the coloring matter of the original extract is discharged; test for starch as usual with iodine.

Starch may be easily detected in the finely powdered sample, mounted in Canada balsam, on examination by the microscope, with polarized light. Starch, when examined as above, shows a dark cross on a white field.

In Part II of this bulletin (Plates XIII to XXVIII), a number of starches are illustrated as shown by the microscope with polarized light and plain illumination. In the identification of starches it is well to have a series of freshly-prepared slides of the materials from known sources for purposes of comparison.

Sufficient of the original structures of the cereals or leguminous seeds will usually remain for their identification with the microscope. In this work it is well to have a series of slides prepared from the raw and roasted materials for comparison.

The following statement of the percentage of ash in cereals is given for comparison with that of coffees. The mean ash of the cereals given is considerably lower than that of coffee.

Ash² of cereals—(American).

Cereals.	Mean.	Highest.	Lowest.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Wheat.....	1.9	3.6	0.8
Corn.....	1.5	3.1	1.0
Barley.....	2.9	4.4	1.5
Oats.....	3.2	2.9	.9
Rye.....	2.1	3.7	1.3

Canna seed, etc.—M. Mansfeld³ reported the presence of canna seed in a coffee, and in an examination made in Austria, sawdust, oak-bark, baked liver, etc. Hassall⁴ mentions the use of sawdust, etc., in the adul-

¹ Food, Arthur H. Hassall, Longmans, Green & Co., London, p. 167.

² Dictionary of Applied Chemistry, Thorpe, 1, 490. Analyses by Clifford Richardson, U. S. Department of Agriculture.

³ Rev. Int. Scien. et Pop. des Falsification, 4, 40.

⁴ Hassall, Food, p. 159.

teration of coffees. None of these substances, except sawdust, have been detected in the investigations in connection with this report. Chemical tests are not applicable. On the detection of a foreign matter, which is not among those described, evidently the only course to pursue is to prepare slides for microscopic comparisons with others of known composition.

Mogdad coffee.—This adulterant or substitute consists of the seeds of the *Cassia occidentalis*.¹ E. Geissler² examined Mogdad coffee by Hager's method and found that it sinks very rapidly in water and colors sodium chloride solution more intensely than coffee does; its infusion is not indifferent to ferric chloride or tannic acid; it contains no starch. Janceek³ analyzed Mogdad coffee and found a different tannin from that in the seed of *Coffea Arabica*, but no caffeine.

Mussaenda coffee.—This⁴ substance was supposed to be seeds derived from *Mussaenda borbonica*, but later investigations made at Kew Gardens show these seeds to be from *Gaertnera vaginata*. It is stated that *Mussaenda* coffee contains no caffeine.

Cocoa husks.—Cocoa husks may be identified by the methods given under cocoa preparations.

Sugar and sirup.—Coffees are sometimes treated with sugar or sirup, then roasted. When the caramel formed on roasting amounts to an appreciable weight, it should be considered an adulterant. Stutzer⁵ and Reitnair recommend the following method for the examination of coffees supposed to have received this treatment: 20 grams of whole coffee beans are transferred to a litre flask, covered with 500 cc. water and the flask is then violently shaken for five minutes. After shaking complete the volume to 1,000 cc., mix and filter off 50 cc. of the solution into a tarred dish; evaporate to dryness on a water-bath, then transfer to an oven heated to 95°–99° and dry two hours; weigh the residue, incinerate, and deduct the ash before calculating the organic matter extracted. Pure roasted coffee treated by this method gave from 0.44 to 0.72 per cent organic matter, and colored the water only slightly, while coffees which had been roasted with sugar colored the water more or less strongly and gave from 1.81 to 8.18 per cent organic extract.

The glazing of coffees, according to König,⁶ is objectionable, not that the glazing material is unwholesome, but because coffees so treated retain an excess of moisture in the roasting process. The following comparative analyses quoted by König show the effect of roasting with and without sugar. The sirup employed for glazing was simply a solution of starch sugar.

¹ J. Moeller, Pharm. Centralhalle, **22**, 133; Zeitsch. f. anal. Chem., **21**, 438.

² *Op. cit.*, **22**, 134.

³ Chem. Ztg., 1880, 442; Jahresb. d. Chem., 1880, 1070.

⁴ Pharm. J. Trans., Nov. 16, 1889, 381; Am. J. Pharm., **20**, 4, 174.

⁵ Zeitsch. f. angew. Chem., 1888, 701, also *op. cit.*, 1890, 706.

⁶ Zeitsch. f. angew. Chem., 1888, 631.

A.—*Coffees roasted with sugar.*

	I.	II.	III.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moisture	9.91	10.46	4.41
Calculated on the dry substance:			
Soluble matter adhering to the beans	7.72	7.59	5.91
Reducing sugar, calculated as dextrose	1.49	1.49	.91
Total water soluble matter	28.12	27.71	26.07
Fat (volatile oil, ether extract)	12.62	12.34	9.45

B.—*Coffees roasted without sugar.*

	I.	II.	III.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moisture	3.14	2.73	2.79
Calculated on the dry substance:			
Soluble matter adhering to the beans	4.77	4.15	4.43
Reducing sugar present calculated as dextrose	.44	.34	.19
Total water soluble matter	24.09	21.81	25.97
Fat (volatile oil, ether extract)	16.29	13.44	12.06

The large percentage of moisture retained by the glazed coffee and the decided increase in the soluble matters adhering to the beans must certainly prove of considerable profit to the roaster without advantage to the consumer.

SUBSTITUTES FOR COFFEE.

A number of substitutes for coffee have been proposed. Many of these have little claim to be entitled substitutes, since they simply furnish a decoction more or less bitter and of a coffee color.

Substitutes, no matter how nutritious, should never be sold in mixtures with genuine coffee, except in properly labeled packages. The package should be distinctly branded in such a way as to avoid misleading the purchaser and should indicate from what raw material the substitute has been prepared.

Besides chicory, Mogdad, and Mussaenda coffee, acorns, figs, leguminous seeds, and cereals have been employed as coffee substitutes.

In this connection it may be well to call attention to the fact that the bogus coffee, known in Germany as "Kunst Kaffee," is largely imported into this country. That this product is a fraud is evident from the fact that it is molded in imitation of genuine coffee and in mixtures is sold as such.

Kornauth¹ examined chicory and figs, and considers them of considerable nutritive value for the poorer classes.

¹ Rev. Internat. Scient. et. Pop. des Falsifications des Decivee's Aliment, 3, 8; Centralbl. 1890, 605.

¹The fruit of the wax palm (*Corypha cerifera* L., or *Copernicia cerifera*, Mart.), which yields carnauba wax, is used in Brazil for the preparation of a coffee substitute, for which purpose it is roasted in the usual way. Chemical investigation of samples of this fruit procured from Brazil, by König, gave:—

	Raw.	Roasted.
	<i>Per cent.</i>	<i>Per cent.</i>
Water	9.37	3.76
Raw protein	6.54	6.99
Pure protein.....	5.82	6.14
Fat (ether extract).....	10.57	14.06
Sugar and dextrine	1.67	1.25
Starch	2.47	5.46
Non-nitrogenous extract matter	23.01	27.79
Fiber	44.31	38.45
Ash	2.06	2.24
Mean K ₂ O	.63	.69
Mean CaO	.42	.45
Mean P ₂ O ₅	.41	.43
Water soluble matter.....	12.17	12.50

In the raw state the fruits have a stony consistency. Starch could not be detected and, if present, was in very slight quantity. The substance presented in the table as starch was obtained by treating the mass, after extraction with water, with diastase, then heating three hours in a Soxhlet pressure steam oven. The mixture was then filtered, the filtrate inverted with HCl and precipitated with Fehling's solution. The fat from the fruit has apparently a different constitution from the wax from leaves (carnauba wax).

Only a slight amount of water soluble substances are found in the roasted fruit, as is the case with acorns.

IMITATION COFFEES.

Within two or three years the coffee markets have been flooded with imitation coffees. The first official action toward suppressing this fraud in this country was probably that of the New Jersey officers. It is claimed that these fraudulent coffees are no longer to be found in the markets of that State. Reports from dealers in various parts of the country indicate that the sale of imitation coffees has been very general. On the the following page is a list of the imitation coffees examined in connection with this report, together with a description and statement of their probable composition.

¹ J. König, Central-Organ. f. Waarenkunde und Technologie, 1891, 2, 1; Chem. Ztg., 15, 19, Aug., 1891.

Serial No.

6872. Imitation coffee beans, roasted; probably the same composition and manufacture as 8957.
8766. Coffee, bran, and molasses; roasted; not molded.
8767. Bran and molasses; roasted; not molded; samples 8766 and 8767 were obtained through the courtesy of Dr. J. N. Hurty, chemist, Indianapolis, Ind.
8491. Imitation coffee beans, roasted. Composition: Wheat flour. Manufacturer not known, but probably same as 8957.
8859. Imitation coffee beans, roasted. Composition: Wheat flour, coffee, and chicory.
8883. A rather poor imitation of roasted whole coffee beans. Imported as a coffee substitute under the name "Kunst Kaffee." Imported by M. Kliemand, 159 Front street, New York City; manufactured by Erhorn & Dierchs, Hamburg, Germany. Price, 11 cents per pound. Composition: Wheat flour, coffee, and chicory. A German analysis of an imitation coffee termed "Kunst Kaffee" is given on p. 919, by K. Portele.
8884. Imitation roasted coffee beans. Composition: Wheat flour, coffee, and chicory. Manufacturer not known.
8885. Imitation green coffee. This sample contains two kinds of berries, one composed of wheat flour and the other of wheat flour and coffee. Sample was obtained in Philadelphia. Manufacturer not known.
8950. Imitation coffee beans, roasted. Composition: Wheat flour, chicory, and coffee. Manufacturer not known.
8951. Coffee pellets, molded, but not in the form of coffee beans. When mixed with ground coffee would escape the notice of the purchaser; also probably in mixture with whole coffee. Composition: Wheat flour and bran, rye also probably present. Manufactured by the Clark Coffee Company, office 156 State street, Boston; factory, Roxbury, Mass. Price, 6 cents per pound, or 5½ cents in 10-barrel lots. The manufacturers claim that an addition of 33 per cent of these "pellets" to genuine coffee will make "an equal drink to the straight goods." The manufacturers, after making extravagant claims for their product, state, with evident intention to further a fraud, that "it is uniform in color, and can be furnished with any desired color of roast."
8952. Coffee pellets, same manufacture and composition as 8951. Grains lighter color and longer than 8951.
8953. Cracked coffee pellets, same manufacture and composition 8951.
8954. Ground imitation coffee. Contains a small amount of chicory, a considerable proportion of leguminous seeds (peas or beans), and barley, wheat, oats, and fragments of buckwheat. Manufactured by the Swedish Coffee Co., of New York.
8955. Imitation coffee beans. Composed of wheat flour; light roast. Manufactured by the Swedish Coffee Co., New York.
8956. Similar to 8955 and of the same manufacture. Composition: Wheat flour and probably saw dust. Dark roast. Two kinds of berries.
8957. Imitation coffee beans. Composition: Wheat flour. Manufactured by L. H. Hall, 1017 Chestnut street, Philadelphia, Pa.
8958. Granular imitation coffee. Composed of the hulls of leguminous seeds, probably peas, formed into granules with molasses and roasted; source not known.
8963. Imitation roasted coffee beans. Composition: Wheat flour. Probably of the same manufacture as 6872, 8491, and 8957.
8996. Sample package, marked "Coffee Substitute, Columbia AAA." Composed of bran and molasses, formed into small lumps and roasted. Manufactured by E. A. Sibell, 19 and 20 Wabash avenue, Chicago, Ill.
10483. Imitation roasted coffee beans. Composition: Wheat or rye flour and corn. Manufacturer not known.
10484. Imitation green coffee. The same composition and manufacture as 10483.

Serial No.

10515. Granular imitation coffee. Composition: Pea hulls and bran. Manufactured by the Powell Manufacturing Company, 120 Front street, New York. See circular letter, p. 918.

Abstracts and copies of a number of the circular letters sent out to dealers by the manufacturers of spurious coffees may be of interest and value. The following are copied in full with the exception of the address:

DEAR SIR: I send you by this mail a sample of "imitation coffee."

This is a manufactured bean, and composed of flour; you can easily mix 15 per cent of this substitute in with genuine coffee that ranges in price from 20 to 22½ cents, and it will improve the flavor of the same; it granulates the same as coffee. If you deal with us it will be in the most strict confidence.

This S. S. Coffee (Superior Substitute) is packed in barrels, weight about 170 pounds to barrel. By the use of our bean you can increase your profits to 1½ cents per pound and improve the flavor. Try a sample barrel. Price 11½ cents per pound net, ten days. No attention paid to postal cards.

Yours,

L. H. HALL,
Sole Agent.

I would not show samples even to employés.

GENTLEMEN: We desire to call your attention to our improved grade of coffee pellets, which we mail you sample of to-night. The bulk of the goods has been increased, and the drinking merit still further enhanced, as a test of the goods will demonstrate. To exhibit the value of our goods, and show the extent they can be used with good results, we mail you sample of a compound coffee made as follows: 75 per cent pellets, 15 per cent coffee, 10 per cent chicory.

This makes a very desirable cup of coffee, equal to any low grade straight coffee, and costs, on basis of 5 cents per pound for pellets, 7 cents per pound. By the use of 33 per cent of our new pellets in fine goods, the same result is obtained as from the straight goods alone.

We believe a thorough test of the coffee pellets will convince you that the goods are the most valuable adjunct of the coffee business on the market.

We will be pleased to quote prices on quantities.

Yours, respectfully,

CLARK COFFEE COMPANY.

GENTLEMEN: We desire to make a proposition to you, which will be for your interest to consider.

We are manufacturing a coffee substitute which is unquestionably far ahead of anything in its line, which we term "coffee pellets." The merit of these we will not dilate upon, but simply ask that you give them a fair trial. They are put out in size used for crushed coffees, and are the best for that purpose upon the market. We understand you are using peas extensively, and we propose, if we can get your attention on the goods, to compete with that article on a basis of uniform price. Our goods are 4 ounces lighter in bulk to the pound than peas, and double the quantity can be used, with better results. As far as appearance is concerned, of course our goods have considerably the best of it. We mail you sample to-night, and our Mr. Clark will be in your city the first of next week, and we would ask that you see if it is not for your advantage to use our goods.

Hoping you will give it your attention, we remain,

Yours, respectfully,

CLARK COFFEE COMPANY.

"THE SUCCESS OF THE SEASON."

Java coffee compound, 1-pound packages, whole bean, 60 pounds in case; cost, 10 cents per pound; retail it for 15, and give better value than you now do for 24. Send us your order at once. Draft or post-office order must accompany it. If for any reason you wish to return Java coffee compound within thirty days of its receipt, do so at our expense and we refund money. You won't return it, but will order more.

Very truly yours,

THE DOWLING MFG. CO.

Reference, Produce National Bank.

GENTLEMEN: We have sent you by mail a sample of our "coffee substitute," which is being used by the trade very generally in this city. We claim for our goods that, unlike any other mixture in the market, it is positively neutral in character and can be used in larger quantities and to better advantage than any other, while the style is always uniform both in color and grain. Those goods are put up in barrels of about 200 pounds net, and are sold "F. O. B." at 5 cents, less 10 per cent, ten days. We would be pleased to ship you any quantity for a trial, should you desire it.

Very respectfully,

POWELL MANUFACTURING CO.,
Per J. D. M.

(See p. 916, serial No. 10515.)

Abstract from letter of M. Kliemand,¹ 327 Degraw street, Brooklyn.

I beg to mail you a sample of a *coffee substitute*, "Kunst-Kaffee," manufactured by Messrs. Erhorn & Dierchs, Hamburg, who appointed me general agent for the United States.

Advantages.—It is animating, but not exciting, and very nutritious and wholesome, *softening the taste of the inferior coffees*; quality unimpaired for twelve months or longer.

Price.—Eleven cents per pound, New York, net, per Pennsylvania Railroad.

The above so-called coffee substitute (Department serial No. 8883) is admitted at the port of New York as a "substitute for coffee" at the rate of 1½ cents per pound.

Dr. Van Hamel Roos calls attention to the following novel scheme for the sophistication of coffee berries:²

The microscopical examination of a sample, rendered suspicious by its dark color, showed the structure of genuine coffee, but the fat globules, which are always abundant in pure coffee, were almost entirely absent. The ether extract from pure coffee is 13 to 14 per cent. In this sample it was less than 1 per cent. It is evident that the roasted coffee had been treated for the manufacture of coffee extract, after which the grains were roasted a second time with the addition of a little sugar to cover the berries with a deceptive glazing. The dark color of the beans was due to the second roasting.

Owing to lack of time no chemical analyses of artificial coffees were made in connection with this report. A large number of analyses have

¹ Address given by the customs authorities is 159 Front street, New York City.

² *Revue Intern. des Falsifications*, 4, 10, 166, May 15, 1891.

been published in the journals, from which those given in the following table have been taken:

Imitation coffee beans.

Analyst.	Water.	Protein matter.	Fat.	Cellulose.	Sugar.	Extract matter.	Ash.	Caffeine.	Water extract.	Substances forming glucose with dilute sulphuric acid.
Artificial coffee beans: ¹	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
W. Kisch	5.14	10.75	2.19	3.96		76.66	1.20		29.88	
E. Fricke ²		17.90	2.03	10.83	1.99	64.04	2.27	.94	24.85	
Stroyer and Reitner ..	8.30						1.10		34.34	
Monheim and Gilmer ..	2.26	11.46	2.78			1.94	1.77	.55	27.58	
K. Portèle ³	1.46	13.93	3.80	15.83	.71	63.30	2.53	.071	21.53	50.02
Barley coffee:										
C. Kornauth	3.45 6.41	9.38 10.56	3.25 1.04	4.25 10.56	6.18	70.13 68.36	3.36 3.04		31.20 34.37	69.28 67.19

¹ From a tabulation by C. Kornauth, Rev. Internat. Scient. et Pop. des. Falsifications des Dérivées Aliment., 3, 195-196.

² Zeit. f. angew. chem., 1889, 310-311; Chem. centralbl., 1889, 154.

³ Original article, Zeit. f. Nahrungsmitteluntersuch. u. Hygiene, 3, 221-222; Chem. Centralbl., 1890, 135.

Kornauth (*loc. cit.*) states that he has analyzed artificial coffee beans which were very similar to the genuine, having a specific gravity of 1.26. Specific-gravity determinations of the samples examined in connection with this report gave the following numbers: Serial No. 8491, 1.195; No. 8933, 1.073; No. 8859, 1.198; No. 8883, 1.111; No. 8951, 1.119; No. 8952, 1.183; No. 8953, 1.194; No. 8955, 1.211; No. 8956, 1.174 (light-colored grains) and 1.131 (dark-colored grains), and No. 8957, 1.118. In making these determinations a solution of sodium chloride was employed. Twenty imitation coffee beans were immersed in this solution, the density of which was then gradually changed until ten beans floated near the surface and ten at the bottom of the solution. The specific gravity of the modified solution was then taken and recorded as the specific gravity of the imitation coffee.

These specific gravities are only an approximation, owing to variations in the density of artificial coffees even from the same sample.

Kornauth, in the article cited, states that he supposes the imitation coffee to be composed of grape sugar and dextrin, which are mixed with fat and sugar and the mass pressed in forms, roasted, and glazed. He states that imitation coffee beans sink in 40 per cent (by volume) alcohol, while the genuine beans float.

Stutzer and Reitnair suppose that the imitation coffee beans are composed of roasted sugar; König, of wheat bran; Fricke, of sugar and lupine flour; Hanausek, of wheat bran and the refuse from white peppers; Pavlicek, of sugared bran, and Portèle supposes them to be composed of sugar, cereals, and legumes. Fricke considers lupine seeds

harmful. These opinions are from Kornauth's article cited above. The observations of Portèle agree more closely with the results of the investigations of this laboratory.

A recent journal¹ reports the following as the composition of an imitation coffee seized by the Government of Roumania: Coffee grounds (spent coffee), chicory, and pease. The mixture had been molded in a special machine in imitation of coffee beans.

A factory for the manufacture of imitation coffee was recently seized at Lille, France, by the French Government.² The capacity of this factory was 40 to 50 kilos of imitation coffee per day. The composition of the product, as shown by the testimony at the trial of the manufacturers, was as follows: chicory, 15 kilos; flour, 35 kilos, and sulphate of iron, 500 grams.

L. Jammes³ examined a sample of imitation coffee and found it to be composed of acorns and cereals.

DETECTION OF IMITATION COFFEES.

Roasted imitation coffees may usually be very easily detected. As a rule, genuine roasted coffee will float on water, and the artificial product, roasted, will sink; there are, however, exceptions to this. Coffee that has been "overroasted" will sometimes sink in water. The artificial coffees examined in connection with this report, with one exception, sank in water. These remarks apply to whole coffee. Kornauth⁴ states that imitation coffee beans sink in 40 per cent (by volume) alcohol. Certain coffees will also sink in alcohol of this density; hence this alone is not a sure test.

In examining roasted coffee for the imitation product a portion of the sample should be thrown on 40 per cent (by volume) alcohol, and those beans which sink should be subjected to a further examination. The genuine coffee bean always has a portion of the fine membrane with which it was originally invested still adhering in the cleft. This test alone will distinguish the genuine from the imitation coffee beans. On the examination of a section of an imitation coffee bean it may be seen that the structure is uniform, while that of the genuine bean is not. The imitation coffee generally contains starch, a substance which is never present in the genuine beans. If starch is absent the sample should be examined microscopically for chicory or similar roots.

In the examination of a ground sample tests should be made for starch and chicory. A portion should be thrown on cold water. Chicory, if present, will quickly color the water, while cereals will sink, often imparting little, if any, color to the water. It should be noted that coffee, deprived of its oil, will sink in water and that cereals or chicory

¹ *Revue Intern. des Fals.* 4^e Année, N. 11, 188.

² *Revue Intern. des Fals.*, 4^e Année, No. 11, 185.

³ *Chem. Centralbl.*, 1891, 1, 935.

⁴ *Rev. Internat. Scien. et Pop. des Falsifications*, 3, 195.

treated with oil will float. The cold-water test should always be made, since valuable indications as to the purity of the samples may usually be obtained. A low percentage of ash indicates an adulterated sample. The microscope must be employed for the final identifications of the constituents of the sample.

REPORT OF EXAMINATION OF SAMPLES BOUGHT IN THE OPEN MARKET.

The samples included in the following report were purchased in stores ranging from the best class to the poorest. The grades of coffee fairly represent the market:

Description of samples examined.

Date.	Serial number.	Name and address of retail dealer.	Retail price per pound.	Name under which sold.	Description. ¹	Remarks.
1890.			<i>Cents.</i>			
June 20	6853		25	Rio	Roasted ..	
	6854	W. R. Brown, 20th and Pennsylvania avenue NW.	35	Podung Java ..	Ground ..	
	6855	P. H. Ward, 21st and Pennsylvania avenue NW.	25	Java	Green	
	6856	W. H. & B. Reynolds, 2919 M street NW.	30	Johnson's Coffee.	Roasted ..	
	6857	M. Cropley, 3101 M street NW.	30	Rio	Ground ..	
	6858	Atlantic and Pacific Tea Co., 30th and M streets NW.	35	Flavored Java.	...do ...	
	6859	...do	25	8 o'clock Breakfast.	...do ...	Dealer says contains chicory.
	6860	Oppert & Bros., 32d and M streets NW.	25	Rio	Green	
	6861	Reckert, 3232 M street NW.	30	...do	Ground ..	
	6862	Lowe, Potomac and M streets NW.	28	...do	...do ...	
	6863	W. T. Dyer, 3418 M street NW.	35	Mocha and Rio.	Roasted ..	
	6864	Goddard, 30th and M streets NW.	22	Rio	Green	
	6865	P. J. McIntyre, 2534 K street NW.	25	...do	Ground ..	
	6866	...do	40	Java	Roasted ..	
	6867	...do	30	Maraealibo	...do ...	
	6868	C. D. Kenny, 7th and I streets NW.	32	Java	Green	
	6869	...do	25	Second quality Java.	...do ...	
	6870	...do	35	Java	Roasted ..	No. 6868, roasted.
	6871	...do	32	Second quality Java.	...do ...	No. 6869, roasted.
	6872	C. I. Kellogg, Masonic Temple.	25	Rio	...do ...	
	6873	Carl Mueller, 8th and H streets NW.	35	Java	Ground ..	

¹ "Roasted" indicates coffees roasted but not ground.

Description of samples examined.

Date.	Serial number.	Name and address of retail dealer.	Retail price per pound.	Name under which sold.	Description. ¹	Remarks.
1890.			<i>Cents.</i>			
June 20	6874	— Burchard, 4½ street and Pennsylvania avenue.	40	Mocha	Roasted..	
	6875	— do	4	Hummel's Essence of coffee.		Price given is that of small package.
	6878	— Stentz, 639 Pennsylvania avenue SE.	25	Rio	Ground..	
	6879	J. T. Earnshaw, 8th and G streets SE.	25	Mocha and Java	do	Dealer states probably contains chicory.
	6880	— Tolson, 701 7th street SE.	35	Java	do	
June 21	6884	Geo. E. Kennedy, 1209 F street NW.	38	Mocha (best) ..	Roasted..	
	6885	— Bryan, New York avenue, near 15th street NW.	35	Java (best)	do	
	6886	Great Atlantic and Pacific Tea Co., 503 7th street NW.	28	Java (very best)	do	
	6887	Great China and Japan Tea Co., 731 7th street NW.	22	Yellow Rio	do	
	6888	— Cornwell & Sons, Pennsylvania avenue, near 15th street NW.	40	Java and Mocha	do	
	6889	— Metzgers, 417 7th street NW.	28	Rio	do	
	6890	— Goddard, 13th street and New York avenue NW.	35	Java and Mocha	do	
June 23	6895	J. H. Magruder, New York avenue NW.	50	Dunn's Essence of coffee.	do	Package price.
	6897	G. G. Cornwell, Pennsylvania avenue, near 15th street NW.	50	Borden's Extract of coffee.		Do.
	6904	— Burchard, 4½ street and Pennsylvania avenue NW.	27	Rio	Roasted..	Do.
1891.						
Feb. 26	8712	— Burchell, 1325 7th street NW.	32	Java	Green....	
Apr. 10	8770	Chas. I. Kellogg, Masonic Temple.	25	Rio	Roasted..	
Apr. 11	8771	Alexander Clark, 7th street and Florida avenue NW.	25	do	do	
	8772	Arbuckle Bros., Pittsburg, Pa., and New York.	30	Ariosa	do	
Apr. 13	8773	Alexander Clark, 7th street and Florida avenue NW.	20	do	Ground..	
Apr. 15	8775	A. Orison, Maine avenue and 4½ street SW.	25	Johnson's Breakfast.	do	Sold in packages.
	8776	W. A. Barnes, 212 4½ street SW.	25	Java	Roasted..	
	8777	Jno. B. Prout, 411 4½ street SW.	25	Blended coffee, Java and cereals.	Ground..	Dwinell, Hayward & Co., Boston. Packages.

¹ "Roasted" indicates coffees roasted but not ground.

Description of samples examined.

Date.	Serial number.	Name and address of retail dealer.	Retail price per pound.	Name under which sold.	Description. ¹	Remarks.
1891.			<i>Cents.</i>			
Apr. 15	8778	Smith & Razen, 601 4½ street SW.	25	Maracaibo	Roasted..	
	8779	— Newman, G and 4½ streets SW.	25	Rio	do	
	8780	Wm. A. L. Hunt & Co., 801 4½ street SW.	25	Mixed	do	
	8781	— Lacky, corner M and 4½ streets SW.	30	Brazil	Ground..	Sold in packages.
	8782	— Tolson, 1345 4½ street SW.	30	Java.....	do	
	8783	— Tyne, corner L and 6th streets SW.	25	Rio	Roasted..	
	8784	B. F. De Atley, C and 3d streets SW.	25	do	Ground..	
	8785	T. T. Keane, 429 3d street SW.	30	Mocha and Java	do	
	8786	Curtis & Bros., 217 Virginia avenue.	25	do	do	
	8787	C. A. Hammer, corner Virginia avenue and 2d street SW.	25	Rio and Arabuckles.	do	
	8788	C. L. Callis, Virginia avenue and 1st street SW.	28	do	do	
	8789	P. A. Cudmore, 101 B street SE.	25	Rio	Roasted..	
	8790	T. F. McCanley, 209 Pennsylvania avenue SE.	25	do	do	
	8791	Enterprise Tea Co., Jno. H. O'Donnell, prop., Pennsylvania avenue and 4th street SE.	25	Java.....	do	
	8792	D. E. Baldwin, 637 Pennsylvania avenue SE.	25	Rio	do	
	8793	Henry Kuhn, corner Pennsylvania avenue and 11th street SE.	28	do	do	
	8794	Jno. Hessel, 326 11th street SE.	28	do	Ground..	
	8795	R. C. Smallwood, corner South Carolina avenue and 11th street SE.	28	Rio	Roasted..	
	8796	Wm. F. McAllister, corner 6th and C streets NE.	25	Sunrise, blended	Ground..	Sold in packages.
	8797	Thos. A. Rover, 714 North Capitol street.	25	Rio	Roasted..	
	8798	J. E. Connelly, 1st and K streets NW.	25	Mixed	do	
	8799	— Murphy, O and 4th streets NW.	20	Rio	Ground..	
	8800	E. Kleps, R and New Jersey avenue NW.	25	do	Roasted..	

¹ "Roasted" indicates coffee roasted but not ground.

Description of samples examined—Continued.

Date.	Serial number.	Name and address of retail dealer.	Retail price per pound.	Name under which sold.	Description. ¹	Remarks.
1891.			<i>Cents.</i>			
Apr. 15	8801	Mrs. Emma, 7th and Pome-roy.	30	Maracaibo	Ground . .	
	8802	Grocery and variety store, 2128 7th street NW.	25	do	do	
	8803	M. P. Sullivan, 2222 7th street NW.	20	Rio	do	
	8804	— Quilter, 2226 7th street NW.	30	Rio and Maracaibo.	do	
	8805	Goldmar & Rubin, 1720 7th street NW.	25	Rio	Roasted . .	
May 2	8821	R. F. H. Lawson, 515 North Clark street, Baltimore, Md.	23	...do	Green	
	8825	Atlantic and Pacific Tea Co., 213 North Entaw street, Baltimore, Md.	22	...do	do	
	8826	N. T. Baker, Pearl and Lexington streets, Baltimore, Md.	20	...do	do	
	8827	United States Tea Co., Pearl and Lexington streets, Baltimore, Md.	26	Java	do	
	8828	Bryant & Clarvoe, 128 North Pacastreet, Baltimore, Md.	21	Rio	do	
	8829	Great China Tea Co., 425 Lexington street, Baltimore, Md.	22	...do	do	
	8830	M. J. Fadgen, corner Baltimore and Harrison streets, Baltimore, Md.	28	Java	do	
	8831	J. N. Anderson, 213 East Pratt street, Baltimore, Md.	22	Rio	do	
	8832	N. Reiter, 709 East Baltimore street, Baltimore, Md.	20	...do	do	
	8833	A. D. Landin, 707 East Baltimore street, Baltimore, Md.	30	Java	do	
	8834	H. S. Potts, 327 High street, Baltimore, Md.	23	Rio	do	
	8835	W. M. Maynadier, 404 North Calvert street, Baltimore, Md.	30	Java	do	
	8836	Hoffer, McGaw & Co., 222 North Charles street, Baltimore, Md.	28	Rio	Roasted . .	
	8837	Thos. M. Reese & Sons, 347 North Charles street, Baltimore, Md.	26	...do	do	
	8838	G. C. Raffel, 332 Park street, Baltimore, Md.	24	...do	do	

¹ "Roasted" indicates coffees roasted but not ground.

Description of samples examined—Continued.

Date.	Serial number.	Name and address of retail dealer.	Retail price per pound.	Name under which sold.	Description. ¹	Remarks.
1891.			<i>Cents.</i>			
May 2	8839	Reitz Bros., 206 North Eutaw street, Baltimore, Md.	26	Rio	do	
	8840	W. B. Bealmar & Co., 216 North Eutawstreet, Baltimore, Md.	26	do	do	
	8841	A. Katzenberg, 222 North Eutaw street, Baltimore, Md.	26	do	do	
	8842	— Meyers, corner Pearl and Chestnut streets, Baltimore, Md.	26	do	do	
	8843	— Acombe, corner Pearl and Mulberry streets, Baltimore, Md.	28	do	do	
	8844	— Schooly, 205 North Pearl street, Baltimore, Md.	24	do	do	
	8845	N. T. Baker, northeast corner Pearl and Lexington streets, Baltimore, Md.	26	do	do	
	8846	— Hickmen, 637 West Fayette street, Baltimore, Md.	27	do	do	
	8847	— James, corner Fayette and Arch streets, Baltimore, Md.	35	Java	do	
	8848	Wm. Lutzer, 26 Harrison street, Baltimore, Md.	27	Rio	do	
	8849	Atlantic and Pacific Tea Co., 613 East Baltimore street, Baltimore, Md.	24	do	do	
	8850	Frey & Co., 1000 East Fayette street, Baltimore, Md.	25	do	do	
	8851	Franklin & Cane, corner High and Gay streets, Baltimore, Md.	26	Levering's coffee.	do	
	8852	Great China Tea Co., 615 East Baltimore street, Baltimore, Md.	12	Coffee substitute.		
	8853	do	16	Japan coffee ..	Ground ..	50 per cent coffee 50 per cent chicory.
	8854	H. Kettenboch, 18 Harrison street, Baltimore, Md.	20	Golden, blended	do	
	8860	C. C. Bryan, 1413 New York avenue, Washington, D. C.	35	Java	Roasted ..	
	8861	do	35	do	do	
	8862	G. G. Cornwall & Sons, Pennsylvania avenue near 15th street.	38	Mocha and Java	do	
June 17	8950	On sale in Kansas		Aromatic Aurora coffee.	do	Sold in packages.

¹ "Roasted" indicates coffees roasted but not ground.

Description of samples examined—Continued.

Date.	Serial number.	Name and address of retail dealer.	Retail price per pound.	Name under which sold.	Description.	Remarks.
1891			<i>Cents.</i>			
June 19	8961	John Hockmeyer, Center Market.	28	Rio	Roasted..	
	8962	John H. O'Donnell, Center Market.	28	...do	...do ...	
	8963	Capital Tea Co., Center Market.	25	...do	...do ...	
June 26	8987	C. C. Bryan, 1413 New York avenue.	40	Pulverized Java	...do ...	Chase & Sanborn Boston, Mass., packages.
June 26	8988	Great Atlantic and Pacific Tea Co., market 21st and K Streets.	28	Rio	...do ...	

SUMMARY.

	Number of samples.
Ground coffees	30
Roasted coffee ¹	60
Coffee extracts	3
Green coffees	18
Coffee substitute	1
Total number of samples	113

¹ Roasted indicates coffees roasted but not ground.

ROASTED COFFEES.

But three of the samples of whole roasted coffees were adulterated. Knowing the large amount of imitation coffee that is on the markets this result was unexpected. The adulterated samples were Nos. 6872, 8950, and 8963; each of these contained imitation coffee. The composition of the imitation coffee is given on page 916 under these numbers. No. 6872 contained a small percentage of imitation coffee; No. 8950 contained 50 per cent, and No. 8963 contained 1½ per cent.

The imitation coffee in the case of No. 6872 was introduced by the roaster. No. 8950 is a package coffee, sold largely in Kansas. Its origin is not known. It is very probable that roasted coffees are adulterated to a very much greater extent for sale in packages in districts not readily accessible to a roaster. Large quantities of package coffees are sold in Kansas. Samples Nos. 8840 and 8849 contained an excessive proportion of coffee screenings. While coffee screenings consist largely of fragments of pure coffee beans, they should, notwithstanding this, be considered adulterants. One, sample No. 8772, was roasted with a large amount of a glazing material. According to König (see page 913) this is objectionable, since it would increase the weight of the roasted coffee without corresponding advantage to the consumer.

It is not unusual for roasters to employ a small amount of glazing material, as they claim, to protect the coffee.

The accompanying table gives the results of a few examinations of coffees by Stutzer and Reitnair's method (see page 913) for the determination of the cold-water extract:

Serial No.	Extract.	Serial No.	Extract.
	<i>Per cent.</i>		<i>Per cent.</i>
6853.....	0.93	6885.....	0.63
6856.....	.87	6886.....	.85
6863.....	.76	6887.....	1.12
6870.....	.40	6888.....	.35
6871.....	.57	6889.....	.92
6872.....	.92	6890.....	.54
6873.....	.70	8772.....	1.76
6884.....	.57		

Stutzer and Reitnair give from .44 per cent to .72 per cent as the range of the organic extract as determined by their method.

Seven of the samples included in the above table gave a higher extract than the upper limit given by these chemists, hence it is fair to presume that these coffees were roasted with a glazing material. No. 6872 contains imitation coffee, which may account for the high organic extract.

It is probable that the retail dealer is not usually directly to blame in foisting imitation coffee upon the market, though he must in many cases know that he can not obtain a good merchantable coffee for the price he is paying. In most cases the imitation coffee is probable introduced by the roaster. The price paid for roasting coffees is usually very small and the competition is very sharp. A dishonest roaster can so easily increase his profits by the addition of imitation coffee, and with so little fear of detection, that he is often tempted and probably as often yields to temptation.

GREEN COFFEES.

A large number of green coffees, bought on the open market, were examined, all of which were genuine. It was impossible to detect cases where coffee of one grade was sold for that of another. There were undoubtedly many such samples.

GROUND COFFEES.

Ground coffees afford a very wide field for adulterations. This class of coffees is sold largely by the smaller dealers. The large stores usually grind the coffee to order, but in the latter case the presence of the purchaser does not always insure pure coffee.

Ground coffees.

Serial.	Water test. Coloration in five minutes.	Water test. Proportion sinking in water in five minutes.	Microscopic examination.	
			Approximate coffee.	Adulterants.
			<i>Per cent.</i>	
6854	Slight		100	Imitation coffee.
6857	No color		75 to 90	
6858	...do		100	
6859	Highly colored		75	Chicory and wheat. Dealer admitted presence, of chicory.
6861	No color		100	Chicory and wheat. Wheat partly in masses, probably Graham bread crumbs; rest in fragments.
6862	...do		100	
6865	Highly colored		25	
6874	Slight		Nearly 100	Chicory.
6878	Colored		75	Chicory and pease.
6879	...do		Less than 25	Chicory, pea hulls, and wheat.
6880	No color		90	Imitation coffee.
8773	...do		100	Sample consists of coffee screenings.
8775	Highly colored	Two-thirds ...	50	Much peas (especially the testa), wheat bran and chicory.
8777	...do	One-half	50	Wheat and chicory.
8781	...do	One-half	50	Wheat, chicory, and pease, and a little corn.
8782	Slight	One-fourth	50 to 75	Pease and chicory, with a little corn.
8784	Highly colored	One-third	50	Chicory and barley, with small amount of pease and corn.
8785	Slight	One-third	50 to 75	Chicory and pease.
8786	Highly colored	Two-thirds ...	25 to 50	Small amount chicory, wheat bran, buckwheat bran, and pea hulls, and fragments of pease.
8787	Slight		Nearly 100	Small amount wheat, pease, and chicory.
8788	Highly colored	One-half	25 to 50	Chicory and barley, with occasional fragments corn.
8794	Slight	One-fourth	100	Chicory and pease.
8796	Highly colored	Three-fourths	50 to 75	
8799	...do	Nearly all	No coffee ...	
8801	...do	Three-fourths	25 to 50	About 25 per cent chicory; rest wheat bran and pea hulls mashed together.
8802	No color	One-fifth	50	Mostly pea hulls, fragments of pease, wheat, and small per cent chicory.
8803	Slight	One-fourth ...	25 to 50	Wheat, probably Graham bread.
8804	...do	One-half	25 to 50	Chicory and wheat.
8853	Highly colored		25 to 40	Pea hull, with small per cent wheat and chicory.
8854	Colored		25 to 50	About 50 per cent chicory; rest pease and wheat. ¹
				Wheat, chicory, and pease.

¹ Dealer stated that this sample contains 50 per cent coffee and 50 per cent chicory.

The results of the examination of thirty samples are given in the table of ground coffees. Twenty-six samples, or 86 $\frac{2}{3}$ per cent of the samples examined, were adulterated, or if we include serial No. 8773, this percentage is increased to 90. One sample, sold as ground Rio, contained no coffee at all. In fifteen samples the purchaser obtained half, or less

than half, the coffee he paid for. The price paid for samples Nos. 6874 and 6880 was certainly high enough to have insured a pure coffee.

The following table gives a comparative statement of the adulterated coffees, showing the price and the quality; the latter only so far as the percentage of pure coffee is concerned:

Table showing name under which adulterated ground coffees were sold, and the price per pound.

Serial No.	Name under which sold.	Price per pound.	Approximate per cent pure coffee.	Remarks.
		<i>Cents.</i>		
6857	Rio	30	75 to 90	
6859	8 o'clock breakfast ...	25	75.....	
6865	Rio	25	25.....	
6874	Mocha	40	92.....	Ten per cent infusion indicated from 7.7 to 8.7 per cent chicory.
6878	Rio	25	75.....	
6879	Mocha and Java.....	25	Less than 25.....	
6880	Java	35	90.....	
8773		20	Coffee screenings	A sample of coffee screenings examined contained 10 per cent sand; also sticks and beans.
8775	Johnson's breakfast ..	25	50.....	
8777	Blended coffee, Java and cereals.	25	50.....	
8781	Brazil	25	50.....	
8782	Java	30	50 to 75	
8784	Rio	25	50.....	
8785	Mocha and Java.....	30	50 to 75	
8786		25	25 to 50	
8787	Rio and Arbuckle's...	25	Nearly 100	
8788		28	25 to 50	
8796	Sunrise blended	25	50 to 75	
8799	Rio	20	No coffee	
8801	Maracaibo (?).....	30	25 to 50	
8802		25	50.....	
8803	Rio	20	25 to 50	
8804	Rio and Maracaibo....	30	25 to 50	
8853	Japan coffee	16	25 to 49	Dealer states that sample contains 50 per cent chicory.
8854	Golden blended.....	20	25 to 50	

EXTRACTS.

Three samples of coffee extracts were examined, viz: Serial Nos. 6875, 6895 and 6897. No. 6875 contained no caffeine. On further examination it was found to contain cereals or other starchy bodies and no coffee. This sample contained tin and traces of copper. The former amounted to 0.248 grams per 1,000 grams of the extract.

Sample No. 6895 contained 1.19 per cent caffeine. This is about König's mean (see p. 903) for roasted coffees; hence pound for pound this is about equal to average coffee. This sample contained considerable dextrose.

Sample No. 6,897 is a preparation of coffee with milk and sugar. This sample contains .72 per cent caffeine. On the basis of König's mean percentage of caffeine two-thirds of a pound of average coffee is equal to about 1 pound of this preparation. This sample contains both tin and copper in the following proportions per kilogram of the preparation:

	Grams.
Compounds of tin (calculated as metallic tin)	0.338
Compounds of copper (calculated as metallic copper)	0.023

Judging from the observations of Dr. Van Hamel Roos the amount of tin in these samples is excessive and the risk of poisoning is great. The article quoted by Dr. Van Hamel Roos is well worth reproducing.

TIN POISONING BY PRESERVES IN TIN CANS.¹

It seems to me that sufficient attention has not been paid to the impurities of preserves and to poisoning by compounds of tin. It is only during recent years that chemists and hygienists have given this subject serious consideration. It is the purpose of these pages to make known the facts heretofore proved and to point out certain means for the prevention of like impurities.

Messrs. Nüger and Bodländer were first to call attention to the presence of relatively large amounts of tin in preserved asparagus. Sæche found 70 milligrams of tin in 29 pieces of asparagus (see this *Revue*, 1, 91.) Likewise, Messrs. A. Menthe (*Chem. News*, July, 1871) and Schner (*The Analyst*, 1880, p. 318) proved the presence of more or less important quantities of tin, not only in preserved fruit, but in other food materials, liquids, meat, and other preserved foods.

Sedgwick cites for the first time a case of food poisoning which must be unquestionably attributed to tin (*Archives de Pharmacie*, 1888). The poisoning was caused by pears prepared in a tinned stewpan. [A member of the congress, present session, informs me that a patient died from chronic metallic poisoning, resulting from the prolonged use of metalliferous preserved vegetables.] He afterwards examined several fruits preserved in tin cans and found very pronounced reactions for tin in all of them.

Prof. Beckurts presented some very important data concerning the presence of tin in foods preserved in tin cans, at the session of the congress of German physicians held at Heidelberg, September 25, 1889. He emphasizes the importance of the sulphide of tin, which is formed by the action of the albuminous matter of vegetables, meat, etc., on the tin of cans. At the current session, Dr. Nehring stated that he had proved the presence of 0.186 grams, 0.3146 grams, and 0.2269 grams, respectively of tin in three tin cans containing asparagus; whence the congress decided that the use of tin cans for the preservation of foods must be interdicted.

During the preceding year Prof. Blarez communicated to the *Journ. de Pharm. et de Chimie* that he had found a considerable amount of tin in pears in tin cans. I am not able to confirm this result, as by repeated trials I only obtained traces of tin. I presume that the presence of a considerable quantity of salt has favored the solution of tin in the sample examined by M. Blarez.

Capitaine-Intendant Winckel reported to the Congress of Industrial Hygiene, held at Amsterdam, September 1890 (see No. 3 of this *Revue*), that 270 soldiers became ill after having eaten lettuce and meat preserved in tins. According to the determinations of Prof. Wefers Bettink, of Utrecht, the amount of tin present was from 19 to 72 milligrams per kilogram.

¹ Dr. Van Hamel Roos, *Rev. Intern. des Falsifications*, 4, 10, 179, translated by Mr. E. E. Ewell for this report.

Prof. Kayser, of Nuremberg, reported in this *Revue* (4, p. 29), that he had found a considerable amount of tin, about 0.19 per cent, in preserved eels. Several persons had become very ill after eating them.

These facts move me to point out the necessity of coating the interior of tin cans with a varnish or substance capable of resisting the action of a weak acid or of organic substances. I am pleased that I am able to say that a Holland manufacturer, C. Verwer, of Krommeine, has succeeded in preparing a varnish which, according to my investigations, answers the purpose perfectly, especially in regard to the manner of its application to the tinned surface (an important consideration, since the application of this useful invention rests not only upon the composition of the varnish, but in the first place upon the process for making the varnish perfectly adherent to the inner tinned surface).

It is evident that time plays a great rôle in the question of the solubility of the interior tinned surface. Evident proof of this is furnished by a can of asparagus preserved since 1860, a period of thirty-one years. (This box was opened and its contents were exhibited at the current session.) The tin of this can having entirely disappeared, was dissolved in the liquid.

I will now give the results of my own investigations.

A can containing beef, preserved eight years and weighing 976 grams (beef and liquid), contained 77 milligrams of oxide of tin; a can of asparagus, preserved six years, contained 56 milligrams of oxide of tin, while another can of asparagus which had been kept only four months contained 11 milligrams. Another can of asparagus, preserved two years and having a net weight of 635 grams, contained 36 milligrams of oxide of tin and 6 milligrams of copper. Some apricots contained 20 milligrams per can, and some fine apples, imported from Singapore, a considerable quantity of tin, about 178 milligrams per kilogram. Fortunately the taste of this last sample was so disagreeable that its consumption, if not its sale, was impossible. Several other preserved fruits and vegetables, such as purslane, sauerkraut, pears, carrots, etc., as well as meat and soup, all contained more or less tin, depending, in the case of vegetable and fruits, on the quantity and kind of organic acids. I am perfectly confident of the correctness of the opinion of Prof. H. Wefers Bettink, of Utrecht, as communicated to the Congress, that malic acid is the principal solvent in the case of fruits and vegetables.

In order to determine whether the above-mentioned varnish was capable of preventing the solvent action of acids on tin, some of the most acid foods, such as sauerkraut, buttermilk, and pears in wine, after four to nine months' preservation in varnished tin cans, were subjected to examination. Only imponderable traces of tin were found. The same results were obtained with a sample of beans coming from France, and examined after being preserved twelve years in a varnished can. This latter example is of less importance, since beans and pease free from salt dissolve almost no tin. I have been unable to procure acid foods which have been preserved for as long a time, but in my opinion the experience acquired after four to nine months suffices to heartily recommend the varnishing process, in consideration of the strong reactions which occur in unvarnished cans. In accordance with experience thus far gained, sorrel is the only vegetable whose action the varnish is unable to resist. After a few months the varnish was already detached and a considerable amount of tin dissolved.

The writer closes his paper by recommending that varnished cans alone be used, especially for acid foods, and deems it important that the various governments give the matter consideration. Several manufacturers have already adopted the process for acid foods and beverages.

The amount of copper in sample No. 6897 is quite large, and is sufficient to condemn the preparation.

SUBSTITUTES.

The number of coffee substitutes on the market is large. Many of these are sold under this name and others are simply designated as substitutes. Sample No. 8852 was sold as a coffee substitute. It is composed largely of chicory (50 to 75 per cent), with wheat, and peas or beans.

There is no objection to the so-called coffee substitutes, provided they are sold as such and do not contain harmful ingredients. All substitutes should be sold in packages, bearing labels distinctly stating their composition.

CONCLUSION.

The examination of the coffees and coffee preparations on our markets shows that the consumers, and especially the poor, are being grossly deceived. Very little pure ground coffee is sold, and even whole coffee does not escape sophistication. The purchase of green coffee for home roasting does not insure a pure product, since even the green coffee is imitated. Stringent laws are certainly needed to suppress these frauds.

That there is a large demand for imitation coffee is evidenced by the fact of its importation from Germany. The manufacture of these coffees in imitation of the form of the genuine bean should be interdicted, even if the product is to be sold as a substitute.

COCOA PREPARATIONS.

By ERVIN E. EWELL.

THE NATURE, SOURCE, COMMERCIAL IMPORTANCE, ETC., OF THE COCOA BEAN.¹

The raw material from which the cocoas and chocolates of commerce are manufactured is the "cocoa bean," the seed of the cocoa, or cacao, tree (*Theobroma cacao*). While this tree has been successfully introduced into various warm countries, tropical America, its native land, still furnishes the larger and more highly valued portion of the world's supply of cocoa. From Mexico to Peru on the west coast, Mexico to Bahia, Brazil, on the east coast, and on the West India Islands, the most favorable conditions for its cultivation are met.

The tree, 6 to 12 meters in height, blooms continuously and yields two crops a year. The lemon yellow, fleshy fruit, 10 to 15 centimeters long, 5 to 7 centimeters in diameter, resembles in general appearance a cucumber, constricted at the upper end, tapered to a point at the lower end, and having ten longitudinal ridges. Twenty-five to forty, sometimes more, seeds are arranged in the fleshy pulp in five longitudinal rows. When first removed the seeds are colorless, fleshy, and covered with mucilage. On drying, with exposure to air and light, they become golden yellow to red or brown in color, and hard and brittle. They are egg-shaped, somewhat compressed, 1.2 to 2 centimeters long, and 0.6 to 1 centimeter broad.

¹ For more detailed information concerning cultivation, preparation for market, manufacture, etc., of cocoa, see the following works: *Cultivation, harvesting, etc.*: An anonymous article on the cultivation of the cocoa tree in Colombia, *Phar. Jour. Trans.*, [3] **970**, 591; Boussingault, *Compt. Rend.*, **96**, 1395; *Jour. Chem. Soc.*, 1883, **44**, 933; Boussingault, *Ann. Chim. Phys.*, [5] **28**, 433; *Jour. Chem. Soc.*, 1884, **46**, 202; *Chem. Ztg.*, 1883, 203 and 902; Holm, *American Chem.*, **5**, 320; *Jahresb. d. Chem.*, 1875, 1121; Smith, *Dictionary of Economic Plants. Manufacture*: Bernhardt, *Chem. Ztg.*, 1889, 32; Saldau, *Die Chokolade Fabrikation*, 1881; Tresca, *Les Mondes*, July 22, 1869. *General description of tree, cultivation, harvesting, manufacture, etc.*: Blyth, *Foods: their Composition and Analysis*; Hassall, *Food: its Adulteration and the Methods for their Detection*; König, *Die menschlichen Nahrungs- und Genussmittel, ihre Herstellung, Zusammensetzung und Beschaffenheit, ihre Verfälschungen und deren Nachweisung*; Macé, *Les substances alimentaires étudiées au microscope*; Moeller, *Mikroskopie der Nahrungs und Genussmittel aus dem Pflanzenreiche*; Schaedler, *Die Technologie der Fette und Oele des Pflanzen- und Thierreichs*.

After removal from the fruit two processes are used for the preparation of the seeds for market. For the production of "unfermented cocoa," they are freed from adhering fruit pulp and at once dried in the sun. For the production of "fermented cocoa," the beans are placed in piles in sheds or are buried in trenches and allowed to ferment for a time before being completely dried in the sun. When buried the beans are now placed in casks or other coverings; hence, the earthy coating is no longer a mark for determining the process of preparation. Much of the acidity and bitterness disappears in this process of fermentation; the beans so prepared have a mildly oleaginous, pleasant, slightly bitter taste, and are more or less aromatic. The value of the product therefore greatly depends upon the care bestowed upon this operation.

Bernhardt¹ has made a careful study of the losses occurring in the preliminary processes of manufacture. He notes four main operations:

(1) The sifting of the raw cocoa to remove sand, dust, small stones, etc.

(2) The separation by hand of the larger stones, empty beans, grass, wood, etc.

(3) The roasting.

(4) The breaking up and cleaning to remove the husks.

He gives the results of thirty determinations, made in actual factory work, of the losses in each of these processes. The means, maxima, and minima of these determinations will be found in the table given below. In this table the term "beans" is used to denote both the good broken pieces and the small particles, which, of less value and constituting 9 to 15 per cent of the whole bean, are used for the preparation of cheaper chocolates.

Losses in preliminary preparation for manufacture.

	Means.	Maxima.	Minima.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Sifting.....	2.80	5.49	1.10
Picking	.80	2.09	.25
Roasting.....	5.51	7.05	4.61
Cleaning.....	13.00	16.04	10.08
Total loss.....	22.11	25.78	16.76
Total beans.....	77.89	74.22	83.24

The following table, from the Annual Report of the Bureau of Statistics of the United States Treasury Department for 1890, is given to show something of the commercial importance of this commodity and the extent of the use of its preparation in the United States.

¹ Chem. Ztg., 1889, 32.

Importation of cocoa or cacao, crude, and the leaves and shells of, for the year ending June 30, 1890.

Countries from which imported,	Pounds.	Value.
Brazil.....	2, 125, 614	\$192, 866
Guatemala.....	1, 000	125
Honduras.....	3, 036	412
Nicaragua.....	48, 750	8, 424
Salvador.....	769	157
China.....	52, 304	10, 227
Columbia.....	401, 759	83, 294
Ecuador.....	1, 260, 191	150, 156
France.....	40, 181	6, 358
Germany.....	86, 160	16, 334
England.....	1, 607, 821	335, 337
British West Indies.....	5, 382, 498	674, 165
Guiana.....	12, 084	1, 286
East Indies.....	153, 101	29, 944
Haiti.....	1, 303, 114	101, 396
Mexico.....	468	149
Netherlands.....	191, 970	25, 361
Dutch Guiana.....	4, 128, 374	460, 282
Peru.....	81, 051	13, 026
Portugal.....	54, 103	5, 730
San Domingo.....	54, 276	6, 202
Venezuela.....	1, 277, 547	191, 550
Total.....	18, 266, 177	2, 312, 781

During the same period, 634,551 pounds of chocolate, valued at \$146,476, and 993,402 pounds of manufactured or prepared cocoa, valued at \$400,385, were imported.

The *Shipping and Commercial List and New York Price Current* for October 7, 1891, gave the following quotations for cocoa :

	Cents per pound.
Caracas.....	14 to 15
Trinidad.....	13½ to 14
Guyaquil.....	13 to 14½
Bahia.....	13
St. Domingo.....	8 to 8½

CHEMICAL CONSTITUENTS.

On account of the peculiar properties of the cocoa bean, its preparations merit a place on our tables for two reasons: In addition to being, like tea and coffee, the material for the preparation of a pleasant and exhilarating beverage, it is a valuable food material. Not only is it much richer in nutritive substances than tea or coffee, but both the soluble and insoluble portions become a part of the beverage, while only the constituents soluble in hot water are obtained in the beverages prepared from tea and coffee. The investigations of Stutzer (see below, under head of nitrogenous constituents) and others clearly prove, how-

ever, that the food value of cocoa preparations has been greatly overestimated and that many of the present modes of preparation do not develop in the highest possible degree the pleasing aroma and flavor. The inventive energy of many manufacturers seems to be spent on the production of a highly nutritive and easily digestible preparation; the valuable fat is removed and the delicious aroma and flavor destroyed by chemicals for the ostensible purpose of rendering more digestible a residue of doubtful food value.

The more important constituents of the husked cocoa bean are fat, theobromine, the nonalkaloidal nitrogenous substances, starch, the coloring matter called cocoa red, and the mineral matter.

The fat, cocoa or cacao butter, in consequence of its quantity and peculiar excellence, is unquestionably the constituent of the cocoa bean possessing highest food value. It usually forms 45 to 55 per cent of the husked bean, rarely falls below 45 per cent, and only one recent analysis shows as low as 36 per cent. At ordinary temperatures it is a white, or slightly yellowish, brittle solid, having a pleasing taste and odor, and showing but little tendency to become rancid. Its melting point being below the temperature of the body, insures its being presented in liquid form to the action of the digestive juices. Chemically, it is a mixture of the glycerides of stearic, palmitic, oleic, and arachidic acids.¹ It is readily soluble in ether, acetic ether, chloroform, oil of turpentine, and hot absolute alcohol, but only $\frac{1}{2}$ per cent remains in solution when the alcohol becomes cold;² fully soluble at ordinary temperatures in 2 parts ether, $\frac{1}{2}$ part of benzol, 100 parts of cold and 20 parts of hot alcohol.³

The physical and chemical constants of value in investigations for identity and purity have been arranged in tabular form in the table given on page 938. In addition to numbers there given, Valenta⁴ has found the temperature at which the solution in hot glacial acetic acid becomes turbid to be 105° C.

The low melting point, the little tendency to become rancid, and other properties render cocoa butter peculiarly suitable for the basis of many pharmaceutical preparations. This by-product of the manufacture of cocoa preparations has, therefore, a well-established place in commerce. *The Shipping and Commercial List and New York Price Current* for October 7, 1891, quotes foreign cocoa butter at 31 to 37 cents per pound and domestic at 40 to 42 cents per pound.

Schaedler² thus describes the process of extraction on the large scale:

In earlier times the ground and roasted beans were boiled with ten parts of water,

¹ Benedikt, *Analyse der Fette und Wachsarten*. Schaedler, *Die Technologie der Fette und Oele des Pflanzen- und Thierreichs*.

² Schaedler, *Die Technologie der Fette und Oele des Pflanzen- und Thierreichs*.

³ Blyth, *Foods: their Composition and Analysis*.

⁴ Dingler's polyt. Jour., **252**, 296; *Zeitsch. f. anal. Chem.*, **24**, 295.

the fat skimmed off, and the residue pressed out. The beans are now roasted, husked, and very finely ground. The mass is heated to 70°–80° C., packed in sacks of ticking, and submitted to pressure between previously warmed or steam-heated plates. The fat expressed, about 30–35 per cent, is filtered through dry filters. The pure residue, containing 10–15 per cent of oil, is made into chocolate. For the preparation of an entirely fat-free cocoa powder, the roasted and ground beans are exhausted with benzine or ether.

Theobromine, the alkaloid of cocoa, is very closely related chemically (it is dimethyl xanthine, $C_5H_2(CH_3)_2N_4O_2$, while caffeine is trimethyl xanthine, $C_5H(CH_3)_3N_4O_2$) to caffeine, the alkaloid of tea and coffee, and has similar effects on the system; the power possessed by the beverages prepared from these substances, "to cheer and not to inebriate," being largely due to the presence of these alkaloids.

Separated from the bean, it is a white powder, permanent in the air, crystallizable in microscopic rhombic needles, and having a very bitter taste. While neutral in reaction, it acts as a weak base, uniting with acids to form crystallizable salts, which become basic on treatment with water; its salts of volatile acids give up their acids on heating, at or below 100° C. According to Blyth,¹ it begins to sublime at 134° C., and yields distinct crystals at 170° C. and above; Keller, 1854², reports the subliming point as 290°–295° C.; other writers note it as about 290° C. Treumann³ has reported its solubility in water to be 1 in 148.5 at 100° C., and 1 in 1,600 at 17° C.; in absolute alcohol, 1 in 422.5 at the boiling point, and 1 in 4,284 at 17° C.; in boiling chloroform, 1 in 105. Husemann⁴ states its solubility in water to be 1 in 55 at 100° C., 1 in 660 at 20° C., and 1 in 1,600 at 0° C.; in cold alcohol, 1 in 1,460, and in boiling alcohol, 1 in 47; in cold ether, 1 in 17,000, and in boiling ether, 1 in 600; more soluble in chloroform and warm amyl alcohol than in water, less soluble in benzol, and insoluble in petroleum ether.

¹ *Op. cit.*, note 3, p. 936 of this work.

² *Op. cit.*, note 3, p. 938 of this work.

³ *Archiv. d. Pharm.*, [3] **13**, 5; *Jahresb. d. Chem.*, 1878, 872.

⁴ Husemann's *Pflanzenstoffe*.

Physical and chemical constants for the fat of the cocoa bean.

Analyst.	Character of samples.	Physical constants for the fat.				Chemical constants for the fat.				The fatty acids.	
		Specific grav- ity at 15° C.	Melting point.	Congea- ling point.	Crystal- lizing point.	Refrac- tive index.	Hehner's number.	Hübl's iodine number.	Köttstor- fer's number.	Melting point.	Congea- ling point.
Bensemann ¹	Maracalbo.....		25-26				94.59			48-52	
	Caracas.....		27-28				95.31			48-52	
	Trinidad.....		26-27				95.65			49-53	
	Porto Plata.....		28-29				95.46			49-53	
	Machala-Guayaquil.....		28-29				95.24			49-53	
Rüdorf ²			33.5		27.3						
Chateau ³		0.89-0.92	28.0		23.0						
Herbst ²			30-33								
Hübl ²								34.0		52	51
Wimmel ²			33.5-34	20.5	27-29.5						
Dieterich ²		0.980-0.981									
Hager ²	Fresh.....	0.950-0.952									
	Old.....	0.945-0.946									
Moore ²									199.8		
Schaeffer ⁴		0.890-0.900	25-27 or 28	20.5	26-26.5						
Blyth ⁵			29-30	24							
Skalweit ⁶						1.468					
Filsinger ⁷		At 100° C., H ₂ O, at 15° C.=1.	32.1-33.6					34-37.5	192-202		
Allen ²		0.857									
Weigmann ⁸									198.4-203		

¹ Rep. f. anal., Chem., 4, 165; Zeitsch. f. anal. Chem., 24, 628.² Benedikt, Analyse der Fette und Wachstern.³ Prescott, Organic Analysis.⁴ Op. cit., note 2, p. 336 of this work.⁵ Op. cit., note 3, p. 336 of this work.⁶ Rep. f. anal. Chem., 6, 181; Zeitsch. f. anal. Chem., 28, 386.⁷ Chem. Zeit. Rep., 13, 309; Chem. Centralt., 1890, 130.⁸ König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.

For percentages of theobromine in cocoa and cocoa preparations, see tables of analyses given below; for methods of quantitative determination, see methods of analysis given below.

The commercial importance of theobromine at present offers no temptation to remove it from cocoa preparations before placing them on the market.

Small percentages of caffeine have been found in cocoa beans, especially in the shells. It is separated from the theobromine by solution in cold benzol, in which the theobromine is practically insoluble. Weigmann¹ reports 0.17 per cent in cocoa mass and 0.113 to 0.190 per cent in cocoa shells; Bell,² traces to 0.25 per cent in the bean and 0.33 per cent in the shells.

Nonalkaloidal nitrogenous substances.—Stutzer³ classifies the nitrogenous constituents of cocoa, as follows:

(1) Nonproteids, substances soluble in neutral water solution in presence of $\text{Cu}(\text{OH})_2$ (theobromine, ammonia, and amido compounds).

(2) Digestible albumen, insoluble in neutral water solution in presence of $\text{Cu}(\text{OH})_2$, but soluble when treated successively with acid gastric juice and alkaline pancreas extract.

(3) Insoluble and indigestible nitrogenous substances.

In the same article he writes as follows in regard to the food value of the nitrogenous constituents of cocoa:

The group of nitrogenous constituents is of great importance in all foods, especially their content of digestible albumen. Among the general public the opinion is very widely extended that cocoa belongs to the very easily digestible foods. The extremely favorable mechanical treatment is certainly such that the digestive fluids have no considerable mechanical resistance to overcome in the assimilation of the nutritive constituents of cocoa. On the contrary, the chemical reaction between the solvent constituents of the digestive juices and the nitrogenous nutritive constituents of cocoa can not be called an especially strong one. A large proportion of these constituents, in spite of apparently favorable conditions, remain entirely indigestible, entirely worthless. I have already called attention to the slight digestibility of the albuminous substances of cocoa,⁵ and once illustrated their difficult digestibility graphically in the Berliner Hygiene-Ansstellung. In the meantime, these observations have been confirmed by investigations of H. Weigmann, which he undertook on his own account. Weigmann found only 42 per cent of the nitrogenous substances in cocoa to be digestible.

¹ *Op. cit.*, note 8, p. 938 of this work.

² Bell, *Analysis and Adulteration of Food*.

³ *Zeitsch. f. angew. Chem.*, 1891, 368.

The results of Stutzer's own investigations, referred to above,¹ were obtained with three samples of cocoa powder, and are given as follows:

Of the total nitrogen present, there was found—	Sample No. 1.	Sample No. 2.	Sample No. 3.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Nitrogen in form of soluble nitrogenous compounds, including theobromine.....	31.43	26.95	29.79
Nitrogen in the form of digestible albumen	33.34	40.61	22.62
Nitrogen in the form of indigestible substances.....	35.33	32.44	47.83
	100.00	100.00	100.00

For quantitative data concerning the nitrogenous constituents, see tables on page 960 *et seq.*

Starch.—Cocoa contains 5.78 per cent to 15.13 per cent of starch. (See tables of analyses.) A more detailed description of this starch will be found in paragraphs devoted to the microscopy of cocoa.

Cocoa red, the coloring matter of the bean, seems to be related to the tannins, but authorities differ as to whether it is a decomposition product of a tannin, or whether a tannin is the result of its decomposition.

It is obtained from the aqueous or alcoholic decoction by precipitation with lead acetate and decomposition of the washed precipitate with hydrogen sulphide. The solution thus prepared has a bitter taste; according to Watts, gives a dark green or brown precipitate with ferric and a green or sometimes violet precipitate with ferrous salts; absorbs oxygen, becoming acid, the coloring matter being converted into a kind of tannic acid, which is precipitated by gelatin—cocoa red in its unaltered state not being thus thrown down (Hassall).²

It is colored blue by iron salts and dissolved out by caustic potash with a green color, by sulphuric acid with a red color, and by acetic acid and alcohol with a violet color. Cold water dissolves the pigment with difficulty, while warm water dissolves it entirely. Since the fresh seeds are colorless, Mitscherlich presumes that cocoa red is formed by a process of oxidation from a body related to the tannins (Moeller).³

It is changed to tannin by oxidation and colored green by iron salts (Tuchen).⁴

Concerning the astringent principle of cocoa and cocoa red, Thorpe⁵ writes as follows:

The astringent principle is of the nature of tannin, but is different from the tannin of tea and coffee. It rapidly changes during analysis to cocoa-red, which renders its quantitative estimation difficult. It is precipitated by subacetate of lead and may be obtained from this precipitate by decomposition with hydrogen sulphide;

¹ Rep. f. anal. chem., 1882, 88 and 165; Hygiene-Bericht, 1882-'83, 1, 217; also *op. cit.*, note 8, p. 938 of this work.

² Hassall, Food: its Adulteration and the Methods for their Detection.

³ Moeller, Mikroskopie der Nahrungs- und Genußmittel aus dem Pflanzenreiche.

⁴ Tuchen, Dissertation über d. organ. Bestandtheile der Cacao.

⁵ Thorpe, Dictionary of Applied Chemistry.

gives a green precipitate with ferric chloride, not unlike that given by caffeic acid under similar treatment (Bell).

Cocoa-red is not present in the fresh beans, but is the product of the oxidation of the natural tannin of the seed. It consequently appears in different quantities in different cocoas. It has the characters of a resin and exhibits variable degrees of solubility, probably coinciding with the extent of oxidation which it has undergone.

Bell¹ reports 2.20 per cent of cocoa red in raw beans; Muter, 3.96 per cent.

Gum.—It is precipitated from the aqueous solution of the fat-free beans by alcohol. After drying it resembles gum arabic; by treatment with nitric acid, yields mucic acid; and, when ignited, leaves an ash consisting of basic phosphate of magnesium. Its specific rotatory power is $[\alpha]_D^{20} = + 68.6$ (Boussingault²).

The beans contain 2.17 per cent of gum according to an analysis by Bell,¹ and 2.5 per cent according to Boussingault.²

Tartaric acid.—Weigmann³ estimates tartaric acid by precipitation of the aqueous extract, after neutralization with ammonia, with calcium chloride, redissolving in hydrochloric acid and reprecipitating with sodium hydrate. The amount of tartaric acid is calculated from the amount of calcium oxide contained in this precipitate. By this method he found 4.34 per cent to 5.82 per cent of tartaric acid in the raw, whole beans. Boussingault² reports 3.4 per cent and 3.7 per cent.

The aroma of cocoa is considered to be due to the presence of minute quantities of an aromatic volatile oil. Boussingault² proves its presence by distillation of the roasted grains with water.

THE COMMERCIAL PREPARATIONS OF THE COCOA BEAN, THEIR NATURE, METHODS OF PREPARATION, AND ADULTERATION.

The preparations of cocoa are so numerous that more or less confusion of terms naturally arises. Most American manufacturers prepare a plain chocolate (known in Europe as cacao-masse), made by reducing the roasted and husked beans to a paste and pressing into the form of cakes. When this is combined with much or little sugar (generally much), vanilla and spices, the various "sweet," "vanilla sweet," "vanilla," "spiced," etc., chocolates are produced. These are also usually met in the form of cakes, but are sometimes pulverized and sold as "powdered chocolates." The high percentage of fat renders a permanent powder impossible without its partial removal or the addition of some diluent, as sugar, starch, or flour. The preparations in powder, known as "cocoas," "bromas," etc., are prepared in accordance with one or the other, or a combination of these methods.

¹ *Op. cit.*, note 1, p. 939 of this work.

² *Ann. Chim. Phys.* [5], **28**, 433; *Jour. Chem. Soc.*, 1884, **46**, 202; *Chem. Ztg.*, 1883, 203 and 902.

³ *Op. cit.*, note 8, p. 938 of this work.

Cocoa shells are offered on the market in bulk and in packages, but their use seems to be quite limited at present.

For more detailed information in regard to the preparations found in American markets, see tabulated results of investigations given below.

Blyth¹ says:

The commercial varieties of cocoa are very numerous: *Cocoa nibs* are simply the bruised, roasted seeds deprived of their coverings, and *flake cocoa* is composed of the nibs ground in a particular form of mill. The soluble cocoas are ground cocoa, diluted with sugar and starches.

In the manufacture of chocolate, the cocoa nibs are ground in a mill, the rollers of which are usually heated by steam, so as to soften the cocoa butter; and in this way a paste is formed which is mixed with refined sugar, and very often other substance, and pressed into molds.

The Association of Swiss Analytical Chemists² has adopted the following definitions of terms:

(1) *Cacao-masse* (pure pâte). The beans are roasted, husked, ground, and pressed into forms.

(2) *Cacao deprived of its fat* is prepared by heating cacao-masse, removing about one-half of its oil by pressure, and pulverizing the residue.

(3) *Soluble cacao-powder* is prepared by treating the cacao deprived of its fat with ammonia, alkaline carbonates, or steam, so as to destroy the cellular structure and convert the albumenoid constituents into a soluble modification.

(4) *Cacao-powder* is cacao-masse, wholly or partially deprived of fat and flavored with spices (vanilla, cinnamon, cloves, allspice, etc.).

(5) *Chocolate* is pure cacao-masse mixed with sugar and pressed into forms or powdered. Other admixtures, such as spices, flour, etc., must be designated on the package.

(6) *Couverture*,

(7) *Chocolat à la noisette*, etc., are prepared from the above by adding almonds, hazelnuts, etc.

Moeller³, writing in Germany, notes the following preparations:

The cocoa preparations of commerce and consumption are cocoa flour or cocoa deprived of fat, cocoa butter, Holland cocoa, cocoa tea, and, most important of all, chocolate.

Cocoa seeds contain 35 per cent to 54 per cent of fat, which renders them difficultly digestible for children and convalescents, to whom a nourishing and stimulating food must be given. Therefore, the greater part of the fat is removed by pressure and the press residue designated as cocoa deprived of fat.

If the fat is not entirely removed by the pressure and the residue is still plastic, from it is prepared "Gesundheits Schokolade," under which name are also found shameful adulterations.

The desire to make an easily digestible cocoa preparation without removing the fat, led to the preparation of the so-called Holland cocoa. (It is, however, not certain that this object is in reality accomplished. By many it is stated that the alkalis directly hinder the digestion by neutralization of the acid of the stomach;

¹*Op. cit.*, note 3, p. 936 of this work.

²*Op. cit.*, note 3, p. 949 of this work.

³*Op. cit.*, note 3, p. 940 of this work.

and besides, by saponification of the fat, because experience shows that the soap is not agreeable to the stomach. Contrary opinions are asserted from the standpoint of sanitary police. By application of alkalis, the ash content is considerably raised [to over 9 per cent against 4 per cent in cocoa], which, according to the Deutschen Reichs-Gesundheitsamtes, is to be considered an adulteration. Recently, May 24, 1884, a process for making cocoa preparations soluble was patented by Lobeck & Co. In this process the material is heated, with or without water, in a closed vessel for thirty minutes at 150° C.) The cocoa beans are soaked several hours in water in which potassium or sodium hydrate (2 per cent to 4 per cent) and magnesia are dissolved, then dried and pulverized.

All good cocoa preparations should be made from the cotyledons only. For this purpose the seeds are always husked and the husks, under the name of "cocoa tea," constitute an independent article of commerce. The husks serve for the preparation of cheaper kinds of chocolate and for adulteration; when, in consequence of the small amount of theobromine contained therein, the addition of husks is usually considered unallowable.

The most general preparation of cocoa is chocolate. For its preparation the husked seeds, with the addition of sugar (50 per cent or more) and spices, are ground to a paste at an elevated temperature and pressed into forms.

Results of the examination of four cocoa powders by Stutzer¹ for the purpose of determining the effect of the process of manufacture on the chemical constituents.

[I is composed of 40 per cent Ariba, 40 per cent Machala, and 20 per cent Bahia cocoa, and was made in Wittekop & Co.'s factory in Braunschweig, without the use of chemicals. II is a sample of Holland cocoa. III and IV are German cocoas, and, in Stutzer's opinion, were prepared by use of ammonia.]

	I.	II.	III.	IV.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Theobromine.....	1.92	1.73	1.98	1.80
Ammonia.....	0.06	0.03	0.46	0.33
Amido compounds.....	1.43	1.25	0.31	1.31
Albumen, digestible.....	10.25	7.68	10.50	7.81
Indigestible nitrogenous substances.....	7.18	9.19	7.68	8.00
Total nitrogenous substances.....	20.84	19.88	20.93	19.25
Fat.....	27.83	30.51	27.34	33.85
Fiber.....	3.36	37.48	39.99	36.06
Nitrogen-free extract.....	38.62			
Water.....	4.30	3.83	6.56	5.41
Ash.....	5.05	8.30	5.18	5.43
Total amount of P ₂ O ₅	1.85	2.52	2.14	2.05
Amount of ash soluble in water.....	3.76	4.76	2.82	2.76
Amount of P ₂ O ₅ soluble in water.....	1.43	0.50	0.74	0.77
Water-soluble ash, per cent total ash.....	74.	57.	54.	49.
P ₂ O ₅ in water-soluble ash, per cent total P ₂ O ₅	77.	19.	34.	37.
Total nitrogen.....	3.68	3.30	3.95	3.57
Nitrogen in form of theobromine.....	0.61	0.55	0.63	0.57
Nitrogen in form of ammonia.....	0.05	0.03	0.36	0.26
Nitrogen in form of amido compounds.....	0.23	0.02	0.05	0.21
Nitrogen in form of digestible albumen.....	1.64	1.23	1.68	1.25
Nitrogen in form of indigestible nitrogenous substances.....	1.15	1.47	1.23	1.28
Nitrogen in form of indigestible nitrogenous substances, per cent total nitrogen.....	31.2	44.5	31.2	35.8

¹*Op. cit.*, note 3, p. 939 of this work.

König and Wesener¹ report a cocoa sweetened with saccharine, of which it contained 0.40 per cent. Another analysis is reported showing 0.76 per cent of saccharine (Chem. Ztg., 1888, 106; also König).¹

Stutzer² has made a careful study of the effect of different processes of manufacture on the chemical constituents of cocoa, the quality of the product, etc. From the results of these investigations, which are given in the table on page 943, he draws the following conclusions:

(1) The roasting processes hitherto used are faulty. The duration of the roasting is too long. The temperature at which the process is carried on can not be accurately regulated in most apparatus in use. Empyreumatic substances having unpleasant odors are imperfectly removed; hence it occurs that the aroma of the cocoa, in contrast with previous opinion, is only developed to be destroyed by the formation of empyreumatic substances.

(2) The faults mentioned disappear when the new, double, centrifugal roasting apparatus is used. This apparatus is much more suited to the purpose than apparatus now in use, and insures a marked economy of time and fuel. The duration of the roasting is short, the temperature of the inner part of the apparatus can be accurately regulated during the roasting and the empyreumatic gases are removed. The husks are more easily removed from the seed after the roasting and the losses of cocoa mass are smaller in consequence of this better separation. The natural aroma of the cocoa is better developed and, consequently, the poorer grades of cocoa can be more profitably marketed than was hitherto possible.

(3) The addition of potash, soda, or ammonia for the opening of cocoa is unnecessary, if the beans are roasted with the new apparatus. The additions at present in use (including ammonia) can be detected with certainty by the analysis of the cocoa powder.

(4) It is for the interest of the public and of the manufacturers that the artificial perfuming of cocoas be abandoned in future, and that only such preparations be brought into the market as contain the natural cocoa aroma in pure unadulterated condition. The technical arrangement of roasting apparatus and the methods of preparation heretofore in use seem to render this artificial perfuming necessary. This, as well as the addition of alkalis or ammonia, becomes unnecessary when Salomon's apparatus is used.

(5) The value of cocoa as a mere pleasant addition to the table depends entirely upon the content of the natural aroma. The finer it is the higher the price that will be paid for the product. The manner of preparation, especially the manner of roasting, in a marked degree influences the development and maintaining of the aroma.

The quantity of the physiologically important constituent appears to vary only slightly with the different kinds of cocoa and different methods of preparation.

(6) The value of cocoa as a nutritive material is essentially dependent on the content of the cocoa powder in digestible albumen. The amount of cocoa butter should not exceed 30 per cent as a rule. The digestible albumen can easily be rendered indigestible by too high a temperature in roasting. By examination of a well-prepared cocoa powder, we found the relation of the quantity of digestible albumen to the quantity of indigestible nitrogenous substances to be nearly 4 : 3. If too high a temperature be used, this relation rises to 4 : 4, or even to 4 : 5. In the four samples investigated, No. 1, which was roasted in C. Salomon's apparatus, shows in this regard the most favorable and the Holland cocoa the most unfavorable relation.

In the same article he gives the following results to show the effect of roasting on the digestible albumen. The figures given are for the

¹ *Op. cit.*, note 8, p. 938 of this work.

² *Op. cit.*, note 3, p. 939 of this work.

per cent of the total nitrogen existing in the form of indigestible nitrogenous substances:

	Raw.	Roasted.
	<i>Per cent.</i>	<i>Per cent.</i>
Ariba beans	13.2	39.7
Machala beans.....	22.8	40.3
Bahia beans	19.3	40.3

A discussion of Stutzer's method for the detection of the use of fixed alkalis and ammonia will be found under the head of methods of analysis and under the head of judgment of samples.

Adulterations of cocoa preparations.—Perhaps no food material offers conditions so favorable for profitable adulteration and so well utilized by its manufacturers as do cocoa preparations. While a discussion of the adulteration of preparations sold in American markets is presented in the tabulated results of investigations given below and in the text accompanying them, a brief review of the favorite substances and methods for adulteration is not considered out of place here.

There is probably no more misleading or more abused term in the English language than the term "soluble cocoa." No cocoa in the market contains a very considerable percentage of matter soluble in water, unless the material so dissolved is foreign soluble material that has been added during the process of preparation. The term seems to be used to denote a preparation that allows none of the insoluble matter to deposit from the beverage prepared from it. This purpose may be accomplished in two ways,—the material may be so finely divided that a very long time will be required for its deposition, or foreign substances (as starch or sugar) may be added to render the liquid of so high a specific gravity, or so pasty, that the insoluble matter will not deposit. The first method is decidedly to be preferred; it accomplishes the object in view and puts the preparation in better condition for the action of the digestive juices—all this without the addition of a cheap diluent that is always at hand in every kitchen, should its use be desired. Any additions of this kind should be considered adulterations unless their nature and quantity are accurately stated.

Attempts at the preparation of easily digestible cocoas (preparations to which pepsin or other digestive ferments have been added do not come in question here, since the favorable condition of the preparation is not involved, but the supplying of a deficiency in the strength of the digestive juices) seem to fail in purpose and to be attended with the introduction of objectionable substances. The use of alkalis for this purpose is quite generally regarded as injurious, and the investigations of Stutzer¹ show that the effect is opposite to that desired. He also states

¹ *Op. cit.*, note 3, p. 939 of this work.

that these substances are used to aid the opening (aufschliessen) of the beans and not for the purpose of increasing the digestibility of the preparation.

The removal of the fat is not considered to be an adulteration when it is acknowledged. It seems important, however, that the public have a means of accurately knowing to what extent it has been removed. Concerning the removal of the fat Macé¹ writes:

The most frequent adulteration of cocoa powder consists in depriving it of a part of its fatty matter, cocoa butter. The best means of detecting this is to estimate the fat by means of ether. Cocoa thus adulterated has lost a great part of its nutritive power, and manufacturers often make great claims for their fraudulent practice, pretending to furnish a product which is more easily digested.

The adulterants added are reported to be, besides sugar and starches, other substances of organic and inorganic origin, to increase the weight and bulk; ferruginous and other pigments to restore the color of highly diluted preparations; and foreign fats to restore the normal percentage of fat or to give the preparation the plasticity required for molding.

As examples of the nature of adulterants reported, the following list of H. W. Warren² may be cited: Sand, ferric oxide, clay, potassium chromate, copper sulphate, and nickel sulphate are used as coloring materials. Finely powdered tin is sometimes added to give the chocolate a metallic luster.

The husk, because of its coarse nature and consequent tendency to act as an irritating substance in the alimentary canal, and in consequence of its poverty in the constituents that render cocoa valuable, is regarded as an adulterant when not removed or when added to increase the weight or bulk of the preparation.

MICROSCOPICAL EXAMINATION.

For a thorough study of cocoa preparations, a microscopical examination is indispensable. An accurate knowledge of the structure of the cocoa bean and of the substances used as adulterants is necessary for the successful carrying out of this investigation. While this information is only to be gained by actual study of the materials in question, its acquirement is greatly facilitated by the use of descriptions and illustrations.

The literature of the subject will be found somewhat contradictory (even with comparatively recent writers) in some details, but nothing of importance in investigations for detection of adulteration seems to be subject for debate at the present time. The works of Moeller and Macé will be found to furnish valuable assistance in investigations of this kind. The microscopical characteristics of the starches and other materials used for adulteration have been so well described in various works and in previous bulletins of this Department that any detailed description of them seems unnecessary here.

¹ Macé, *Les substances alimentaires étudiées au microscope*.

² *Chem. News*, 62, 99.

The cocoa bean is inclosed in a thin, brittle, reddish brown seed coat, called the husk or shell. On the surface of the husk are often found numerous delicate, tubular cells, which come from the pulp of the fruit. The important structures of the husk proper are the following:

- (1) The epidermal layer.
- (2) The loose parenchyma.
- (3) The fiber bundles with small spiral cells.
- (4) The layer of characteristic thick-walled cells.

After softening the husk by soaking the bean in water, a portion of the epidermal layer is readily torn away with the forceps, freed from adhering fragments of the adjacent tissue, and placed on the slide for examination. It is found to consist of a layer of moderately thick-walled, somewhat elongated, irregularly polygonal cells (see Plate XLVI). By careful dissection and careful manipulation of the light and the micrometer screw, a layer of exceedingly delicate, transversely elongated cells can be seen to lie directly under the layer just described, but it is so very delicate that it is rarely seen in the examinations of preparations of cocoa for adulterants, and is consequently of almost no importance in such investigations. If some of the underlying tissue exposed by the removal of the epidermis be transferred to a slide and dissected apart, it will appear as a mass of loosely aggregated, rather large, thin-walled, slightly elongated cells, those constituting the inner layers containing a large amount of mucilaginous matter that swells up in contact with water and ruptures them. This parenchymatous tissue, which makes up the greater part of the husk, is pierced in all directions by small, ramifying fiber bundles inclosing small spiral cells and stone cells; near the inner surface of the husk it is interrupted by a single layer of small, very thick-walled cells (see Plate XLVI), which are very characteristic and withstand the disintegrating processes of manufacture better than any other part of the husk. In fact, these processes are often carried so far that it is only by very diligent search that one is able to find any recognizable structures besides these cells and the starch grains of the cotyledons. For the detection of the presence of husks in cocoa preparations, these thick-walled cells are first sought for; and after these the epidermal and parenchymal structures. The fiber bundles, with inclosed spiral cells, are not readily distinguished from those of the cotyledon.

If the brown husk be entirely removed from the remaining part of the bean, a thin, transparent membrane will be observed, which partly comes away with the husk and partly remains adherent to the bean proper, dipping into all the clefts and plications of the latter. When a portion of this membrane is examined with the microscope, it appears as a single layer of small polygonal cells which are filled with granular matter (see Plate XLVII). By careful manipulation one or more layers of parenchymatous cells can be found underneath the layer just described. Adherent to this membrane, especially to the folds entering the clefts of the cotyledons, are numerous yellow, club-shaped masses

of cells, known as "Mitscherlich bodies." They are now considered to be epidermal hairs, but it is not decided as to whether they belong to the membrane just described or to the surface of the cotyledon. Neither the settlement of this question nor the membrane and hairs, are of any great importance for our purpose, since both of these structures are very rarely met in recognizable form in the commercial preparations.

After the removal of the husk and the membrane just described, the two fleshy, much-folded cotyledons, or seed leaves, remain, inclosing the radicle (the embryo stem of the undeveloped plant) at the larger end of the bean in a manner not a little suggestive of the retracted head of a turtle. Examination of a thin section shows the cotyledon to be made up of comparatively thin-walled, closely packed, polygonal cells (see Plate XLVII). The most of these cells are filled with starch grains, fat, and albuminous material; isolated or small groups of cells are homogeneously filled with the reddish brown to violet pigment, cocoa red. Fiber bundles with spiral cells, similar to those of the husk, occur in the veins.

The starch grains are spherical, or nearly so, have a very indistinct nucleus, react only feebly with polarized light, rapidly lose the blue coloration imparted by iodine, and show only a slight tendency to unite in the form of compound grains; three or four is usually the maximum number so united. The size of the grains of cocoa starch is usually stated to be 0.003-0.010 mm. The following measurements were made in this laboratory:

Measurements of starch grains from a sample of unprepared cocoa beans.

	Millimeters.
5 grains measured.....	0.003
11 grains measured.....	0.004
14 grains measured.....	0.005
6 grains measured.....	0.006
11 grains measured.....	0.007
2 grains measured.....	0.008
1 grain measured.....	0.009
1 grain measured.....	0.010
51 Average of 51 grains	0.0054

Measurements of starch grains from Walter Baker & Co's. "cracked cocoa."

	Millimeters.
3 grains measured.....	0.002
9 grains measured.....	0.003
12 grains measured.....	0.004
14 grains measured.....	0.005
14 grains measured.....	0.006
28 grains measured.....	0.007
7 grains measured.....	0.008
3 grains measured.....	0.009
8 grains measured.....	0.010
2 grains measured.....	0.012
100 Average of 100 grains	0.0062

For the preparation of the sample for microscopical examination, a convenient portion is washed with ether by decantation in a small test tube; when the ether has evaporated from the residue, a portion is triturated in a small mortar with a small quantity of solution of chlorinated soda until nearly or just decolorized; the mixture is then washed into a large test tube, or conical glass, diluted with much water, and set aside to settle. The sediment thus obtained is removed with a pipette for the microscopical examination. If both color and form are found necessary for the identification of any structures, the reserved portion of unbleached and fat-free material is used for this purpose.

A knowledge of the structures of the spices used for flavoring cocoa preparations, sufficiently accurate to enable one to readily distinguish them from substances added as adulterants, is very important; to identify the individual spices when several have been used in the same preparation is a very difficult and generally a highly unimportant problem. The ordinary flavoring materials are vanilla and cinnamon; cloves, nutmeg, mace, cardamom, Peruvian balsam, and artificial vanillin are also reported as flavoring materials.

Starch, flour, and husks are the more ordinary adulterants to be detected with the microscope; Moeller¹ reports the materials most commonly used to be wheat and potato starches, next rice and arrowroot, and finally wheat, acorn, and rye flours, ground peanuts, and in malted preparations raw malt as a substitute for malt extract; to these Macé² adds almond cake and sawdust. As the collecting of valueless waste products and the converting them into suitable materials for the adulteration of commodities of value has become a well-established industry, every investigator must be prepared to find many new substances used for this purpose, and must not be disappointed if he finds none of those that have recently been reported. To know what to expect to find, he should consider the current prices, abundance, and suitability of available materials, as well as the results of previous investigations.

METHODS OF ANALYSIS REPORTED BY VARIOUS CHEMISTS.

A brief review of the methods that have been used or recommended for the analysis of cocoa and its preparation is here given. If any important recently reported methods have been omitted, it is due to a greatly regretted and unintentional oversight.

Preparation of the sample.—The method of analysis adopted by the Association of Swiss Analytical Chemists³ directs that the substance should always be finely powdered or rasped, and kept in well-stoppered bottles. Bensemann⁴ prepares the beans by grinding in a porcelain mortar, separates the husk by use of a knife, and

¹ *Op. cit.*, note 3, p. 940 of this work.

² Macé, *Les substances alimentaires étudiées au microscope*.

³ *Vierteljahresschrift ii. d. Fortschritte auf d. Gebiete d. Chem. d. Nahr. u. Genussmittel*, 1890, **2**, 171.

⁴ *Rep. f. anal. Chem.*, 1884, **4**, 213.

scrapes chocolate with a knife to prepare it for analysis. Mansfeld¹ recommends that the material be finely rasped or powdered.

Determination of moisture.—The Association of Swiss Analytical Chemists² makes this determination in cocoa powder. Two grams are spread in a thin layer on a watch glass and dried to constant weight at 105° C. Bensemann³ dries 2 grams to a constant weight at 100° C.; Herbst,⁴ a weighed quantity, mixed with twice its weight of sand, at 60° to 80° C.; Mansfeld,¹ 5 grams to constant weight at 100° C.

Quantitative and qualitative examination of the ash.—For quantitative determination, the method of the Association Swiss Analytical Chemists² directs that the material be incinerated in a platinum dish, carefully burned at a low red heat until the ash is white, and after moistening with $(\text{NH}_4)_2\text{CO}_3$, gently reignited. The ash is also examined qualitatively for mineral substances and pigments; as ocher, bolus, excess of alkaline carbonates used in making the cocoa soluble, etc.

Bensemann,³—Two grams of material are carbonized, extracted with water, and the residue washed and incinerated. The aqueous extract is added to the ash and the whole evaporated to dryness. The residue is dried at 100° C., weighed, and the result taken as ash. All ashes were of a light gray, almost white, color, so that the addition of ocher, bolus, etc., could be easily detected by a red color of the ash.

Mansfeld¹ burns 5 grams at a low temperature and tests the ash for mineral additions; as ocher, excess of alkaline carbonates, etc.

Stutzer⁵ mixes 2 grams with sand, incinerates at a moderate heat, adds NH_4NO_3 , and ignites again until the ash is white and free from CO_2 and nitrates. In a recent article⁶ this writer has called attention to the value of the additional determinations of ash soluble in water, total P_2O_5 and P_2O_5 soluble in water, as a means of detecting the use of fixed alkalis and ammonia in the process of manufacture. His results are given in the table on page 943.

Quantitative determination of fat.—Association of Swiss Analytical Chemists.²—The sample is mixed with sand, extracted with absolute ether in a Soxhlet apparatus for six to eight hours; the ether is removed by careful evaporation, and the fat dried at 100° C.

Bensemann,³—Two grams of the substance are rubbed to a powder in a mortar warmed to 60° C., some gypsum being added to facilitate the pulverizing. The mass is extracted with ether in a Soxhlet apparatus and the extract is dried at 100° C.

Blyth,⁷—The best method of extracting the fat is to exhaust the nibs with ether in a Soxhlet apparatus.

Boussingault,⁸ mixes the material with sand and extracts with carbon disulphide.

Hassall,⁹—Three grams of cocoa are dried in the water bath and exhausted with ether; the ether is evaporated off, and the residue dried and weighed. This residue contains some or all of the theobromine, the amount of which must be determined and subtracted from the ether extract.

Herbst,⁴—The residue from the moisture determination is extracted with ether and the extract dried at 100° C.

¹ Zeitschr. d. allgem. österr. Apotek. Ver., **44**, 329.

² Vierteljahresschrift ü. d. Fortschritte auf d. Gebiete d. Chem. d. Nahr. u. Genussmittel, 1890, **2**, 171.

³ Rep. f. anal. Chem., 1884, **4**, 213.

⁴ Rundschau, 1882, 443; Rep. d. anal. Chem., **2**, 236; Zeitsch. f. anal. Chem., **22**, 278.

⁵ Rep. f. anal. Chem., 1882, 88 and 165; Hygiene-Bericht, 1882-'3, **1**, 217.

⁶ Op. cit., note 3, p. 939 of this work.

⁷ Op. cit., note 3, p. 936 of this work.

⁸ Ann. Chem. Phys., [5], **28**, 433; Jour. Chem. Soc., 1884, 46, 202; Chem. Ztg., 1883, 203, and 902.

⁹ Op. cit., note 2, p. 940 of this work.

Mansfeld.¹—Five grams of cocoa, or 10 grams of chocolate, are mixed with an equal weight of sand and extracted in a Soxhlet apparatus with petroleum ether.

Wolfram² uses petroleum ether for the extraction of the fat.

*Qualitative examination of the fat.*³—The Association of Swiss Analytical Chemists⁴ recommends the determination of the melting point by Rühdorf's method, Hübls' iodine number, and Köttstorfer's saponification number. Björklund's ether test and Filsinger's ether-alcohol test are also stated to be of value.

Björklund⁵ covers about 3 grams of the fat in a test tube with double its weight of ether, closes with a cork, and tries to bring the mass in solution by shaking at 18° C. When wax is present, a cloudy solution results, which is not changed by warming. If the solution is clear, the tube is placed in water at 0° C. and the time observed after which the solution begins to become milky or to deposit white flakes; then the temperature is noted at which the mixture becomes clear on removing from the water. When the solution becomes cloudy after ten to fifteen minutes and at 19° to 20° C. is again clear, the cocoa butter is pure. For a cocoa butter containing 5 per cent of beef tallow, these numbers are eight minutes and 22° C.; 10 per cent tallow, 7 minutes and 25° C., etc.

Dieterich.⁶—Equal parts of the fat and paraffin are melted together, a drop of the mixture placed on a slide and covered with a cover glass. After twelve hours this is examined with a power of 20 diameters and polarized light, at a temperature not exceeding 5° C. Pure cocoa butter shows palm-leaved crystals; 10 per cent of tallow, circular group of crystals.

Filsinger.⁷—The iodine and Köttstorfer's numbers are determined in the dried fat. If these leave any doubt, Björklund's ether test or Filsinger's ether-alcohol test may be applied. This writer has modified the ether test as follows: 2 grms. of fat are melted in a graduated tube with 6 cc. of a mixture of 4 volumes of ether (sp. gr. 0.725) and 2 volumes of alcohol (sp. gr. 0.810), shaken and set aside. The pure fat gives a solution that remains clear.

Hager's anilin test⁸ is conducted as follows: About 1 gram of cocoa butter is warmed with 2 to 8 grams of anilin until dissolved; the mixture is allowed to stand 1 hour at 15° C., or 1½ to 2 hours, when temperature is 17° to 20° C. Pure cocoa butter floats as a liquid layer on the anilin. If the cocoa contains tallow, stearic acid, or a little paraffine, cloddy particles, which remain hanging on the upper wall, on gentle agitation are deposited in the oil layer; if wax or much paraffin is present, the oil layer solidifies; if much stearic acid is present, there will be no separation into two layers, but the whole solidifies to a hard crystalline mass; with pure cocoa butter, the oil layer hardens only after many hours. A parallel test should be made with pure cocoa butter.

Hassall.⁹—Melting point is determined. Foreign fats become rancid and tallowy in a few days.

Herbst.¹⁰—Melting point determination and Björklund's ether test are recommended.

¹ *Op. cit.*, note 1, p. 950 of this work.

² Jahresbericht d. k. chem. Centralstelle f. öff. Gesundheitspflege in Dresden, 1878; Zeitsch. f. anal. Chem., **18**, 346.

³ See table on page 938 for the chemical and physical constants for cocoa butter.

⁴ *Op. cit.*, note 3, p. 949 of this work.

⁵ Zeitsch. f. anal. Chem., **3**, 233; see also *op. cit.*, note 2, p. 938 of this work.

⁶ Geschäfts-Ber. d. Papier- u. chem. Fabrik in Helfenberg, 1883; Zeitsch. f. anal. Chem., **23**, 567.

⁷ Zeitsch. f. anal. Chem., **19**, 247; Chem. Ztg., 1889, **13**, 309; see also *op. cit.*, note 2, p. 938 of this work.

⁸ Zeitsch. f. anal. Chem., **19**, 246; see also *op. cit.*, note 2, p. 938 of this work.

⁹ *Op. cit.*, note 2, p. 940 of this work.

¹⁰ *Op. cit.*, note 4, p. 950 of this work.

The small quantity of sesame oil, added to give the broken surface of chocolate a smooth appearance, can not be detected with certainty.

Mansfeld.¹—The purity of the fat is determined according to Filsinger. The melting point is determined according to Pohl.

Schädler.²—The comparatively high commercial value of cocoa butter brings adulteration with waxes, stearin, paraffin, and beef tallow. The taste, odor, melting point, and ether test are mentioned as means of detecting foreign fats. Paraffin gives cocoa butter a soapy feel and lowers the specific gravity. An addition of stearic acid is made known by the high melting point and by boiling with dilute NaOH , when the stearic acid goes into solution as stearate of sodium and is reprecipitated by H_2SO_4 .

Determination of theobromine.—Blyth.³—This author outlines the methods of Woskressnsky, Mitscherlich, and Wolfram. He also gives the following "speedy method of determining, with fair exactitude, the per cent of theobromine in cocoa": Weigh out a definite portion and exhaust it with petroleum ether. Mix the residue with a little burnt magnesia and water, evaporate to dryness at 60° to 70°C. , and exhaust the residue with boiling 80 per cent alcohol, which dissolves out the theobromine. After driving off the alcohol, the residue may be purified for weighing by washing with petroleum ether.

Boussingault.⁴—Extract the sample with boiling water and precipitate the decoction obtained with basic acetate of lead. After removing the excess of lead with H_2S , evaporate to dryness and exhaust the residue with boiling alcohol. On cooling this solution the alkaloid separates out as a crystalline powder.

Hassall⁵ uses the method devised by Gehner for the estimation of caffeine in tea, which is conducted as follows: Twenty grams of material are boiled with about a liter of water, cooled, and the solution made up to the mark and filtered. Five hundred cc of the clear filtrate are evaporated on the water bath with the addition of a little MgO . The dry residue is extracted with boiling alcohol. The united extract is evaporated nearly to dryness, taken up with ether, filtered, and evaporated to dryness for weight.

Legler⁶ gives the following modification of Wolfram's method: 20 to 25 grams of cocoa, or 50 grams of chocolate, deprived of fat, are digested several hours with 4 per cent H_2SO_4 . The solution is filtered and the theobromine precipitated with sodium phosphomolybdate. After standing twenty-four hours, filter, wash with 6 to 8 per cent H_2SO_4 , and dissolve the precipitate in NaOH or Na_2CO_3 . Add sufficient H_2SO_4 to the solution to leave it slightly alkaline, evaporate with sand, dry at 110°C. , and extract at 70 to 90°C. , with amyl. alcohol. Evaporate the extract to dryness in a platinum dish, dry, and weigh. Ignite the residue and weigh again. The difference is the weight of the theobromine.

Mansfeld.¹—Another portion of 50 cc of the alcoholic extract, obtained by the author's method for the determination of sugar (see methods for determination of sugar on page 954), is evaporated to dryness with MgO and the finely powdered residue extracted in a Soxhlet's apparatus with CHCl_3 . The extract is evaporated to dryness and the residue dissolved in boiling water. The solution is filtered, evaporated to dryness, and the residue of theobromine weighed; the alkaloid thus obtained is pure.

Mulder.⁷—Ten grams of cocoa are rubbed to a paste with water and boiled for fifteen minutes; some MgO is now added and the mixture evaporated to dryness on the water

¹ *Op. cit.*, note 1, p. 950 of this work.

² *Op. cit.*, note 2, p. 936 of this work.

³ *Op. cit.*, note 3, p. 936 of this work.

⁴ *Op. cit.*, note 2, p. 941 of this work.

⁵ *Op. cit.*, note 2, p. 940 of this work.

⁶ *Ber. d. Chem. Ges.*, **15**, 2938; *Zeitsch. f. anal. Chem.*, **23**, 89.

⁷ *Op. cit.*, note 8, p. 938 of this work.

bath with continual stirring. The residue is extracted with CHCl_3 and the chloroform distilled off. The residue is dissolved in hot water, the solution filtered, evaporated to dryness, and the residue of theobromine dried and weighed. This method includes the traces of caffeine which are present in cocoa. This may be isolated by extracting the residue of theobromine with cold benzol, which dissolves only the caffeine. The benzol is distilled off, the residue shaken with water, and the solution filtered and evaporated.

Weigmann.¹—Twenty grams of the material are rubbed up into a soft paste with hot water; more water is then added and the mixture is boiled for fifteen to thirty minutes. The decoction is then made up to one liter, allowed to settle, and 500 cc filtered off. This is brought to a boil and precipitated with ferric acetate. The precipitate is filtered off, the filtrate concentrated on the water bath, strongly acidulated with H_2SO_4 (the liquid should contain at least 6 per cent H_2SO_4), and precipitated with sodium phosphomolybdate. After standing two to three hours the precipitate is filtered off, washed with acidified water, and its content of nitrogen determined after drying. The results thus obtained are somewhat lower than Wolfram's.

Wolfram.²—If the bean deprived of husks is to be examined, it is first rubbed to a paste in a hot mortar. Ten grams of this mass, or 20 to 30 grams of chocolate, are treated for some time with boiling water, ammoniacal lead acetate added, the solution filtered hot, and the precipitate washed until a drop of the filtrate after cooling gives no precipitate with sodium phosphomolybdate. A volume of 700 to 800 cc is generally necessary. After the addition of NaOH , the filtrate is evaporated to 50 cc. It is then strongly acidulated with H_2SO_4 and the lead sulphate separated by filtration. The filtrate is mixed with a large excess of sodium phosphomolybdate (this reagent is prepared by dissolving 100 grams of sodium molybdate and 60 to 80 grams of sodium phosphate in 500 cc of water acidulated with about 6 per cent of nitric acid). Heating and stirring facilitates the settling of the precipitate. After standing several hours the liquid is filtered and the precipitate washed with 6 to 8 per cent H_2SO_4 . The filter and precipitate are placed in a beaker and $\text{Ba}(\text{OH})_2$ added until the reaction is alkaline. Heating renders the decomposition more rapid. The excess of $\text{Ba}(\text{OH})_2$ is neutralized with H_2SO_4 , and any possible excess of the latter with BaCO_3 . The whole is filtered and washed hot; the filtrate is evaporated in a platinum dish and the theobromine dried and weighed. As barium salts may be present, it is best to ignite, moisten with $(\text{NH}_4)_2\text{CO}_3$, reignite and weigh. The difference is theobromine.

Zipperer.³—The substance is extracted with petroleum ether and then three times extracted with 80 per cent alcohol. The alcoholic extracts are evaporated to dryness on the water bath with 15 grams of $\text{Ca}(\text{OH})_2$. The dry residue is extracted with CHCl_3 , the latter distilled off, the residue dissolved in hot water, the solution filtered and evaporated to dryness. The resulting theobromine is dried and weighed. The results are regarded as unreliable, since the extraction is very questionable.

Determination of sugar by polarization.—Filsinger.⁴—13.024 grams are mixed with water in a 100 cc flask. The solution is clarified with basic lead acetate, made up to the mark, filtered, and polarized in a 200 mm tube.

Mansfeld.⁵—Ten grams of chocolate are heated with 100 cc water in a 250 cc flask to 35°C ., well mixed, clarified with lead acetate and alum, and made up to the mark. The clear filtrate is polarized.

Determination of sugar by inversion.—Ass. Swiss Anal. Chem.⁵—The substance is

¹ *Op. cit.*, note 8, p. 938 of this work.

² *Op. cit.*, note 2, p. 951 of this work.

³ Zipperer, *Untersuch.* ii. Cacao u. dessen Präparate, 1887; see also *op. cit.*, note 8, p. 938 of this work.

⁴ *Op. cit.*, note 7, p. 938 of this work.

⁵ *Op. cit.*, note 1, p. 950 of this work.

extracted with water; the extract is evaporated to a sirupy consistency, treated with alcohol, decolorized by means of lead acetate, the excess of which is removed with H_2SO_4 . The sugar thus prepared is inverted and the invert sugar determined with Fehling's solution.

Hassall.¹—The sugar is dissolved out of the sample with cold water, inverted by boiling with dilute H_2SO_4 , and then estimated with copper solution.

Mansfeld.²—The residue from the fat determination is extracted for three hours with 100 cc of 80 per cent alcohol, and the extract made up to 150 cc. Fifty cc are evaporated to dryness. After weighing, the residue is dissolved in water, made up to 100 cc, heated one-half hour on the water bath with 10 cc $\frac{\text{N}}{2}\text{HCl}$, neutralized with NaOH , and diluted to 250 cc. The invert sugar is then gravimetrically determined with Fehling's solution.

Determination of sugar by direct weighing.—Hassall.¹—Dissolve a weighed quantity of cocoa containing sugar in cold water and collect the residue on a filter. This residue is dried on the water bath and weighed.

100—per cent of insoluble matter=per cent of soluble matter.

Per cent of soluble matter — (per cent of moisture + the approximate per cent of soluble matter in cocoa) = per cent of sugar.

Herbst.³—The residue from the fat determination is extracted with boiling 50 per cent alcohol as long as the extract is colored. This extract is evaporated to dryness and treated with cold water, which dissolves the sugar. This solution is evaporated to dryness, the residue dried in hydrogen and weighed.

Determination of commercial glucose in chocolates.—In regard to the determination of the sugars used for sweetening chocolates, M. Schreder⁴ writes as follows:

"The detection and estimation of less than 5 per cent of commercial glucose in presence of cane sugar by means of copper solution is uncertain, because commercial cane sugar often contains an equivalent amount of reducing sugars. The optical determination by Clerget's method is more satisfactory, since only a small percentage of commercial glucose is necessary to appreciably lessen the degree to which the inverted solution rotates to the left. The evidence obtained by Clerget's method can be strengthened by determining the dextrin which would be present in the residue after fermentation, if commercial glucose were present in the original material."

Determination of starch.—Asborth⁵ has published a method, according to which he adds $\text{Ba}(\text{OH})_2$ to the boiled starch, with which it forms a compound which is insoluble in dilute alcohol. Dr. Mansfeld's modification of this method is given below.

Ass. Swiss Anal. Chem.⁶—The sample, free from fat and sugar, is boiled with water for four hours and the resulting starch solution is inverted with sulphuric acid; the excess of acid is removed by basic acetate of lead; the filtrate is freed from lead with H_2S and the reducing sugars determined by means of Fehling's solution. The starch may also be determined by treating the powder in a Reischauer's pressure-flask, inverting, etc.

Bensemann.⁷—Two grams of the substance, after extraction and washing with cold water, and while still moist, are mixed with 200 cc of water and 20 cc HCl , sp. gr. 1.12. The mixture is heated for at least three hours on the water bath, cooled, and filtered. After making the filtrate alkaline with NaOH , a freshly prepared solution of 4 grams of copper tartrate, 2 grams of tartaric acid, 30 cc of soda lye, sp. gr. 1.13, and 100

¹ *Op. cit.*, note 2, p. 940 of this work.

² *Op. cit.*, note 1, p. 950 of this work.

³ *Op. cit.*, note 4, p. 950 of this work.

⁴ *Zeitsch. f. angew. Chem.*, 1892, 173.

⁵ *Rep. anal. Chem.*, 8, 20.

⁶ *Op. cit.*, note 3, p. 949 of this work.

⁷ *Op. cit.*, note 4, p. 949 of this work.

cc water are added. The mixture is slowly heated to 70 to 80° C. and kept at that temperature for one-half hour. After completely cooling the Cu_2O is collected on a filter, washed cold, dried at 100 to 110° C., and weighed.

1 gram Cu_2O = 0.45315 grams of starch.

Hassall¹ gives a method similar to that of the Ass. Swiss. Anal. Chem. given above.

Mansfeld.²—Two portions of 25 grams of cocoa, or 5 grams of chocolate, are weighed out and placed in 250 cc flasks with 100 cc of water in each. One sample is heated on the water bath; the other is placed in water at 30 to 40° C., and shaken to emulsify the fat. After thirty minutes the cooled samples are shaken with 50 cc of a standard solution of $\text{Ba}(\text{OH})_2$ and 45 per cent alcohol added until the liquid reaches the mark. Cool and make up with similar alcohol if necessary. The $\text{Ba}(\text{OH})_2$ solution is titrated by mixing 50 cc with 100 cc of water and making up to 250 cc with 45 per cent alcohol, using $\text{N}/_{10}$ HCl with phenolphthalein for an indicator, and titrating 50 cc. The sample is allowed to settle, and 50 cc of the yellow supernatant liquid titrated. The difference between these two titrations for pure chocolate is 1.25 cc. The general increase for each per cent of flour is 0.3 cc. Hence:

$$X = 10 \frac{D - 1.25}{3}$$

$$D = \text{number of cubic centimeters difference between the amounts of } \text{N}/_{10} \text{HCl required. } X = \text{per cent of foreign starch in the sample.}$$

Mansfeld.³—The residue left after the removal of petroleum ether and alcohol extracts is dried and mixed with 500 cc of water; the mixture is heated for one-half hour on the water bath, diluted to 1,000 cc, cooled to 55° C., and mixed with 0.1 gram of Lintner's diastase, which has been previously rubbed up with a little water. Keep at 55° to 60° C. until iodine gives no reaction for starch, decant into a 1,500 cc flask and make up to the mark. Heat 100 cc of the clear liquid with 10 cc HCl , sp. gr. 1.125 for three hours on the water bath, cool, neutralize, dilute to 500 cc and determine the dextrose gravimetrically.

Schroeder.⁴—For the inversion of the starch, 3 grms. of the material with 50 cc of water and 1 cc HCl (38.8 per cent) are heated for 1 hour under a pressure of one atmosphere. Results of experiments are also reported to show that this treatment does not convert an appreciable amount of cellulose into dextrose.

Weigmann⁵ uses diastase solution prepared as directed by Stutzer. Ten grams of cocoa, deprived of fat, are boiled one quarter hour with water and made up to 500 cc; 250 cc are removed after shaking, treated with 2 cc of diastase solution for four hours at 60° C., inverted with 20 cc HCl , neutralized and precipitated with lead acetate. After removal of the excess of lead with H_2SO_4 , the filtrate is made up to 500 cc and the reducing sugar determined gravimetrically.

Detection of flour in cocoa preparations.—Reinsch.⁶—Boil one part of the material with ten parts of water, cool and filter. If the sample is pure it will filter rapidly, give a clear filtrate, having a light reddish color, and leave a residue that is not gummy. If flour has been added, it filters murky and slowly, and a gummy mass remains on the filter.

Determination of fiber.—Ass. Swiss Anal. Chem.⁷ used Henneberg and Stohman's method. This, as well as a method given by Mansfeld, does not differ materially from the official method of the Association of Official Agricultural Chemists, an outline of which is given on page 958.

¹ *Op. cit.*, note 2, p. 940 of this work.

² *Zeitsch. f. Nahrungsmittel-untersuchung u. Hygiene*, 1888, **1**, 2; *Deutsch. Chem. Ztg.*, **3**, 91.

³ *Op. cit.*, note 1, p. 950 of this work.

⁴ *Zeitsch. f. angen. Chem.*, 1892, 173.

⁵ *Op. cit.*, note 8, p. 938 of this work.

⁶ *Fürther Gewerbe Ztg.*, 1868, 63; *Zeit. f. anal. Chem.*, **8**, 514.

⁷ *Op. cit.*, note 3, p. 949 of this work.

Determination of water-insoluble organic matter.—Bensemann.¹—Two grams of the substance are covered with cold water and allowed to stand twelve hours, with occasional stirring. The insoluble residue is collected, washed cold, dried at 100° C., weighed, incinerated, and weighed again. Difference = weight of water-insoluble organic matter.

Determination of gum.—Bonssingault.²—An aqueous infusion of the fat-free material is precipitated with alcohol.

Determination of nitrogen.—Mansfeld³ used Kjeldahl's method, starting with 3 grams of material.

Determination of coloring matter and tannin.—Mansfeld.³—The residue from the fat determination (see page 951) is extracted with 80 per cent alcohol. In this extract sugar, theobromine, and total residue are determined. Total residue—(sugar + theobromine) = coloring matter and tannin.

Detection of the use of fixed alkalis and ammonia in the process of manufacture of cocoas.—Stutzer.⁴—The total ash, ash soluble in water, total P_2O_5 and P_2O_5 soluble in water are determined. The relative proportions of these constituents in the ash of a normal cocoa and in the ash of cocoas treated with fixed alkalis and ammonia are given in the table on page 943. Additional evidence of the use of ammonia is obtained by distillation of the sample with magnesia and determination of the ammonia in the distillate. If this process yields more than 0.1 per cent of nitrogen in the form of ammonia, this writer considers the result certain evidence of the use of ammonia, or ammonia salts, in the process of manufacture.

METHODS BY WHICH THE RESULTS GIVEN IN THE TABLE ON PAGE 980 WERE OBTAINED.

The methods outlined below were chosen as best suited for the purpose. While some are only approximate methods, they are sufficiently accurate for the purpose and are much more rapid than some of the methods previously employed.

Microscopical examination.—The method used for the preparation of the sample for the microscopical examination has been outlined on page 949.

Determination of moisture, ash, and of the amount of acid required to neutralize the ash from two grams of material.—Two grams of material are distributed over the bottom of a flat platinum dish and dried to constant weight at 100 to 105° C. The loss = moisture. The residue is carefully ignited in a muffle and weighed for the amount of ash. The ash is then washed into a beaker, dissolved, with gentle boiling, in $\frac{N}{10}$ H_2SO_4 , and the excess of acid determined by titration with $\frac{N}{10}$ $NaOH$. Cc of $\frac{N}{10}$ H_2SO_4 — cc of $\frac{N}{10}$ $NaOH$ = the number given in the table on page 980 as "acid equivalent."

Determination of fat.—Three grams of the substance are placed in a 300 cc flask and covered with 200 to 250 cc of redistilled petroleum ether; after standing 4 to 5 hours with occasional shaking, the flask is filled nearly to the mark with petroleum ether, shaken, and allowed to stand over night. After making up to the mark and thorough shaking, the insoluble portion is allowed to deposit; the supernatant liquid is then quickly decanted into a 100 cc flask until the latter is just filled to the mark, using every precaution to avoid raising the temperature of either flask. This portion of 100 cc is filtered through a Gooch crucible, and the slight residue washed

¹ *Op. cit.*, note 4, p. 949 of this work.

² *Op. cit.*, note 2, p. 941 of this work.

³ *Op. cit.*, note 1, p. 950 of this work.

⁴ *Op. cit.*, note 3, p. 939 of this work.

with petroleum ether until free from fat. The filtrate and washings are received in a weighed flask, in which the fat is weighed, after recovering the petroleum ether by distillation and drying the residue to constant weight at 100° C. in a water oven. The fat obtained is clear, of slightly yellowish color, and undergoes appreciable oxidation only on prolonged heating under the conditions mentioned above. An ordinary air bath is usually too small for the proper heating of a vessel of the size required. Taking the density of the insoluble portion as 1.000, and ignoring the small amount of residue decanted with the 100 cc portion, the following table of corrections is applicable to the per cent of fat obtained by the above method:

When the sample contains approximately	60	} per cent of fat, subtract	0.24	} per cent from the result obtained.
	55		0.25	
	50		0.25	
	45		0.25	
	40		0.24	
	35		0.23	
	30		0.21	
	25		0.19	
	20		0.16	
	15		0.13	
	10		0.09	
	5		0.05	
	1		0.01	

Determinations of fat are very quickly made by this method, and the results are sufficiently accurate for ordinary purposes.

Determination of sugar.—A determination of the amount of sugar added in the process of manufacture is readily made, by means of the polariscope, to within one or two per cent of the truth; a closer determination is neither very easy nor very important. The gum (see page 941) gives the aqueous solution of normal cocoa a slight rotatory power, equivalent to 0.3 to 2.0 per cent of sugar in the bean in several samples tested; the presence of starch necessitates the use of cold water, of which 500 cc or more are necessary for the complete removal of the sugar from 13.024 grams of material. Considering these facts, the following method will be found satisfactory for most purposes: 13.024 grams of material are placed in a small mortar and triturated with alcohol until a smooth paste is obtained; this is transferred to a 500 cc flask, diluted with 400 to 450 cc water and shaken occasionally for three to four hours; 10 cc of a saturated solution of normal acetate of lead are added and the volume brought to 500 cc. After standing for about one hour, with occasional shaking, the solution is filtered and polarized in a 400 mm tube. The per cent of sugar is then obtained by the following formula, in which R = the polariscopic reading, when the normal quantity for the polariscope used is 26.048 grams:

$$\frac{R \left[500 - \left(13.024 - \frac{5 R \times 13.024}{100} \right) \right]}{100} = \% \text{ sucrose.}$$

A portion of the solution, as prepared for polarization, was freed from lead and tested with Fehling's solution for reducing sugar, the result being taken as an indication of the quality of the sugar used in the manufacture of the sample.

For a more exact determination of the sugar, a gravimetric determination of the reducing sugars in the aqueous extract, before and after inversion, is recommended. (Weight of copper obtained after inversion) — (weight of copper obtained before inversion) = weight of copper equivalent to cane sugar present.

Determination of starch.—Mr. K. P. McElroy devised the following method for the determination of starch:

Five grams of chocolate were weighed into an Erlenmeyer flask, wet with alcohol, and 30 to 40 cc of water added. The flask was then shaken at intervals until all sugar present had gone into solution. Fifteen cc of a saturated solution of neutral

lead acetate were then added and the mixture again shaken. After allowing it to settle, the clear liquid was decanted through an asbestos filter and water added to supply its place. This was in turn decanted, and so on until the filter began to clog, when just enough acetic acid was added to the material in the flask to turn it red. Decantation and filtration were then continued till the filtrate came through colorless. Under these conditions filtrations were rapid and filtrate extremely clear. The asbestos filter and contents were then placed in the flask, water enough added to bring the whole to about 100 cc, and the flask placed on the steam bath for three hours. At the end of this time 10 cc of concentrated HCl were added, and the heating continued three hours more. After cooling, Na_2CO_3 was added until the contents of the flask became blackish. The whole was now washed into a 250 cc flask and made up to the mark. Dextrose was oxidized by Allihn's method, 25 cc of solution being used, and the resulting Cu_2O collected on a Gooch crucible, redissolved in HNO_3 , converted into CuSO_4 , and the Cu determined electrolytically.

Five samples of commercial starchy materials were examined for the purpose of determining the amount of starch converted to dextrose by the method employed. Three grams of material were heated on the steam bath for three hours with 200 cc of water; 20 cc of HCl. (sp. gr. 1.125) were then added and the heating continued three hours longer. After neutralization with Na_2CO_3 dilution to a definite volume, and filtration from the slight residue, the dextrose was determined by Allihn's method, the amount of Cu_2O being determined electrolytically. Duplicate samples were inverted and duplicate weighings made from each inverted solution. The results were as follows:

Percentage of starch in commercial starchy materials used in manufacture of cocoa preparations, i. e., per cent of dextrose obtained by inversion $\times 0.9$.

[All samples were air-dried.]

Character of sample.	Solution No. 1.		Solution No. 2.		Mean.
	A.	B.	A.	B.	
Wheat flour.....		71.3	71.6	71.8	71.6
Bermuda arrowroot.....	82.5	82.3	81.9	81.7	82.1
Corn starch.....	83.6	83.8	84.2	83.8	83.8
Potato flour.....	81.9	81.7	79.3	79.2	80.5
Prepared cassava starch.....	83.7		84.5		84.1

Starch determinations are only reliable when the conditions are held rigidly exact. In order that this may be made more certain each set of determinations should be accompanied by a check determination with material of known dextrose-yielding power.

Determination of fiber.—The official method of the Association of Official Agricultural Chemists¹ was adapted to this work, as follows: Two grams of material were placed in a long narrow test tube with a lip, washed with several portions of ether, decanting through a Gooch crucible. After evaporation of the ether, the material in the tube and crucible was washed into an Erlenmeyer flask with 200 cc of 1.25 per cent H_2SO_4 . After boiling a half hour with a reflux condensing tube, the solution was filtered through a linen filter, and the residue thoroughly washed with hot water; while still hot, the filtrate was refiltered through a Gooch crucible, and the slight residue again thoroughly washed with hot water. The material on the linen and in the crucible was rinsed back into the flask with 200 cc of 1.25 per cent NaOH, and the boiling and filtration repeated. The material on the linen was then washed into

¹ Bulletin No. 31, Chem. Div., U. S. Dept. of Agriculture.

a beaker with alcohol and transferred to the crucible through which the alkali extract had been refiltered. After displacing the alcohol with ether, the residue was dried at 110° C., weighed, ignited, and weighed again. The difference = the weight of crude fiber.

SUMMARY OF RESULTS OBTAINED BY VARIOUS ANALYSTS.

A summary of the results of analyses by various chemists is presented in the tables given below. These analyses were obtained, for the most part, from König's *Chemie der menschlichen Nahrungs- und Genussmittel*, 3 Aufl., Band I. The tables of analyses given by König are abridged here, by giving only the means, maxima, and minima of the results reported by each analyst on the same class of samples. It is believed that the tables, with appended notes, will be found self-explanatory.

As will be seen by reference to the tables, most analysts have reported the percentage of total nitrogenous matter, including theobromine. This figure is obtained by multiplying the per cent of total nitrogen by 6.25. This factor assumes the per cent of nitrogen in albuminous matter to be 16 per cent. Theobromine contains 31.1 per cent of nitrogen. The per cent of nitrogenous matter, including theobromine, can, therefore, be changed to the approximate per cent of nitrogenous matter, not including theobromine, by subtracting twice the percentage of theobromine from the number given as nitrogenous matter, including theobromine.

The percentages of theobromine, reported by Zipperer are considered too low by many authorities. This seems to be due to imperfect extraction of the theobromine by the method he used. (For the details of the method see page 953.)

The tables given on pages 967 and 968 will be found to contain interesting data concerning the quantitative relations of the constituents of the cocoa bean and its preparations. Benusemann also reported analyses of the ashes of the same samples, the results of which are given on page 969.

Analyses of cocoa beans made by various analysts.

Number.	Analyst.	Date.	Character of sample.	No. of analyses.	Water.	Nitrogenous matter.	Theo- mine.	Fat.	Starch.	Other non-nitrogenous substances.	Fiber.	Ash.	Sand.	Total nitrogen.	Husks.	Means.
1	Ch. Heisch ¹ .	1876	Husked.....	8	Per et. 4.01	Per et. 10.82	Per et.	Per et. 49.69	Per et.	Per et. 32.87	Per et.	Per et. 3.31	Per et.	Per et.	Per et. 12.6	Means.
					4.40	13.04		54.40		35.70		5.90			15.5	Maxima.
2	G. Laube and B. Aldendorff. ²	1879	do.....	8	3.72	7.31		45.30		26.33		2.35			8.5	Minima.
					3.25	14.76		48.95		13.31	3.68	3.65				Means.
					4.72	16.25		52.14		15.13	4.20	4.21				Maxima.
3	H. Weigmann ³ .	1884-'85	Raw, unhusked.....	7	2.28	13.19		46.18		10.82	8.82	3.07				Minima.
					7.93	14.19		45.57		22.92		3.99				Means.
					8.27	15.37		46.61		26.45		4.91				Maxima.
4	do.....	1883-'84	Roasted, unhusked.	7	7.53	13.50		1.31	44.62	19.40	4.30	3.16				Minima.
					6.79	14.13		46.19		24.10	4.63	3.87				Means.
					8.17	15.75		48.35		26.69		4.49				Maxima.
					5.26	13.56		45.06		21.64		3.33				Minima.
5	P. Zipperer ⁷ .	1884-'85	Raw, husked.	7	7.11			51.78		8.33		3.60				Means.
					8.40			53.66		11.07		4.32				Maxima.
					6.20			50.31		5.78		2.87				Minima.
6	Boussingault ⁹ .	1883	do.....	12	6.60	13.57		44.25				3.30				Means.
					11.60	14.87		53.30				4.00				Maxima.
					4.20	11.75		35.96				2.25				Minima.
7	H. Weigmann ³ .	1884-'85	Roasted, husked.....	7	5.58	14.13		50.09		8.77	13.91	3.93				Means.
					6.57	15.94		51.87		10.04	16.02	4.44				Maxima.
					4.73	13.31		48.01		7.17	12.25	3.16				Minima.
8	P. Zipperer ⁷ .	1884-'85	do.....	7	6.71			49.24		10.43		3.92				Means.
					8.52			52.09		12.64		4.82				Maxima.
					4.04			46.90		8.72		2.88				Minima.
9	G. Wolfram ¹² .	1878	Husked.....	6	(13)			51.68				3.63				Means.
								53.80				4.33				Maxima.
								49.90				3.02				Minima.

Analyses of the husks from cocoa beans reported by various chemists.

Number	Analyst.	Date.	No. of analyses.	Husks.	Water.	Nitrogenous substances.	Theobromine.	Fat.	Other nitrogenous substances.	Fiber.	Ash.	Sand.	Total nitrogen.
				Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
1	C. Portele ¹	1878	1	11.13	25.87 ³			8.22	34.15	13.35	7.28		
2	J. Moser ²	1878	1	11.46	10.32 ³			4.99	49.94	14.81	6.68	2.30	
3	L. Grandean ²	1878	4	12.01	10.23 ³			2.57	47.75	18.82	8.61		Means.
				14.40	12.31			3.22	62.27	24.24	11.85		Maxima.
				9.62	8.44			1.89	39.44	10.05	3.05		Minima.
4	G. Laube and B. Aldendorff ⁴	1879	8	16.34	7.93	14.41		6.38	43.69	14.69	7.12	7.67	Means.
				20.09	9.11	19.12		10.75	48.01	18.00	8.32	18.19	Maxima.
				14.04	6.40	11.68		4.23	35.29	12.79	6.06	0.21	Minima.
5	H. Weigmann ⁴	1884-'85	4	13.99	15.06 ^{3,4}		0.71	2.60	42.75	16.10	7.49 ⁶	1.95	Means.
				14.89	16.25		0.78	3.45	44.89	17.04	9.06	6.26	Maxima.
				12.49	13.18		0.58	2.01	40.30	15.25	6.19	0.27	Minima.
6	P. Zipperer ⁷	1884-'85	4	14.14	12.51	13.69 ³	0.34	4.27	39.11	16.72	10.50		Means.
				15.00	13.09	14.06	0.40	4.74	43.04	18.04	16.73		Maxima.
				12.28	11.90	13.31	0.30	4.00	35.17	14.85	7.31		Minima.
7	G. Wolfram ⁸	1878	6	Material dried at 100° C.			0.76				6.37		Means.
							1.11				13.32		Maxima.
							0.42				5.13		Minima.
8	Legler ⁹	1888	6							12.62			Mean.
										16.16			Maximum.
										10.23			Minimum.

⁶ Pure ash.⁷ Untersuchung ü. Cacao u. dessen Präparate, 1887; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.⁸ Jahresbericht d. k. chem. Centralstelle f. öff. Gesundheitspflege in Dresden, 1878; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.⁹ Rep. f. anal. Chem., 1884, 4, 345.¹ Centrbl. f. Agrikulturchem., 1870, 304; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.² Dietrich und König, Zusammensetzung der Futtermittel; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.³ Total nitrogen $\times 6.25$.⁴ König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.⁵ Weigmann reports the results of determinations of pure protein in these samples by Stutzer's method: Mean, 12.48 per cent; maximum, 13.00 per cent; minimum, 11.86 per cent.

Analyses of commercial preparations of cocoa beans reported by various analysts.

Number.	Analyst.	Description of sample.	Date.	No. of analyses.	Water.	Nitrog. sub- stances (total nitro- gen $\times$ 6.25).	Theo- bro- mine.	Fat.	Sugar.	Starch.	Other non- nitrog- enous sub- stances.	Fiber.	Ash.	Sand.	Total nitro- gen.	Means. Maxima. Minima.
1	H. Weigmann ¹	<i>Cocoa mass.</i>	1884-'85	7	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	
					4.16	13.97	1.56	53.03		21.81	21.81	3.40	3.46	0.17	2.23	
					5.03	15.81	1.75	55.81		24.53	24.53	3.60	3.87	0.34	2.53	
					3.10	13.25	1.25	49.86		19.85	19.85	2.92	2.88	0.07	2.12	
2	A. Stutzer ²	<i>Cocoa powders.</i>														
		Lobeck & Co.'s, Dresden:														
		Cocoa deprived of fat.....	1882	1	6.86	18.91		32.55	1.60	17.11	17.79	5.18			3.02	
3	do.....	do.....	1882	1	6.71	18.60		33.48					5.18		2.97	
		Gerb. Stollwerk's, Cologne:														
4	do.....	Cocoa deprived of fat, No. 1.	1882	1	6.55	20.23		30.95	2.13	15.20	21.16	3.78			3.24	
5	do.....	Cocoa deprived of fat, No. 2.	1882	1	6.50	20.29		32.31	2.53	13.56	19.44	5.37			3.25	

¹ König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.

² Rep. f. anal. Chem., 1882, 161; Hygiene-Bericht, 1882-'83, —, 217; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I. In addition to the results given above, Stutzer also reports the following:

	Sample No. 2.	Sample No. 3.	Sample No. 4.	Sample No. 5.
	Per cent.	Per cent.	Per cent.	Per cent.
Pure albumen.....	6.25	4.16	6.72	8.24
Soluble nitrogenous matter not albumen.....	5.78	5.54	6.36	5.46
Insoluble nitrogenous matter not albumen.....	6.88	8.90	7.15	6.58
Phosphoric acid (P ₂ O ₅).....	1.61	1.67	1.79	1.95



Analyses of commercial preparations of cocoa beans reported by various analysts—Continued.

Number.	Analyst.	Description of sample.	Date.	No. of analyses.	Water.	Nitrogenous substances (total nitrogen $\times 6.25$).	Theobromine.	Fat.	Sugar.	Starch.	Other non-nitrogenous substances.	Fiber.	Ash.	Sand.	Total nitrogen.
					Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
6	J. König, J. Cosack, and H. Weigmann. ³	<i>Cocoa powder</i> —Continued. Gerb. Stollwerck's, Cologne—Continued. 4 marks per half kilo	1884	1	6.81	22.50	2.12	21.95	2.77	15.20	20.71	4.64	5.22	-----	3.60
7	do	2.40 marks per half kilo	1884	1	6.67	23.62	2.25	23.31	3.62	14.26	18.72	4.76	5.04	-----	3.78
8	do		1888	1	5.10	22.31	-----	21.81	-----	17.34	22.55	5.42	5.47	-----	3.57
9	do	Lobeck & Co.'s, Dresden	1888	1	7.62	19.81	-----	26.23	-----	13.30	22.79	4.67	5.58	-----	3.17
10	do	P. W. Gaedke's, Hamburg	1888	1	7.10	25.18	-----	21.68	-----	13.84	20.45	6.05	5.70	-----	4.03
11	do	do	1888	1	6.49	21.94	1.64	28.07	-----	16.82	14.63	6.68	5.37	-----	3.51
12	do	C. S. Van Houten & Zoon's, Amsterdam.	1888	1	5.42	18.97	-----	29.27	-----	13.38	20.24	4.88	7.84	-----	3.17
13	A. Stutzer. ^{4,5}	do	1886	1	4.27	20.50	0.95	32.30	-----	11.85	13.18	8.78	9.12	-----	3.28
14	do	P. W. Gaedke's, Hamburg	1886	1	3.81	23.12	1.28	28.45	-----	15.08	18.43	5.85	5.26	-----	3.70
15	Frühling & Schultz. ^{4,5}	Holland cocoa powder	1880	1	4.60	19.50	-----	31.60	-----	-----	-----	-----	9.10	-----	3.12
16	Belohoubek. ^{4,5}	Van Houten's	1888	1	3.87	14.47	1.74	33.27	-----	34.80	-----	3.89	7.88	-----	-----
17	E. Haselhoff. ^{4,5}	"Nahr-salz-cacao," Howel & Veithen, Cologne.	1889	1	8.00	17.50	1.78	28.26	-----	11.09	26.24	4.21	4.70	-----	2.80
18	(See note). ⁷	Acorn cocoas	1885-'88	6	5.33	13.51	-----	14.82	26.91	-----	30.16	2.91	3.64	-----	Means. 2.16
					7.50	13.25	-----	17.33	39.70	-----	32.78	3.82	3.95	-----	Maxima. 2.44
19	J. König and M. Wesener. ⁵	Saccharine cocoa	1882	1	4.01	11.25	-----	11.68	19.10	-----	22.66	1.88	3.34	-----	Minima. 1.80
		<i>Chocolates.</i>			7.26	20.50	2.09	32.25	(⁶)	13.02	13.51	5.27	5.93	-----	-----
20	J. König, C. Krauch, J. Cosack and H. Weigmann. ⁶	Sweet chocolate	1879	1	2.81	5.56	-----	17.57	54.80	-----	-----	-----	2.98	-----	0.89

21	do.....	1879	1	0.99	4.87		12.03	64.96	4.10		2.18		0.78
22	do.....	1879	1	1.31	4.94		15.52	65.64	3.96	5.51	1.22	1.90	0.79
23	do.....	1879	1	1.09	4.87		16.09	69.84	3.69	1.77	1.10	1.55	0.78
24	Gerb. Stollwerck's, Cologne: 2.40 marks per one-half kilo.	1883	1	2.50	6.62	0.66	27.31	48.59	4.59	5.40	1.30	1.69	1.06
25	do.....	1882	1	2.68	6.81	0.49	21.73	50.65	10.99	3.84	1.87	1.43	1.09

¹ König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.

² Broschüre von P. W. Gaedtke über Hamburger Puder-Cacao; König, Chem. d. menschl. Nahr.- u. Genuss., 3 Aufl., Bd. I.

³ The following additional data are reported for samples Nos. 8 to 14:

Sample No.	Nitrogenous substances.		Per cent of total nitrogen in form of digestible substances.	Substances soluble in water.		K ₂ O.	CaO.	MgO.	P ₂ O ₅ .
	Digestible.	Indigestible.		Organic.	Inorganic.				
									Per cent.
8	12.76	9.55	57.19	4.88	2.04	1.58	0.35	0.93	2.05
9	10.56	8.25	58.35	4.03	1.93	1.83	0.50	0.86	1.92
10	14.06	11.12	55.84	4.34	1.88	1.75	0.50	1.01	2.09
11						1.91	0.28	0.92	1.88
12	8.54	10.43	45.02	4.03	3.03	3.52	0.27	0.81	1.84
Pure albumen.			Of total nitrogen in pure albumen, per cent digestible.						
13	9.18	9.47	49.34				0.16		1.77
14	13.13	7.50	63.64				0.20		1.82

⁴ Correspondenz-Bl. d. Vereins analyt. Chem., 1880, 17; König, Chemie der Nahrungs- und Genussmittel, 3 Aufl., Band I.

⁵ Rep. d. Chem. Ztg., 1888, 270; Böhm. pharm. Ztschr., 7, 311; Chem. Centralbl., 1890, 131; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.

⁶ König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.

⁷ *Op. cit.*, note No. 8, page 938. A summary of analyses by Fresenius, Tschirch, Weigmann, Schwedissinger and Van Hamel Roos. Tannin, as acorn tannin, is also reported; mean 2.42 per cent, maximum 3.23 per cent, minimum 1.95 per cent.

⁸ For ash analyses of these samples, see table on page 969.

⁹ 0.40 per cent saccharine.

¹⁰ Contained 10 per cent of sago starch.

Analyses of commercial preparations of cocoa beans reported by various analysts—Continued.

Number.	Analyst.	Description of sample.	Date.	No. of analyses.	Water.	Nitrog- ous sub- stances (total nitro- gen $\times$ 6.25).	Theo- bro- mine.	Fat.	Sugar.	Starch.	Other non- nitrog- ous sub- stances.	Ether.	Ash.	Sand.	Total nitro- gen.
					Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
26	J. König, C. Krauch, J. Cosack, and H. Weigmann. ¹	5.00 marks per one-half kilo.	1883	1	2.06	6.89	0.79	28.55	37.86	5.85	14.68	2.10	2.01	-----	1.10
27	do.....	3.00 marks per one-half kilo.	1883	1	2.11	6.75	0.68	25.54	45.37	5.83	11.25	1.50	1.65	-----	1.68
28	do.....	do.....	1883	1	2.19	6.93	0.69	24.10	47.29	3.83	12.48	1.50	1.68	-----	1.11
29	do.....	1.60 marks per one-half kilo.	1883	1	1.93	8.18	0.56	22.50	55.31	4.44	5.50	0.70	1.44	-----	1.31
30	E. Haselhoff ¹	"Nährsalz-chocolate," Hewel & Veithen, Cologne.	1889	1	1.88	5.81	0.80	24.12	45.67	6.49	12.14	2.05	1.84	-----	0.93
31	Boussingault ²	French chocolate	1883	1	1.22	4.57	1.26	21.40	59.07	1.83	-----	-----	1.79	-----	-----
32	do.....	do.....	1883	1	1.28	4.57	1.33	22.29	57.47	1.83	-----	-----	1.75	-----	-----
33	do.....	do.....	1883	1	0.98	4.99	1.43	23.80	56.34	0.97	-----	-----	1.87	-----	-----
34	do.....	Spanish chocolate	1883	1	1.51	6.45	1.82	20.50	54.00	1.33	-----	-----	2.43	-----	-----
35	do.....	do.....	1883	1	1.20	8.67	2.64	24.80	41.46	1.84	-----	-----	3.23	-----	-----
36	do.....	do.....	1883	1	1.33	8.21	2.50	26.60	41.40	1.74	-----	-----	3.06	-----	-----

¹ König, *Chemie der menschlichen Nahrungs- und Genussmittel*, 3 Aufl., Band I.² Ann. Chim. d. Phys., 1883, 443; Chem. Ztg., 1883, 204. König, *Chemie der menschlichen Nahrungs- und Genussmittel*, 3 Aufl., Band I.

Analyses of cocoa beans, cocoa husks, and chocolates, reported by R. Bensemann.¹

All dryings were made at 100° C.										
	Moisture at 100° C.	Total organic matter insoluble in water = U.				Organic bodies soluble in water.	Ash, dried at 100° C.	Totals.	Ash of water in- soluble bodies.	Husks in air- dried beans.
		Fat = F.	Starch = S.	Other or- ganic bod- ies in sol- uble in water.	Per cent.					
Air-dried husked beans:	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Maracaibo	6.87	49.18	13.01	17.32	9.20	4.42	100.00	0.84		
Caracas	7.03	49.43	12.74	18.53	8.26	4.01	100.00	0.90		
Trinidad	6.45	51.97	10.15	19.25	8.80	3.38	100.00	0.63		
Machala—Guayaquil	5.81	53.21	10.82	19.38	6.94	3.84	100.00	1.00		
Porto Plata	5.87	53.57	12.04	15.69	9.52	3.31	100.00	1.09		
Means	6.41	51.47	11.75	18.03	8.54	3.80	100.00	0.89		
Air-dried husks:										
Maracaibo	13.08	2.34	8.79	54.43	14.45	26.91	100.00	22.63		12.00
Caracas	13.62	1.81	8.81	48.96	9.74	37.06	100.00	33.60		16.00
Trinidad	13.80	2.37	8.63	48.32	18.91	47.97	100.00	33.80		14.00
Machala—Guayaquil	14.56	2.03	7.07	48.51	14.73	43.11	100.00	41.20		13.00
Porto Plata	11.55	3.95	10.35	45.58	15.53	33.04	100.00	30.21		12.00
Means	13.32	2.50	8.73	49.16	14.67	11.62	100.00	7.83		13.00
Chocolates in cakes, German manufacture, and composed of the husked bean and sugar only:										
2.40 marks per one-half kilo	1.92	22.61	5.20	8.35	59.60	2.32	100.00	0.27		
2.00 marks per one-half kilo	2.25	22.50	4.70	8.82	59.56	2.42	100.00	0.24		
1.60 marks per one-half kilo	1.10	22.48	4.27	8.63	61.81	1.71	100.00	0.34		
1.20 marks per one-half kilo	1.53	21.40	3.92	9.02	62.43	1.70	100.00	0.25		
1.00 marks per one-half kilo	1.43	24.14	4.81	8.09	59.73	1.80	100.00	0.40		
Means	1.65	22.57	4.58	8.58	60.63	1.99	100.00	0.30		

¹ Grayish brown.² Gray.¹ Rep. f. anal. Chem., 1884, 213 and 1885, 178; König, Chemie der menschlichen Nahrungs- und Genussmittel, 3 Aufl., Band I.² White.

Table showing the quantitative relations between the constituents of the cocoa bean (calculated by Bensemann from the analyses given in the table on page 967).

[S = starch, F = fat, U = total organic matter insoluble in water.]

	S U-F.	F U-S.	S U.	F U.	S F.
Air-dried, husked beans:					
Maracaibo	0.4289	0.7395	0.1636	0.6185	0.2645
Caracas	0.4074	0.7273	0.1578	0.6125	0.2577
Trinidad	0.3452	0.7297	0.1247	0.6387	0.1953
Machala-Guayaquil	0.3583	0.7330	0.1297	0.6379	0.2033
Portoplata	0.3660	0.7734	0.1481	0.6589	0.2247
Means	0.3946	0.7406	0.1446	0.6335	0.2283
Air-dried husks:					
Maracaibo	0.1390	0.0412	0.1341	0.0357	3.7564
Caracas	0.1525	0.0356	0.1479	0.0304	4.8674
Trinidad	0.1515	0.0467	0.1455	0.0399	3.6413
Machala-Guayaquil	0.1272	0.0402	0.1227	0.0352	3.4827
Portoplata	0.1850	0.0797	0.1728	0.0660	2.6202
Means	0.1508	0.0484	0.1446	0.0414	3.4920
Chocolate in cakes, German manufacture, and composed of the husked bean and sugar only:					
2.40 marks per one-half kilo	0.3838	0.7303	0.1438	0.6253	0.2300
2.00 marks per one-half kilo	0.3476	0.7161	0.1314	0.6220	0.2112
1.60 marks per one-half kilo	0.3310	0.7226	0.1207	0.6354	0.1899
1.20 marks per one-half kilo	0.3029	0.7035	0.1141	0.6232	0.1831
1.00 marks per one-half kilo	0.3729	0.7490	0.1298	0.6517	0.1992
Means	0.3480	0.7245	0.1282	0.6317	0.2029

Ash analyses reported by various chemists.

No.	Analyst.	Description of sample.	In the crude ash.		Pure ash.	K ₂ O.	Na ₂ O.	CaO.	MgO.	Fe ₂ O ₃ .	Al ₂ O ₃ .	P ₂ O ₅ .	SO ₃ .	SiO ₂ .	Cl.	CO ₂ .	H ₂ O.	O equiv- alent to Cl.
			Crude ash.	Sand and carbon.														
					Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
1	Zedeler ¹	Cocoa beans.....				37.25	1.23	2.90	16.02	0.09		39.73	1.53		1.68			
2	Letellier ²	do.....		1.00		33.74		11.11	17.17			29.90	4.55	3.33	0.20			
3	Rost v. Ton- ningen. ³	do.....	3.87	17.38	3.14	36.69	5.54	2.32				46.21	5.20	1.20	0.54			
4	Belohoubeck ⁴	Van Houten's cocoa.....	7.88			52.89	2.14	1.56	10.45	0.23	0.15	24.91	2.56	0.75	0.89	3.45		
5	Frühling and Schultz. ⁵	Holland cocoa.....	9.10			48.68	1.23	2.08	8.43			20.43	1.76		1.86	15.32		
Insoluble in dilute HCl or HNO ₃ .																		
Portion soluble in dilute HCl or HNO ₃ .																		
			Combustible matter.	Incombustible matter left on ignition.														
6	Bensemann ⁶ ..	Husked beans (ash dried at 100° C.).				35.839	0.515	4.118	15.750	0.182	0.080	27.741	2.632	0.214	0.295	10.349	1.847	0.066
7		Maracabo.....	0.142		0.312	33.844	0.766	5.030	15.151	0.217	0.326	29.302	2.740	0.211	0.341	8.435	1.975	0.077
8		Caracas.....	0.076		1.663	30.845	1.964	4.638	16.060	0.491	0.490	28.624	3.957	0.169	0.427	8.953	2.781	0.096
9		Trinidad.....	0.144		0.553	30.686	4.173	3.112	16.172	0.629	0.432	37.006	2.042	0.134	0.279	2.788	1.912	0.063
10		Machala-Guayaquil	0.074		0.630	29.989	3.427	2.923	17.562	0.303	0.305	35.274	3.952	0.240	0.085	3.481	1.205	0.019
		Portoplata.....	0.198		1.075													
		Means (of 6-10) ..	0.127		0.846	32.351	2.169	3.964	16.139	0.364	0.327	31.588	3.065	0.194	0.285	6.801	1.944	0.064
Husks (ash dried at 100° C.):																		
11		Maracabo.....	0.113		1.917	31.517	4.188	10.134	9.546	0.647	0.281	9.068	3.041	1.180	1.005	25.454	2.135	0.226
12		Caracas.....	0.421		47.711	11.812	3.298	4.458	4.703	0.931	1.554	7.630	1.478	7.975	0.220	5.399	2.458	0.049
13		Trinidad.....	0.979		29.215	25.866	2.726	5.097	5.206	0.339	0.710	4.703	3.398	2.416	1.022	16.290	2.263	0.230
14		Machala-Guayaquil	0.306		37.662	23.117	1.210	3.503	4.837	0.958	1.854	7.288	1.741	4.321	0.255	11.834	1.171	0.057
15		Portoplata.....	1.247		51.513	12.174	2.780	4.401	4.090	0.462	1.046	7.242	2.012	6.780	0.444	4.247	1.662	0.100

¹ Liebig u. Kopp, Jahresb., 1851; Wolff, Aschen-Analysen, Berlin, 1871.² Boussingault's Landwirthschaft, 2. Aufl. der Grägrer sehen Uebersetzung, Bd. 3, 4; Wolff, Aschen-Analysen, Berlin, 1871.³ Kopp u. Will, Jahresb., 1860, 549; Wolff, Aschen-Analysen, Berlin, 1871.⁴ Böhm, pharm. Zeitsch., 7, 311; Chem. Centralbl., 1890, 131; Rep. d. Chem. Ztg., 1888, 270; König, Chemie der mensch. Nahr. u. Genussmittel, 3. Aufl., Bd. I, S. 1027.⁵ Correspondenz-Bi. d. Vereins anal. Chem., 1880, 17; König, Chem. d. mensch. Nahr. u. Genussmittel, 3. Aufl., Bd. I, S. 1027.⁶ Rep. f. anal. Chem., 1885, 178.

RESULTS OF THE EXAMINATION OF COCOA PREPARATIONS IN THE
LABORATORY OF THE U. S. DEPARTMENT OF AGRICULTURE.

As cocoa preparations are always sold in the original packages of the manufacturer, the purchase of samples does not need to be conducted with the care and precautions that are necessary in the case of many other food materials. The samples examined were all purchased in Washington, D. C., and represented very nearly all the brands obtainable at the time the purchases were made.

In accordance with the law that provides for these investigations, a full description of each sample is given in the table on page 971. Each description is accompanied by the laboratory index number and a consecutive number for use in reference to the table of results of analyses that is given on page 980.

In the column headed "Weight of package and cost" the size and nature of the package and the purchase price paid in the retail market are given. The price per pound as given in the next column is calculated from these data. In a few cases where the weight was not given on the package the price per pound is only approximate and is placed in parenthesis with an interrogation mark.

The rather extended quotation of the manufacturer's descriptions of the samples is necessary to do justice to the manufacturers of both adulterated and unadulterated preparations. In several cases the addition of material that should otherwise be considered adulterants is duly acknowledged on the package; in other cases no mention of the fact is made or the statement implies that the sample is pure.

The variety of preparations offered to American consumers is certainly very great; so great, in fact, that a satisfactory classification of them is hardly possible. In the following tables no classification was attempted, further than a separation of plain and sweet chocolates into groups by themselves:

SAMPLES OF COCOA PREPARATIONS.

971

Serial number.	Consecutive number.	Date of purchase.	Retail dealer from whom purchased—all Washington, D. C., N.W.	Where and by whom manufactured.	Label on package, together with statement regarding ingredients, etc.	Weight of package and cost.	Price per pound.	Serial number.	Consecutive number.
6876	1	June 20, 1890	Burchard, corner Four and a Half street and Pennsylvania avenue.	Walter Baker & Co., Dorchester, Mass.	Bakers' Chocolate.....	$\frac{1}{2}$ pound in paper, 20c.	\$0.40	6876	1
6877	2	do	do	H. O. Wilbur & Sons, Philadelphia, Pa.	Wilbur's Chocolate.....	do	0.40	6877	2
8891	3	May 22, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	H. Maillard, 1097 Broadway, N. Y.	Maillard's Chocolate.....	1 pound in paper, 40c.	0.40	8891	3
8903	4	do	do	Rockwood & Co., New York ..	Rockwood & Co.'s Chocolate ..	do	0.40	8903	4
8916	5	do	G. B. Kennedy & Sons, 1209 F street.	Chocolate Works, corner Eighth street and Irving Place, New York.	Huyler's Family Chocolate ..	$\frac{1}{2}$ pound in paper, 20c.	0.40	8916	5
8992	6	June 26, 1891	Great Atlantic and Pacific Tea Company, Center Market.	Runkel Brothers, New York..	Runkel Brothers' Premium Chocolate. "A superior article of chocolate, made of the purest cocoa bean, of the finest quality, without sugar, making this a pure and wholesome preparation of chocolate."	$\frac{1}{2}$ pound in paper, 18c.	0.35	8992	6
8912	7	May 22, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	Stephen F. Whitman & Son, Philadelphia, Pa.	Whitman's Instantaneous Chocolate.....	$\frac{1}{2}$ pound in tin, 45c....	0.90	8912	7
8920	8	do	G. E. Kennedy & Sons, 1209 F street.	E. G. Whitman, 812 Chestnut street, Philadelphia, Pa.	Whitman's Powdered Chocolate	$\frac{1}{2}$ pound in tin, 40c ..	0.80	8920	8
8930	9	do	E. L. Yewell, 1141 Ninth street.	Schilling, Stollwerck & Co., Cologne.	Stollwerck's Princess Chocolate	$\frac{1}{2}$ pound in paper, 25c.	0.50	8930	9
8986	10	June 26, 1891	C. C. Bryan, 1413 New York avenue.	H. I. Rowntree & Co., York, England.	Rowntree's Queen Chocolate, in powder	$\frac{1}{2}$ pound in tin, 30c ..	0.60	8986	10
8888	11	May 22, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	W. Baker & Co., Dorchester, Mass.	Baker's Vanilla Chocolate	$\frac{1}{2}$ pound in paper, 38c.	0.75	8888	11

Description of samples of cocoa preparations examined in the laboratory of the U. S. Department of Agriculture—Continued.

Serial number.	Consecutive number.	Date of purchase.	Retail dealer from whom purchased—all Washington, D. C., NW.	Where and by whom manufactured.	Label on package, together with statement regarding ingredients, etc.	Weight of package and cost.	Price per pound.	Serial number.	Consecutive number.
8892	12	May 22, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	H. Maillard, 1097 Broadway, New York.	Maillard's Vanilla Chocolate.....	$\frac{1}{2}$ pound in paper, 25c.	0.50	8892	12
8893	13	do	S. R. Waters, 1342 Seventh street.	do	Caracas Chocolate, vanilla flavored.....	$\frac{1}{2}$ pound in paper, 10c.	0.40	8893	13
8900	14	June 20, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	do	Double Vanilla Chocolate.....	1 pound in paper, 60c.	0.60	8900	14
8901	15	do	do	do	Triple Vanilla Chocolate.....	1 pound in paper, 75c.	0.75	8901	15
8907	16	May 22, 1891	do	Menier, Noisiel, France.....	Chocolate-Menier, fine vanilla quality.....	$\frac{1}{2}$ pound in paper, 30c.	0.60	8907	16
8919	17	do	G. E. Kennedy & Sons, 1209 F street.	Hawley & Hoops, New York.....	Peerless Vanilla Chocolate.....	$\frac{1}{2}$ jar, 50c.....		8919	17
8921	18	do	J. F. Page, 1210 F street.....	Fullie & Co., Caracas, Venezuela.	La India Vanilla-Primera Chocolate.....	$\frac{1}{2}$ pound in paper, 50c.	1.00	8921	18
8924	19	do	S. B. Taylor, 1610 Seventh street.	(No name given on package).....	Chocolate a la Vanille.....	$\frac{1}{2}$ pound in paper, 10c.	0.40	8924	19
8914	20	do	S. I. Bradley, 1315 Fourteenth street.	Benadorf & Co., Amsterdam, Holland.	Zoete Vanille Chocolate.....	($\frac{1}{4}$ $\frac{1}{2}$) pound in paper, 10c.	(0.40)	8914	20
8894	21	do	S. R. Waters, 1342 Seventh street.	H. Maillard, 1097 Broadway, New York.	Maillard's Sweet Chocolate.....	$\frac{1}{2}$ pound in paper, 6c.	0.24	8894	21
8895	22	do	John S. Cissel, 1014 Seventh street.	do	Panama Sweet Chocolate.....	$\frac{1}{2}$ pound in paper, 10c.	0.50	8895	22
8901	23	do	P. R. Wilson, corner Seventh and K streets.	J. S. Fry & Sons, Bristol and London, England.	Fry's Diamond Sweet Chocolate.....	$\frac{1}{2}$ pound in paper, 10c.	0.40	8901	23
8902	24	do	A. O. Wright, 1032 Fourteenth street.	do	Fry's Sweet Chocolate.....	do	0.40	8902	24
8904	25	do	H. H. Elliott, 1520 Fourteenth street.	Rockwood & Co., New York.....	Rockwood & Co.'s Sweet Chocolate.....	$\frac{1}{2}$ pound in paper, 10c.	0.50	8904	25
8917	26	do	G. E. Kennedy & Sons, 1209 F street.	S. German, Dorechester, Mass.	German Sweet Chocolate.....	$\frac{1}{2}$ pound in paper, 10c.	0.40	8917	26

8918	27	do	(No name)	Mexican Sweet Chocolate.	do	0.40	8918
8922	28	do	H. O. Wilbur & Sons, Philadelphia, Pa.	Paris Sweet Chocolate	($\frac{1}{4}$ t) pound in paper, 10c.	(0.40)	8922
8923	29	do	Runkel Bros., New York	Vienna Sweet Chocolate	$\frac{1}{4}$ pound in paper, 6c.	0.24	8923
8925	30	do	(No name)	French Sweet Chocolate	$\frac{1}{4}$ pound in paper, 8c.	0.32	8925
8926	31	do	Royal Cocoa and Chocolate Company, Philadelphia, Pa.	Gerhard Schmitz's Sweet Spiced Zealand Chocolate.	1 pound in paper, 50c.	0.50	8926
8928	32	do	———, southeast corner Ninth and Q streets.	Crown Sweet Chocolate	do	do	do
8929	33	do	D. Mazzochi, 1504 Fourteenth street.	Broadway Sweet Chocolate	($\frac{1}{4}$ t) pound in paper, 10c.	(0.40)	8929
8993	34	do	J. W. Ruppert & Bro., 711 Seventh street.	Wilbur's Vanilla Sweet Clover Chocolate	3 packages, 30c.	(0.50 t)	8993
8994	35	do	Win. R. Brown, corner Pennsylvania avenue and Twentieth streets.	Rockwood & Co.'s Dutch Sweet Chocolate	4 packages, 20c.	(0.40 t)	8994
8995	36	do	Philip H. Ward, corner Pennsylvania avenue and Twenty-first streets.	Baker's Cocoa	2 packages, 16c.	(0.32 t)	8995
6881	37	June 20, 1890	Tolson, 701 Seventh street	Breakfast Cocoa. "This extract of cocoa, by a peculiar process in its manufacture which preserves the theobromine and nutritive portion, is rendered treble the strength of cocoa as usually prepared. It yields a delicious flavor and is an easily digested and thoroughly wholesome cocoa."	$\frac{1}{4}$ pound in paper, 25c.	0.50	6881
6899	38	do	E. Youngs, Ninth street		$\frac{1}{4}$ pound in tin, 25c.	0.50	6899

Description of samples of cocoa preparations examined in the laboratory of the U. S. Department of Agriculture—Continued.

Serial number.	Consecutive number.	Date of purchase.	Retail dealer from whom purchased—all Washington, D. C., N.W.	Where and by whom manufactured.	Label on package, together with statement regarding ingredients, etc.	Weight of package and cost.	Price per pound.	Serial number.	Consecutive number.
6899	38	June 20, 1890	E. Youngs, Ninth street	Walter Baker & Co., Dorchester, Mass.	"There are very few simple articles of food which possess so many valuable and important dietary properties as cocoa. While acting on the nerves as a gentle stimulant, it provides the body with some of the purest elements of nutrition, and at the same time corrects and invigorates the action of the digestive organs." "Medical men of all shades of opinion have agreed in recommending it as the safest and most beneficial article of diet for persons of weak constitutions." "The excess of oil having been removed, no sensation of heaviness follows the use of this preparation."				
6891	39	June 21, 1890	do	H. O. Wilbur & Sons, Philadelphia, Pa.	Breakfast cocoa: "Wilbur's cocoa is manufactured from the choicest cocoa bean, with the excess of oil removed." "Van Houten's Pure Soluble Cocoa is not raw cocoa, but a greatly improved and highly concentrated cocoa, in which the proportion of natural fat is properly adjusted, and in which important constituents are rendered more soluble and useful, while the flavor and aroma are most highly developed."	1 pound, in tin, 25c.	\$0.50	6891	39
6893	40	do	G. G. Cornwell & Son, Pennsylvania avenue.	C. J. Van Houten & Zoon, Weesp, Holland.		do	0.50	6893	40

6896	41	do	Great Atlantic and Pacific Tea Co.	Sole agents, H. O. Wilbur & Sons, Philadelphia, Pa.	London Cocoa: "Caracas and other cocoas, scientifically blended with sugar and arrowroot. It is guaranteed free from other substances, and being deprived of all indigestible matter, by a process peculiar to this manufactory, is a perfect nutrient for the sick, aged, and children."	do	0.50	6896	41
6901	42	do	Metzger's, Seventh street	H. I. Rowntree & Co., York, England.	Rowntree's elect extract of cocoa	$\frac{1}{2}$ pound, in tin, 25c.	1.00	6901	42
8889	43	May 22, 1891	N. W. Burchell, 1325 F street.	Walter Baker & Co., Dorchester, Mass.	Walter Baker & Co.'s Premium Cracked Cocoa.	$\frac{1}{2}$ pound, paper, 20c.	0.40	8889	43
8896	44	do	G. G. Cornwell & Son, Pennsylvania avenue.	H. Maillard, 1097 Broadway, N. Y.	Maillard's Breakfast Cocoa	$\frac{1}{2}$ pound, in tin, 35c.	0.70	8896	44
8897	45	do	do	do	Maillard's Dietetic Cocoa: "This cocoa is made on the homeopathic principle by extracting a large percentage of the oil or butter, and is especially adapted for the use of invalids and persons whose condition requires such nourishment as will not interfere with digestion."	$\frac{1}{2}$ pound, in tin, 30c.	0.60	8897	45
8899	46	do	R. A. Walker, corner Q and Seventh streets.	J. S. Fry & Sons, Bristol and London, England.	Fry's Cocoa Extract: "We guarantee the absolute genuineness of this pure and delicious cocoa, which consists of choice cocoa only, from which the superfluous oil has been extracted."	do	0.60	8899	46
8900	47	do	G. C. Bryan, 1413 New York avenue.	do	Fry's Malted Cocoa. "A combination of Fry's pure cocoa extract (cocoa deprived of the superfluous oil) with Allen & Hanbury's Extract of Malt."	$\frac{1}{2}$ pound in tin, 40c.	0.80	8900	47
8905	48	do	J. F. Page, 1210 F street.....	Rockwood & Co., New York..	Rockwood & Co.'s Breakfast Cocoa.....	$\frac{1}{2}$ pound in tin, 15c.	0.60	8905	48
8906	49	do	J. W. Hardell, corner 9th and P streets.	do	Rockwood & Co.'s Table Cocoa.....	$\frac{1}{2}$ pound in tin, 20c.	0.40	8906	49

Description of samples of cocoa preparations examined in the laboratory of the U. S. Department of Agriculture—Continued.

Serial num-ber.	Consecutive number.	Date of purchase.	Retail dealer from whom purchased—all Washington, D. C., NW.	Where and by whom manufactured.	Label on package, together with statement regarding ingredients, etc.	Weight of package and cost.	Price per pound.	Serial num-ber.	Consecutive number.
8908	50	May 22, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	The Chas. H. Phillips Chemical Co., New York.	Phillips' Digestible Cocoa. "Cocoas and chocolates, notwithstanding their recognized nutritious properties, can not be used by many, because of the difficulty attending their digestion, on account of the large proportion of fat which they contain; and many manufacturers, seeking to overcome this difficulty, express and remove this fat (cocoa butter) in the preparation of the cocoa from the bean, and furnish that which remains (or the fiber) as a beverage. The fat of the cocoa, however, is one of the most vitalizing principles, and its removal in any degree manifestly impairs the value of the cocoa as a nourishing drink. In this preparation the fat is wholly retained, and emulsified—or reduced to a condition for easy assimilation—by means of pancreatine (nature's digester of fats). The pancreatine (our own manufacture) is intimately combined with the cocoa—acts while it is being made up for the cup—and the fat is so completely digested by it that a continued use of this preparation will not distress the most sensitive stomach, nor cause headaches; and no grease will be seen coming to the surface, as with the ordinary cocoas or chocolates. All the nutriment of	$\frac{1}{4}$ pound in tin, 40c ..	0.80	8908	50

8909	51	do.....	J. and C. Bloeker, Amsterdam, Holland.	the richest cocoa may therefore be had by the most delicate and without disagreement. A small proportion of the soluble phosphates as found in wheat is added, as supplying increased nutriment and aiding the digestion. The phosphates meet particularly well in their combination with cocoa conditions of debility and waste, and, besides imparting their well-known tonic properties, add a very grateful delicacy of flavor."	1. 10	8909	51
8910	52	do.....	James Epps & Co., London, England.	Bloeker's Dutch Cocoa. "For a long time, however, our greatest chemists have in vain endeavored to separate the indigestible properties of the cocoa bean from its nutritious parts. It remained for us after years of careful research to invent a practical method of effecting this separation on a large scale." Prepared Cocoa. "Cocoa contains a bland oil, which is preëminent as a vitalizing substance, and to remove which oil in any degree is to lessen its value as a forcè producer. It is to preserve this oil, when using cocoa as a beverage, that it becomes necessary that the imported seeds from Trinidad and Caracas should be prepared, and scientifically; for to render the oil soluble and easy of digestion, it needs to be combined with just so much loaf sugar and West India arrowroot as will effect its perfect incorporation. In the preparation of the cocoa contained in this tin we guarantee that no other ingredients than those mentioned are used."	0. 50	8910	52

Description of samples of cocoa preparations examined in the laboratory of the U. S. Department of Agriculture—Continued.

Serial number.	Consecutive number.	Date of purchase.	Retail dealer from whom purchased—all Washington, D. C., N.W.	Where and by whom manufactured.	Label on package, together with statement regarding ingredients, etc.	Weight of package and cost.	Price per pound.	Serial number.	Consecutive number.
8913	53	May 22, 1891	G. G. Cornwell & Son, Pennsylvania avenue.	Bensdorp & Co., Amsterdam, Holland.	Bensdorp's Pure Soluble Cocoa.....	$\frac{1}{2}$ pound in tin, 40c....	0. 80	8913	53
8927	54	do	J. S. Clisel, 1014 9th street.....	C. D. Brooks, Dedham, Mass....	Brook's Prepared Cocoa	$\frac{1}{2}$ pound in tin, 25c....	0. 50	8927	54
8932	55	do	Huyler's, Pennsylvania avenue	Huyler, 18th street and Irving Place, New York.	Huyler's Cocoa	$\frac{1}{2}$ pound in tin, 25c....	0. 50	8932	55
6894	56	June 21, 1890	C. C. Bryan, 1413 New York avenue.	Walter Baker & Co., Dorchester, Mass.	Walter Baker & Co.'s Broma. "Broma is a combination of the cocoanut with other ingredients, invigorating and agreeable, both to invalids and to persons in health."	$\frac{1}{2}$ pound in tin, 25c....	0. 50	6894	56
8998	57	May 22, 1891	do	H. Maillard, 1097 Broadway, New York.	Henry Maillard's Broma. "Broma is chiefly composed of the nutritious principle derived from the cocoa bean, by eliminating the butyrons matter."	$\frac{1}{2}$ pound in tin, 20c....	0. 40	8998	57
6892	58	June 22, 1890	E. Youngs, 9th street	H. O. Wilbur & Sons, Philadelphia, Pa.	Wilbur's Cocoa-theta. "Invigorating, antidyseptic, powdered chocolate, warranted strictly pure, * * * affording all the nutritive properties of the cocoa bean, while eliminating the troublesome qualities."	$\frac{1}{2}$ pound in tin, 25c....	0. 50	6892	58
8890	59	May 22, 1891	G. E. Kennedy & Sons, 1209 F street.	Walter Baker & Co., Dorchester, Mass.	Rachout des Arabes	Bottle, 55c.....		8890	59
8911	60	do	G. G. Cornwell & Son, Pennsylvania avenue.	H. Schweitzer & Co., London, England.	Schweitzer's Cocoatina. "Antidyspeptic cocoa or chocolate powder. This unique preparation is the highest class of soluble cocoa or chocolate, being absolutely 'all cocoa,' with the excess of fat extracted."	$\frac{1}{2}$ pound in tin, 45c....	1. 80	8911	60

8915	61	do	N. W. Burchell, 1225 F street..	Smith's Manufacturing Co., New York.	Alkethrepta. "A pure preparation of chocolate, * * * retaining the highly nutritious natural butter of the cocoa, * which is so treated as not to derange the stomach of the most delicate."	$\frac{1}{2}$ pound in tin, 30c....	0. 60	8915	61
8985	62	June 26, 1891	C. C. Bryan, 1413 New York avenue.	M. G. De Langrenier, Paris, France.	Racahout des Arabes	Bottle, \$1.10		8985	62
8989	63	do	R. L. Main, 1527 14th street....	Hawley & Hoops, New York.	Hawley and Hoops's Breakfast Cocoa	$\frac{1}{2}$ pound in tin, 25c....	0. 50	8989	63
10435	64	April, 1892	Physician's sample. From the Cibils Co., of the U. S., Bos- ton, Mass.	Imported and guaranteed by E. D. Lowe, Boston, Mass.	"Windmill brand." "De Jong's pure solu- ble cocoa will dissolve in cold water"	Physician's sample		10435	64

Analyses of cocoa preparations made in the laboratory of the U. S. Department of Agriculture.

[See footnotes for explanation of column headings and signs used in this table.]

Serial number.		Consecutive number.	Microscopical examination.			Chemical examination.											
			Impks. ¹	Foreign starches. ²	Remarks.	Moisture.	Fat.	Sugars.		Crude fiber.	Ash. ³			See foot note ⁵ .	Consecutive number.	Serial number.	
								Cane sugar, by polariscope.	Reducing sugars.		Total ash.	Acid equivalent.	Acid equivalent per cent ash.				
			<i>Plain chocolates.</i>														
6876	1		2	Much wheat starch.....		<i>Pr. et.</i> 3.09	49.81	<i>Pr. et.</i>							2.17	1	6876
6877	2		2	Much wheat flour.....		3.82	49.40								2.34	2	6877
8891	3		1	0		3.18	50.84								2.44	3	8891
8903	4		2	Much wheat starch.....		3.17	45.21								2.37	4	8903
8916	5		2	0		2.70	50.01								2.36	5	8916
8992	6		2	Much arrowroot.....		4.53	44.32								2.34	6	8992
			<i>Sweet chocolates.</i>														
8912	7		1	0		1.88	24.04	53	None	1.32	1.69	2.90	1.72	3.15	7	8912	
8920	8		2	0		1.15	17.73	65	None	.94	1.21	1.50	1.24	1.85	8	8920	
8930	9		1	0	Vanilla.....	1.46	25.74	55	None	1.14	1.54	1.85	1.20	1.76	9	8930	
8986	10		2	Very small amount of arrowroot.	do.....	2.20	25.84	51	None	1.30	1.66	2.25	1.36	2.12	10	8986	
8888	11		2	0	do.....	.88	26.03	55	None	1.25	1.52	2.25	1.48	2.44	11	8888	
8892	12		1	0	do.....	.99	25.00	51	None	1.48	1.81	2.65	1.46	2.39	12	8892	
8893	13		2	0	Vanilla and cinna- mon.	.65	22.49	57	None	1.23	1.52	2.00	1.32	2.02	13	8893	
8990	14		2	0	Vanilla.....	.55	22.52	57	None	1.33	1.67	2.40	1.44	2.33	14	8990	
8991	15		2	0	do.....	.55	25.53	51	None	1.39	1.88	2.80	1.49	2.45	15	8991	
8997	16		0	0	do.....	.88	21.31	58	None	1.10	1.40	2.05	1.47	2.38	16	8901	

8919	17	Hawley & Hoop's Peerless Vanilla Chocolate.	3	0	No vanilla tissue ¹ could be found.	11.04	72	Much.					17	8919
8921	18	Fullé & Co.'s La India Vanilla Primera Chocolate.	2	0	Vanilla and cin- namon.	.78	59	None	1.19	1.71	2.55	1.49	2.48	18
8924	19	Chocolate á la vanille (no name on package, bought of S. B. Taylor, 1610 7th street, Washington, D. C.).	4	Much wheat flour.....	No vanilla tis- sue.	2.42	51	None	1.42	1.89	2.70	1.43	2.31	19
8914	20	Bensdorp's Sweet Vanilla Chocolate.....	4	0	Cinnamon and a very small amount of va- nilla.	.92	61	None	1.25	1.38	1.75	1.27	1.90	20
8894	21	Maillard's Sweet Chocolate.....	2	Small amount wheat starch.		1.18	55	Some	1.33	1.76	2.15	1.22	1.81	21
8895	22	Maillard's Panama Sweet Chocolate.....	4	Considerable amount wheat starch.		1.43	57	None	1.18	1.58	1.90	1.26	1.67	22
8901	23	J. S. Fry & Son's Diamond Sweet Chocolate.	2	Much wheat starch with some arrowroot.		2.02	55	Some	.81	1.16	1.45	1.25	1.86	23
8902	24	J. S. Fry & Son's Sweet Chocolate.....	2	Some wheat flour (not as much as No. 24) and arrowroot.		.85	64	None	.89	1.08	1.45	1.34	2.07	24
8904	25	Rockwood's German Sweet Chocolate.....	2	Much wheat flour.....		1.93	57	Some	1.13	1.38	1.80	1.30	2.00	25
8917	26	S. German's German Sweet Chocolate.....	0	0		1.04	57	None	1.12	1.28	1.65	1.29	1.96	26
8918	27	Mexican Sweet Chocolate (no name on pack- age, bought of G. E. Kennedy & Sons, 1299 F street, Washington, D. C.).	2	0	Vanilla.....	1.55	57	Trace.	1.57	1.67	2.65	1.59	2.73	27
8922	28	Wilbur's Paris Sweet Chocolate.....	4	Much wheat flour.....		2.14	59	Much.	1.10	1.75	2.40	1.37	2.16	28
8923	29	Runkel Brothers' Vienna Sweet Chocolate..	3	Much rice starch.....		2.10	58	Trace.	1.06	1.18	1.70	1.44	2.33	29

¹ In the column headed "husks" the following signs are used: 0 signifies that no characteristic husk tissue could be found; 1 signifies that the husk has probably been mostly removed; 2 signifies that the husk has possibly been partly removed; 3 signifies that the husk is probably all present; 4 signifies that the husk that belongs to the seed is present and probably more.

² A zero (0) is used to indicate those samples that contained no foreign starch. The distinction between wheat starch and wheat flour is neither close nor important. When bran structures occur somewhat abundantly, it is put down as wheat flour; otherwise, as wheat starch.

³ All ashes were white or grayish white.

⁴ The acid equivalent is the number of cc of decinormal acid required to neutralize the ash from 2 grams of material.

⁵ The numbers in this column are obtained by the following formula: acid equivalent ÷ per cent ash = $\frac{\text{acid equivalent} \times 100}{2}$ = the number given.

Analyses of cocoa preparations made in the laboratory of the U. S. Department of Agriculture—Continued.

Microscopical examination.		Chemical examination.														
Serial number.	Consecutive number.	Description of sample.	Husks. ¹	Foreign starches. ²	Remarks.	Moisture.	Fat.	Sugars.		Crude fiber.	Ash. ³			Consecutive number.	Serial number	
								Cane sugar by polariscope.	Reducing sugars.		Total ash.	Acid equivalent. ⁴	Acid equivalent + per cent ash. ⁴			See foot note 5.
8925	30	<i>Sweet chocolates</i> —Continued. French Sweet Chocolate (no name on package, bought of E. S. Schweitzer, 1308 7th street, Washington, D. C.).	4	0		1.75	Pr. ct. 22.78 54	Pr. ct. 54	None	Pr. ct. 1.99	Pr. ct. 2.20	Cc. 3.60	1.64	2.88	30	8925
8926	31	Schmitz's Spiced Zealand Sweet Chocolate (made by Royal Cocoa and Chocolate Co., Philadelphia, Pa.).	2	0	A little vanilla	1.34	18.47	63	None	1.20	1.63	2.60	1.60	2.77	31	8926
8929	32	Crown Sweet Chocolate (no name on package, bought of D. Mazzechi, 1404 14th street, Washington, D. C.).	4	Much wheat flour.....		1.46	21.50	60	None	1.13	1.17	1.70	1.45	2.36	32	8929
8993	34	Wilbur's Broadway Sweet Chocolate.....	2	do		2.06	17.47	63	Some	.95	1.31	2.00	1.57	2.56	34	8993
8994	35	Wilbur's Vanilla Sweet Chocolate.....	2	do	Vanilla		20.59	65	Trace	.74	0.97	1.25	1.29	1.95	35	8994
8995	36	Rockwood's Dutch Sweet Chocolate.....	2	Some wheat flour			22.57	58	None	1.18	1.30	2.05	1.58	3.11	36	8995
6831	37	<i>Cocoa, bromax, etc.</i> Baker's Cocoa.....	3	0			32.52			5.02	4.20	6.25	1.40	2.46	37	6831
6899	38	Baker's Breakfast Cocoa.....	1	0			25.83			4.23	5.05	7.3	1.45	2.34	38	6899
6891	39	Wilbur's Breakfast Cocoa.....	2	Small amount of wheat starch.			30.70			3.79	4.54	6.25	1.58	2.17	39	6891
6893	40	Van Houten's Cocoa.....	1	0			29.81			4.38	8.64	16.05	1.86	3.66	40	6893
6896	41	London Cocoa (H. O. Wilbur & Co., New York, agents).	3	Very largely diluted with arrowroot.			11.13	32	Some	2.13	2.82	3.9	1.38	2.18	41	6896
8901	42	Rowntree's Elect Extract of Cocoa.....	2	0			27.56			4.42	8.48	16.6	1.96	3.70	42	8901
8889	43	Baker's Cracked Cocoa.....					50.85			83.92	3.17	.9	1.55	2.43	43	8889

8896	44	Maillard's Breakfast Cocoa	2	Considerable amount of wheat flour with some arrowroot.		35.85		3.08	3.84	5.2	1.35	2.11	44	8896	
8897	45	Maillard's Dietetic Cocoa	2	Considerable amount of arrowroot.	Barley is also present, pos- sibly added as malt.	25.63	31 None	1.87	2.46	3.35	1.36	2.14	45	8897	
8899	46	J. S. Fry & Sons' Cocoa Extract	1	0		30.95			3.89	4.24	5.8	1.44	2.15	46	8899
8900	47	J. S. Fry & Sons' Malted Cocoa	1	Very small amount of arrowroot.		19.07	(28) Much ¹	3.46	3.91	5.05	1.29	1.97	47	8900	
8905	48	Rockwood's Breakfast Cocoa	2	0		30.12			4.14	4.76	6.75	1.42	2.30	48	8905
8906	49	Rockwood's Table Cocoa	3	0		36.73			3.79	4.23	6.3	1.49	2.46	49	8906
8908	50	Phillip's Digestible Cocoa	3	Much wheat flour.....		28.72	37 None	1.85	3.29	1.5	0.46	0.52	50	8908	
8909	51	Blooker's Dutch Cocoa	0	0		31.48			3.76	6.06	9.6	1.58	2.77	51	8909
8910	52	Epp's Prepared Cocoa	3	Much arrowroot.....		25.94	26 None	1.51	3.15	2.6	0.83	1.05	52	8910	
8913	53	Bensadorp's Royal Dutch Cocoa	2	0		30.98			3.56	6.95	11.1	1.60	2.77	53	8913
8927	54	Brook's Prepared Cocoa	2	Much wheat starch.....		44.78			2.38	3.02	3.85	1.27	1.93	54	8927
8932	55	Hnyler's Cocoa	2	0		38.31			4.06	4.65	6.3	1.35	2.12	55	8932
6894	56	Baker's Broma.....	1	Much arrowroot.....		23.62	25 Much.	1.33	1.46	2.25	1.54	2.62	56	6894	
8898	57	Maillard's Broma	2	Considerable amount of arrowroot.		28.70	25 None	2.62	2.98	4.6	1.54	2.61	57	8898	
6892	58	Wilbur's Cocoa-Theta	3	Much arrowroot		19.18	55 Traco.	1.20	1.60	2.6	1.63	2.66	58	6892	
8890	59	Baker's Racahout des Arabes ⁶	2	do	Considerable amount of powdered salep.	3.40	58 None	.92	0.98	1.6	1.63	2.86	59	8890	

¹In the column headed "hunks" the following signs are used: 0 signifies that no characteristic husk tissue could be found; 1 signifies that the husk has probably been mostly removed; 2 signifies that the husk has possibly been partly removed; 3 signifies that the husk is probably all present; 4 signifies that the husk that belongs to the seed is present and probably more.

²A zero (0) is used to indicate those samples that contained no foreign starch. The distinction between wheat starch and wheat flour is neither close nor important. When bran structures occur somewhat abundantly, it is put down as wheat flour; otherwise, as wheat starch.

³All ashes were white or grayish white.

⁴The acid equivalent is the number of cc. of decinormal acid required to neutralize the ash from 2 grams of material.

⁵The numbers in this column are obtained by the following formula: acid equivalent ÷ per cent ash — $\left(\frac{\text{acid equivalent} \times 100}{2} \right) = \text{the number given.}$

⁶Not comparable with the other results, since a powder can not be as finely divided by ordinary laboratory methods as is done in chocolate works.

⁷Rotation and reduction due to malt extract and not to sugar added as such.

⁸Formula for the preparation of racahout (Pharmaceutical Record, 1892, 13, 305): Powdered chocolate, 1 pound; corn starch, $\frac{1}{3}$ pounds; powdered salep, $\frac{1}{4}$ pound; sugar, 4 pounds. Vanilla to flavor.

Serial number.		Consecutive number.		Microscopical examination.			Chemical examination.							Serial number.	
Description of sample.		Husks. ¹	Foreign starches. ²	Remarks.	Moisture.	Sugars.		Crude fiber.	Ash. ³			See foot note ⁵ .	Consecutive number.		
						Cane sugar, by polariscope.	Reducing sugars.		Total ash.	Acid equivalent.	Acid equivalent ÷ per cent ash. ⁴				
8911	60	Schweitzer's Cocoatina	1	0		Pr. et. 31.13		Pr. et. 3.70	Pr. et. 6.33			1.48	2.44	60	8911
8915	61	Smith Manufacturing Company's Alkethrepta.	2	Much corn starch.....		20.16	47	None	1.26	1.38	2.2	1.60	2.75	61	8915
8985	62	De Langrenier's Racahout des Arabes ⁶	1	Much potato and rice starches.	Vanilla	7.23	51	Trace.	.52	0.70	1.0	1.43	2.33	62	8985
8989	63	Hawley & Hoop's Breakfast Cocoa	2	Small amount arrow-root.		26.97			3.99	4.69	6.0	1.28	1.94	63	8989
10485	64	De Jong's Cocoa.....	0	0						7.89	14.4	1.83	3.54	64	10485

¹ In the column headed "husks" the following signs are used: 0 signifies that no characteristic husk tissue could be found; 1 signifies that the husk has probably been mostly removed; 2 signifies that the husk has possibly been partly removed; 3 signifies that the husk is probably all present; 4 signifies that the husk that belongs to the seed is present and probably more.

² A zero (0) is used to indicate those samples that contained no foreign starch. The distinction between wheat starch and wheat flour is neither close nor important. When bran structures occur somewhat abundantly, it is put down as wheat flour; otherwise, as wheat starch.

³ All ashes were white or grayish white.

⁴ The acid equivalent is the number of cc. of decinormal acid required to neutralize the ash from 2 grams of material.

⁵ The numbers in this column are obtained by the following formula: acid equivalent $\div$ per cent ash $\times$ (acid equivalent $\times .0053 \times 100$) = the number given.

⁶ Formula for the preparation of racahout (Pharmaceutical Record, 1892, 13, 305): Powdered chocolate, 1 pound; corn starch, $\frac{1}{4}$ pound; powdered salep, $\frac{1}{4}$ pound; sugar, 4 pounds. Vanilla to flavor.

Summary of the results of analyses made in the laboratory of the U. S. Department of Agriculture.

Number of samples examined.	Character of samples.	Number of samples containing large additions of starch or flour.	Number of samples containing large amounts of cocoa husks.	Samples containing sugar.			
				Number containing 25 to 40 per cent.	Number containing 40 to 50 per cent.	Number containing 50 to 60 per cent.	Number containing 60 to 72 per cent.
6	Plain chocolate	4					
30	Sweet chocolate	11	8			22	8
28	Cocoas, bromas, etc.	12	6	7	1	3	
64	Total	27	14	7	1	25	8

Determinations made to show the solubility of cocoa and certain of its constituents in water.

Consecutive number.	Serial number.	In the air-dry sample.					Number of parts of ash in the water-soluble portion, per 100 parts of ash.	Number of parts of P_2O_5 in the water-soluble portion, per 100 parts P_2O_5 .
		Total ash.	Total P_2O_5 .	Total matter soluble in water.	Portion soluble in water contains—			
					Ash.	P_2O_5 .		
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		
38	6899	5.05	-----	18.27	3.87	1.27	77	-----
40	6893	8.64	1.99	19.84	5.77	1.02	67	51
42	6901	8.48	1.71	-----	-----	0.87	-----	51
43	8889	3.17	0.94	11.28	2.70	0.80	85	85
51	8909	6.06	1.72	17.92	2.27	1.27	37	74
53	8913	6.95	1.94	19.17	4.21	0.77	61	40
64	10485	7.89	-----	19.76	5.36	0.95	68	-----

Before proceeding with a discussion of the results obtained in this laboratory, a brief review of the methods proposed by various analysts for the judgment of samples and the interpretation of analyses is not considered out of place.

The Association of Swiss Analytical Chemists¹ regards the following determinations as absolutely necessary:

(a) Microscopical examination for foreign starches, cocoa husks, flour, etc.

(b) Quantitative estimation of moisture (in cocoa powder), ash, fat, sugar (in chocolate), and fiber.

(c) Taste, odor, and color of the aqueous infusion, as well as the appearance of the surface of fracture in cake chocolate and cacao-masse.

It is sometimes of value to determine—

(a) Quantitatively: Theobromine, tannic acid, and starch.

(b) Qualitatively: Fat and ash (for alkaline carbonates, mineral pigments, etc.).

Not more than 2 per cent of alkaline carbonates in soluble cocoas is considered allowable by this association. The ash in normal samples of cacao-masse may vary from 2 to 5 per cent; the fat, from 48 to 54.5 per cent.

¹ *Op. cit.*, note 3, p. 949 of this work.

Mansfield¹ states the average amount of starch to be 5 per cent in chocolate and 10 per cent in cocoa. He also gives the maximum amount of cellulose as 2.5 per cent for chocolate and 5 per cent for cocoa.

Bensemman² proposes the determination of water-insoluble organic matter (=U), fat (=F) and starch (=S), as a means of judging the percentage of cocoa and flour in chocolate. He calculates these percentages from S and the coefficient $\frac{S}{U-F}$, which he calls the starch coefficient.

The following results were obtained by Bensemman:³

	I.	II.	III.	IV.	V.	VI.
Per cent insoluble organic bodies dried at 100 to 110° C, =U.....	69.0	35.5	36.0	37.0	37.0	30.0
Per cent fat, extracted with ether, dried at 100 to 110°, =F.....	23.0	22.5	21.0	17.5	19.0	6.5
Per cent starch, estimated as starch sugar by amount CuO, =S.....	13.0	4.5	8.5	12.0	11.5	17.0
Starch coefficient, = $\frac{S}{U-F}$	0.317	0.346	0.567	0.615	0.639	0.723

I. Was so-called soluble Dutch cacao.

II. Table chocolate (about 60 parts sugar, 40 parts cacao).

III. Crumb chocolate (about 60 parts sugar, 25 parts cacao, and 15 parts flour).

IV and V. Table chocolate (about 60 parts sugar, 20 parts cacao, and 20 parts flour).

VI. Chocolate flour (about 60 parts sugar, 10 parts cacao, and 30 parts flour).

Filsinger⁴ makes the following recommendations:

Quantitative determination of ash (qualitative examination, if ash runs above 5 per cent in cocoa and 2.5 to 3 per cent in chocolate), fat and sugar. The fat is also examined qualitatively. The microscopic examination is considered very important.

Herbst⁵ determines in chocolate: Moisture, fat (quantitatively and qualitatively), ash, and sugar; and makes a microscopical examination. The ash should not exceed 2 per cent.

The following is abridged from Bernhardt:⁶

In many cases chocolates, to which a large amount of flour and starch has been added, must be colored. The author found a Spanish chocolate, which contained 3.2 per cent ash, to be colored with 1.5 per cent ochre. As these additions are only assimilated by the chocolate in proportion to the amount of fat present, the addition of coloring matter necessitates the adulteration with foreign fats, so that we may obtain a chocolate that contains no cocoa whatever. The author has in reality found chocolates which consisted of cocoa-remnants, fat, sugar, spices, and coloring matter.

As cocoa butter is quite expensive, other fats are often added, and this writer therefore considers the examination of the fat as the most important test. The following fats are mostly used as substitutes; cocoanut butter, rasped cocoanut, hazelnuts, almonds, animal fats, margarin, cotton and sesame oils, etc.

¹ *Op. cit.*, note 1, p. 950 of this work.

² Rep. f. anal. chem., 1883, 119.

³ See also tables on pp. 967 and 968.

⁴ *Op. cit.*, note 7, p. 938 of this work.

⁵ *Op. cit.*, note 4, p. 950 of this work.

⁶ Z. Nahrungsm. Hyg., 1890, 4, 121.

Vanilla and vanillin are often replaced by balsam Peru, storax, tolu, and gum ben-zoin.

The melting and congealing points of the fats and of the free fatty acids are no indication whatever, as pure cocoa butter shows great variations.

Legler¹ regards the cellulose determination as an uncertain means of detecting husks, because the results vary with the method used.

DISCUSSION OF THE RESULTS OBTAINED.

While the time at our disposal has not permitted as thorough an investigation in some details of the work as seemed desirable, sufficient data have been obtained to show the general character and extent of the adulteration of the cocoa preparations sold on our markets.

The determination of the ash serves as a means of determining the extent of dilution with foreign materials (the nature of these materials being known) of organic or inorganic origin. The per cent of ash decreases with the addition of substances low in ash, as starch, flour, etc.; increases with the removal of fat, the addition of mineral matter for weight or color, or the addition of alkaline carbonates in the process of manufacture. The percentages of ash, fat, sugar, and starch must be considered with reference to each other in order to determine whether any one is abnormal. Any addition of mineral matter that could possibly be of value for increasing the weight or color of the preparation would certainly render the ash conspicuously abnormal in amount or color.

For the detection of the use of fixed alkalis in the process of manufacture, the number of cubic centimeters of decinormal acid necessary to neutralize the ash from 2 grams of material was determined. The number thus obtained is put down in the table of results as *acid equivalent*. By use of this number and the number expressing the per cent of ash in the sample, the following additional numbers were calculated:

$A = \text{acid equivalent} \div \text{per cent ash.}$

$B = \text{acid equivalent} \div \text{per cent ash} - \text{the per cent of alkali in the ash (calculated as } \text{Na}_2\text{CO}_3) = \text{acid equivalent} \div \text{per cent ash} - \left(\frac{\text{acid equivalent} \times .0053 \times 100.}{2} \right)$

In the samples yielding a normal ash, A varies from 1.20 to 1.72 and exceeds 1.50 in eleven cases; B varies from 1.67 to 3.15 and exceeds 2.80 in three cases.

The determinations proposed by Stutzer were made in only six samples. It is greatly regretted that time did permit more of these determinations, since the method seems to be of considerable value, although the differences in the results are not as marked as are those obtained by Stutzer (see pp. 943 and 985).

The only decidedly abnormal ashes were those from samples Nos. 40, 42, and 64 which were unquestionably prepared by use of alkalis (see table of results). The ash figures obtained for samples 51 and 53, while

¹ Rep. f. anal. chem., 1884, 4, 345.

not so pronounced, are sufficiently high to cause suspicion of the use of alkalis.

A determination of the fat shows the amount of this important food material in the preparation, and is of value in determining the extent of adulteration with other substances. The amount of fat was not sufficiently inconsistent with the amount of the other ingredients to attract suspicion to any one of the samples. Time did not permit a qualitative examination of the fat from each sample.

Sugar and starch are used to a most deplorable extent, as the results in the table show. The quality of the sugar used is generally good, however, as is shown by the somewhat rare occurrence of reducing sugars.

A determination of theobromine is of very little value, since it has no commercial importance to tempt its removal, as is the case with cocoa butter. Its determination can, therefore, only be of value for judging the extent of adulteration with other substances, for which purpose a nitrogen determination would serve just as well (in the absence of nitrogenous adulterants), and is much more easily made.

A fiber determination is of value for the detection of the presence of husks and other substances rich in cellulose, and for judging the extent to which these substances have been added. The value of the determination is greatly reduced, however, by the variation in the result that follows the degree of fineness of the powder. It is very difficult indeed to produce a sample by laboratory means that will give results that are comparable with those obtained with samples ground in chocolate works. Our comparisons must be, therefore, between commercial samples of known purity and those of questionable purity.

In adding "phosphates as found in wheat," the manufacturers of sample No. 50 seem to have overlooked the fact that the husked cocoa bean is fully as rich in phosphoric acid as is wheat.

Beef tea was once considered to be a very concentrated and easily digestible food, and was given to invalids in small quantities with full confidence in its great, almost miraculous, nourishing power. It has long since been degraded very nearly to the rank of a mere stimulant and is never intelligently administered except when accompanied by an ample amount of nourishing food. As a concentrated and easily digestible food for invalids cocoa preparations are already beginning to share the same fate; as material for the preparation of pleasant, exhilarating, and slightly nutritive beverages for both weak and strong, the career of cocoa preparations is only just begun. Moreover, their progress in popular favor will keep pace with the manufacturers' appreciation of this fact.

For a study of the nutritive value of cocoa, the following data can be taken as fairly representative of cocoas from which a part of the fat has been removed, and to which no foreign substances have been added:

Approximate average composition of pure cocoas, partially deprived of fat.

Nutritive constituents.	Per cent.
Nitrogenous substances.....	20
Fat.....	30
Carbohydrates ("nitrogen-free extract," including starch, etc.)	30

It must be remembered, however, that only about one-half of these nitrogenous substances are digestible protein, and the carbohydrate figure given includes several substances of doubtful nutritive value.

Amount of nutritive materials required per day by a man doing moderate manual labor.¹

Nutrients.	Authority.	
	Voit.	Atwater.
	<i>Grams.</i>	<i>Grams.</i>
Protein.....	118	125
Fat.....	56	125
Carbohydrates.....	500	450

¹ W. O. Atwater and Charles D. Woods, Fourth Annual Report of the Storrs School Agricultural Experiment Station, Storrs, Conn., 1891.

The amount of cocoa used for the preparation of a cup of the beverage is generally less than 2.5 grams. From these data, the weights of the principal nutrients in a cup of cocoa and the equivalent parts of an average daily ration, given in the following table, were calculated:

Nutrients in a cup of cocoa, made from 2.5 grams of material.

Nutrients.	Weight in grams.	Part of an average daily ration.
Protein.....	0.5	$\frac{2}{3}$ to $\frac{1}{2}$
Fat.....	0.6	$\frac{1}{3}$ to $\frac{1}{4}$
Carbohydrates.....	0.6	$\frac{1}{3}$ to $\frac{1}{4}$

In these calculations the variable additions of milk and sugar are disregarded.

The results of these investigations emphasize in many ways the many pleas that have been made for the establishment of standards of purity, strength, and quality for foods—for some certain means of enabling the public to know the strength, quality, and degree of purity of the food materials on the markets. The question of economy alone is sufficiently important to justify serious consideration of this need, for no question can be of more importance to a great part of our nation than questions of economy in food, drink, and clothing.

APPENDIX A.

BIBLIOGRAPHY OF THE LITERATURE OF TEA, COFFEE, AND COCOA PREPARATIONS.

GENERAL WORKS.

- BATTERSHALL. Adulteration of food. 1887.
- BELL. Analysis and adulteration of food.
- BLYTH. Foods: Their composition and analysis. London, 1882.
- CHEVALLIER. Dictionnaire des altérations et falsifications des substances alimentaires, médicamenteuses et commerciales, avec l'indication des moyens pour les reconnaître. Paris, 1878.
- COSTER, HOORN and MAZURE. The adulteration of tea, coffee, and chocolate in Holland. Rev. internat. scient. et pop. d. falsifications d. denrées aliment., 4, 7.
- DAMMER. Illustriertes Lexicon der Verfälschungen und Verunreinigungen der Nahrungs- und Genussmittel. Leipzig, 1886.
- DIETRICH und KÖNIG. Zusammensetzung der Futtermittel.
- DIETZSCH. Die wichtigsten Nahrungsmittel und Getränke, deren Verunreinigungen und Verfälschungen, u. s. w. Zürich, 1884.
- ELSNER. Die Praxis des Nahrungsmittel-Chemikers. Hamburg—Leipzig, 1882.
- FRANÇAISE RÉPUBLIQUE, PRÉFECTURE DE POLICE. Documents sur les falsifications des matières alimentaires et sur les travaux du laboratoire municipal.
- HANAUSEK. Die Nahrungs- und Genussmittel aus dem Pflanzenreiche. Kassel, 1884.
- HASSALL. Food: Its adulterations and the methods for their detection. London, 1876.
- HUSEMANN. Pflanzenstoffe.
- KÖNIG. Die menschlichen Nahrungs- und Genussmittel, ihre Herstellung, Zusammensetzung und Beschaffenheit, ihre Verfälschungen und ihre Nachweisung. Berlin, 1882.
- KÖNIG. Chemie der menschlichen Nahrungs- und Genussmittel, 3. Aufl., Band I. Berlin, 1889.
- MACÉ. Les substances alimentaires étudiées au microscope. Paris, 1891.
- MOELLER. Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche. Berlin, 1886.
- PRESCOTT. Organic analysis. New York, 1887.
- SCHIMPER. Anleitung zur mikroskopischen Untersuchung der Nahrungs- und Genussmittel. Jena, 1886.
- SMITH. Dictionary of economic plants.
- SOUBEIRAN. Nouveau dictionnaire des falsifications et des altérations des aliments, etc. Paris, 1874.
- THORPE. Dictionary of applied chemistry. London, 1890.

- WALCHNER. Die Nahrungsmittel der Menschen, ihre Verfälschungen und Verreinigung. Berlin, 1875.
- WANKLYN. Tea, coffee, and cocoa.
- ZIPPERER. Microchemie des Cacao und Thees. Bericht. siebenten Vers. bayer. Vertreter d. a. Chemie, 64.

BIBLIOGRAPHY OF THE LITERATURE OF TEAS.

- ALLEN, A. H. Tea, adulterations of. Chem. News, **29**, 167, 189, 221; **30**, 2.
- ANONYMOUS.—Batalin, a substitute for tea. Chem. Centrbl., **1**, 52.
- ANONYMOUS. TEA, Brick. Chem. Ztg., 1889, No. 11; Zeitsch. f. Nahr.-Unters. u. Hyg., 1889, 25.
- Teas. Pharm. Jour., Nov. 19, 1887; Journal de Pharm. et de Chim., Feb. 15, 1888, p. 196; Revue Agricole de Maurice, 3^{me} année, 4, 92; Pharm. Jour., **6**, 1875, 261, 263, 281, 282, 402, 404.
- Tea ash. Analyst, **7**, 1882, 7.
- Tea, Brush. Pharm. Jour., 1853, 172.
- Tea, Green. Pharm. Jour., [3], **6**, (1876), 746.
- Tea, Japanese. Pharm. Jour., [2], **9**, (1868), 343.
- BALL, SAMUEL. Tea: Its cultivation and manufacture. London, 1848.
- BAILDON, SAMUEL. Tea in Assam. Calcutta, 1877.
- BELL, J. C. Tea, Ash of. Analyst, 1882, 7; Jsb. d. Chem., 1882, 1451.
- BING, I. Teas and coffees, nitrates in. Jour. pr. Chem. [2] **22**, 348.
- BLYTH, A. W. Tea, India. Chem. News, **30**, 212.
- CAMPBELL-BROWN, J. Tea plant, agricultural chemistry of. J. Chem. Soc. [2] **13**, 1217.
- DRAGENDORF. Tea, adulterations of. (Seven samples contained exhausted leaves; 16 adulterated with foreign leaves.) Chem. Centrbl., **2**, 492.
- EDER. Catechu and logwood, detection of in teas. Zeitsch. f. anal. Chem. **19**, 372.
- EDER, J. M. Teas, analysis of. Dingl. polyt. Jour., **231**, 445, 526.
- GRISLER, J. F. Teas, Analyses of. Analyst, **9**, 220; orig. American Grocer, Oct. 23, 1884.
- GRIFFITH, WILLIAM. Tea Plant of Assam. London, about 1834.
- HAGER, H. Teas, Analysis of. Zeitsch. f. anal. Chem., 1880, 371.
- HILL, A. Tea, Analyses of. Analyst **6**, 232.
- HODGES. Teas, eachar. Chem. News, **30**, 114.
- HORSFORD. Tea, composition of ash. Sill. Am. J. [2], **11**, 249. Jsb. d. Chem., (1851), 717.
- HUSSON, C. Tea and coffee, examination of. Ann. chim. phys. [5] **16**, 419.
- INDIA, GOVERNMENT OF. (Home department.) Tea cultivation in the northwest provinces and in the Punjab. Report on the present condition and future prospects. Calcutta, 1857.
- KOSSEL, A. Théophylline, a new alkaloid in tea. Journ. de Pharm. et de Chim., March, 1889, 297; Zeitsch. f. phys. Chem., **13** (1888), 298.
- KOZAI, Y. Tea leaves, comparison between the composition of unprepared green and black. Journ. Tokyo Chem. Soc., 1889, **10**, No. 8; Rep. **12**, 109.
- Tea. Researches on the manufacture of. Imp. College Agric. Tokyo, Komaba, Japan, Bull. No. 7.
- KRAUS, C. Tea, percentage of extract. Neues Rep. Pharm., **21**, 327; Jsb. d. Chem., (1872), 805.
- LEHMANN, J. Tea and coffee, analysis of. Liebig's chem. Briefe, 3 ansl., 611; Jsb. d. Chem., 1851, 717.
- LEEDS, A. R. Tea, substitutes and adulterations. Chem. News, **44**, 244; Am. J. Chem. Soc., **3** (1881).

- LEES, NASSAU W. Tea districts of Eastern Bengal, a memorandum written after a tour through the, in 1864-'65. Calcutta, 1866.
- LEES, NASSAU W. Tea cultivation, cotton, etc., in India. London, 1863.
- MCGOWAN, A. T. Tea planting in the outer Himalaya. London, 1861.
- MCPHERSON, JAMES. Tea planter, the Neilgherry. Madras, 1870.
- MONEY, EDWARD. Tea, the cultivation and manufacture of. London, 1878.
- MIRSTADT. Tea, adulteration of. Rev. Intern. Scien. et Pop. des Falsif. des Denrées aliment., **4**, 39.
- SUMNER, JOHN. Tea, a popular treatise on; its qualities and effects. Birmingham, 1863.
- STOKER, T. G. Tea plant, notes on the management of the. Calcutta, 1874.
- WIGNER, G. W. Teas, analyses of. Chem. News, **32**, 166, 189, 235. Phar. J. Trans. [3], **6**, 261, 281, 402.
- Tea, composition of. Phar. J. Trans. [3], **4**, 909.
- WILSON, A. S. Teas, analyses of. Chem. News, **28**, 307.
- WINICKI. Tea, adulteration of. Pharm. Centr. in Dingl. polyt. Jour., **217**, 256.
- ZIPPERER, PAUL. Teas and cocoas, microchemistry of. Ber. d. 7ten Vers. d. F. Vereinigung bay. Vertreter d. ang. Chem., Speier., **10**, 11, 12 Sept., 1888, 64.
- ZÖLLER, PII. Tea leaves, ash of. Ann. Chem. Pharm., **158**, 180; Neues Rep. Pharm., **20**, 457; Pharm. J. Trans., 1871 [3], **2**, 161.

BIBLIOGRAPHY OF THE LITERATURE OF COFFEES.

- ALLEN, A. H. Coffee, examination of. Chem. News, **29**, 129, 140, 167.
- ANONYMOUS. Coffee, acorn. Pharm. Jour., 1876, 772.
- Coffee, colored. Jour. de Pharm. et de Chim., 15 Feb., 1888, 198.
- Coffee, extract of. The Jour. Soc. Chem. Industry, 1887, p. 377-577.
- Coffee and its adulterations. Pharm. Jour., **10** (1851), 394-396.
- BIBRA. Coffee, adulteration of. Abhandl. d. natnrwiss.-techn. Commission d. k. bayr. Akad. d. Wissenschaft., **2**, 219; Jsb. d. Chem., 1858, 659.
- BOUSSINGAULT. Coffee, sugars in. Compt. Rend., **91**, 639; Jsb. d. Chem., 1880, 1069.
- BERNHEIMER. Coffee, products of roasting. Wien. Akad. Ber. (2 Abth.) **81**, 1032; Jsb. d. Chem., 1880, 1069.
- BOUTFRON-CHARLARD et ROBIQUET. Coffee, analysis of. Ann. de Chem. et de Pharm., **23**, 93.
- CECH, C. O. Coffee oil. J. pr. Chem. [2], **22**, 395; Jsb. d. Chem., 1880, 1082.
- COMMAILLE, A. Coffee, analysis of. Monit. scient. [3], **6**, 779.
- DIERBACH. Coffee, green. Ann. Chem. Pharm., **14**, 231.
- DRAPER, J. C. Coffee adulterations, detection of. Phil. Mag. [4], **34**, 104; Zeitsch. f. anal. Chem., **7**, 388.
- DUNSTAN. Coffee, Mussaenda. Phar. J. Trans., Nov. 16, 1889, 381; Am. J. Pharm., **20**, 4, 174.
- EDDY, C. E. Coffee, analysis of. Am. Chem. J., **7**, 45; Jsb. d. Chem., 1876, 1149-50.
- EDSON, C. Coffee, imitation Java. Analyst, **9**, 128.
- FRICKE, E. Coffee, imitation. Zeit. f. angew. Chem., 1889, 310-311; Chem. Centrbl., 1889, 639.
- FERRARI. Coffee syrup. Ann. Chem. Pharm., **9**, 246.
- GEISSLER, E. Coffee, Mogdad. Pharm. Centralhalle, **22**, 134; Zeitsch. f. anal. Chem., **21**, 438.
- GRAHAM, STENHOUSE and CAMPBELL. Coffee, ash analysis of. J. Chem. Soc., **9**, 33.
- GRAHAM, STENHOUSE and CAMPBELL. Coffee adulterations. Phar. J. Trans., **26**, 228, 521; Jsb. d. Chem., 1856, 814.
- HAGER, H. Coffee substitutes. Chem. Centrbl., 1880, 398; Jsb. d. Chem., 1880., 1070.
- HEHNER. Coffee leaves, examination of. Analyst, 1879, 84.

- HEHNER, O. Coffees, roasted and unroasted, analyses of. *Analyst*, 1879, 84.
- HLASIWETZ. Caffetannic acid, formula of. *Ann. de Chem. et de Pharm.*, **142**, 219.
- HLASIWETZ, H. Hydrocoffeic acid. *Wien Acad. Ber.*, **55**, 337; *J. prakt. Chem.*, **103**, 41; *Chem. Centrbl.*, 1867, 1893.
- HUSEMANN. Fat of coffee. *Pflanzenstoffe*, p. 1367.
- JANECEK. Coffee, Mogdad. *Chem. Ztg.*, 1880, 442; *Jsb. d. Chem.*, 1880, 1070.
- JANKE, L. Coffee, analysis of. *Veröff. d. Reichsgesundheitsamtes*, 1880, 157, 175, *Jsb. d. Chem.*, 1880, 1343.
- KÖNIG, J. Coffee beans, coating of. *Chem. Centrbl.*, 1889, **20**, 1, 51.
- KÖNIG, J. Coffee, imitation. *Chem. Centrbl.*, (1889), **20**, 1, 51.
- KORNAUTH, C. Coffee beans, imitation. *Rev. internat. scient. et popul. des falsifications des denrées aliment.*, **3**, 195-196.
- KORNAUTH, C. Coffee and coffee substitutes. *Mitt. a. d. pharm. Ins. und Lab. f. angew. Chem.*, Erlangen, Heft 3, 1-56.
- KORNAUTH, C. Coffee, figs and chicory as substitutes. *Rev. internat. scient. et popul. des falsifications des denrées aliment.*, **3**, 8; *Chem. Centrbl.*, 1890, 605.
- KORNAUTH, C. Coffee substitutes. *Zeitsch. f. angew. Chem.*, 1891, 645.
- LUDWIG, H. Coffee tree, ash of various parts of. *Arch. Pharm.* [3], **1**, 482; *Jsb. d. Chem.*, 1872, 804.
- LEHMAN, J. Coffee, ash of aqueous extract of. *Liebig's Chem. Briefe*, 3 Aufl., 611; *Jsb. d. Chem.*, 1851, 717.
- LEVI. Coffee, ash, analysis of. *Ann. de Chem. et de Pharm.*, **50**, 424.
- LEVESIR, O. Coffee, analysis of. *Arch. Pharm.* [3], **8**, 294; *Jsb. d. Chem.*, 1876, 888.
- MANSFELD, M. Coffee, adulteration of. *Rev. internat. scient. et popul. des falsif. des denrées aliment.*, **4**, 40.
- MOELLE, J. Coffee, Mogdad. *Pharm. Centralhalle*, **22**, 133; *Zeitsch. f. anal. Chem.*, **21**, 438.
- MULDER. Javas, yellow and blue. *Jsb. d. Chem.*, 1858, 659.
- NANNING. Coffee coloring. *Weekblad vor Pharm.*, Nov., 1883; *Rep. d. anal. Chem.*, 1884, 12.
- PAUL and COWNLEY. Coffee, caffen in, roasted and unroasted. *Phar. J. Trans.*, 1887, **17**, 821.
- PAYEN. Coffee bean, composition of. *Jsb. d. Chem.*, 1849, 486; *Ann. chim. phys.*, [3], **26**, 108.
- PAYEN. Coffees, roasted and unroasted, analysis of. *Ann. chim. phys.* [3], **26**, 168.
- PFAFF. Caffetannic acid, isolation of. *Schweigg. Journ.*, 62, 31.
- PADÉ, L. Coffee, new adulterations of. *Chem. Centrbl.*, **20**, 2, (1889), 341.
- PADÉ, L. Coffee, imitation Java. *Bull. de la Soc. Chim. de Paris*, **47**, 7; *Chem. News*, **56**, 24.
- PORTELE. Coffee, imitation. *Zeitsch. f. Nahrungsmitteluntersuch. u. Hygiene*, **3**, 221-22; *Chem. Centrbl.*, 1890, 135.
- ROBIQUET ET BOUTRON. Coffee, analysis of. *Jour. f. prakt. Chem.*, **13**, 257.
- ROCHLEDER. Coffee bean, acids of. *Jsb. d. Chem.*, 1851, 410.; *Wien Acad. Ber.*, **7**, 815; *Jour. prakt. Chem.*, **56**, 93.
- ROCHLEDER. Fat of coffee, composition of. *Wien Akad. Ber.*, **24**, 40.
- ROCHLEDER. Coffees, roasted and unroasted, analysis of. *Ann. de Chem. et de Pharm.*, **50**, 224; **59**, 300; **63**, 193; **66**, 35; **82**, 194.
- SAMELSON, J. Coffee, imitation. *Zeitsch. f. angew. Chem.*, 1890, 482.
- SKALWEIT, J. Coffee, determination of extract in. *Russ. Zeitsch. Pharm.*, **21**, 691; *Jsb. d. Chem.*, 1882, 1327.
- SMETHAM, A. Coffee, analysis of. *Analyst*, 1882, 73; *Jsb. d. Chem.*, 1882, 145.
- STENHOUSE. Coffee leaves. *Pharm. J. Trans.*, **13**, 382. *Ann. de Chem. et de Pharm.*, **89**, 244.
- STUTZER, A. Coffee, imitation. *Jour. Franklin Inst.*, **127**, 234; *Zeitsch. f. angew. Chem.*, 1888, 699; *Zeitsch. f. angew. Chem.*, 1890, 549.

- SYKORA, T. Coffee colorings. *Chem. Centrbl.*, 1887, No. 47; *Rep. f. anal. Chem.*, 1887, 765.
- THIEL, C. Coffee substitutes. *Zeitsch. f. angew. Chem.*, 1892, 75.
- TIEMANN and NAGAI. Caffeic acid, relation of to ferulic acid. *Berichte*, 1878, 646; *Jsb.*, 1878, 799.
- TRILLICH, H. Malt-coffee and coffee substitutes. *Zeitsch. f. angew. Chem.*, 1891, 719.
- VOGEL, A. Coffee-beans, composition of roasted and unroasted. *N. Jahrb. d. Pharm.*, 8, 193; *Dingl. polyt. Jour.* 147, 395; *Chem. Centrbl.*, 1858, 367.
- WALLER, E. Coffee, colored imitation Java. Report N. Y. board of health; *Analyst*, 9, 128.
- WEYRICH. Coffee, ash analysis of. *Pharm. Z. Russl.*, 12, 362.
- WEYRICH. Coffees, roasted and unroasted. Analysis of; *Pharm. Z. Russl.* 12, 362.
- WITTSTEIN, G. C. Coffee, polishing of; *Chem. News*, 33, 194.
- WOLFFENSTEIN. Coffee substitutes, analysis of. *Zeitsch. f. angew. Chem.*, 90, 84; *Chem. Centrbl.*, 1890, 606.

COCOA AND COCOA PREPARATIONS.

- ANONYMOUS. Adulteration of chocolate. *Ann. Chem. Pharm.*, 16, 80.
Cacao und dessen Präparate. *Chem. Ztg.*, 1889, 32.
Coloring matter for chocolate. *Analyst*, 1888, 13, 96.
Cultivation of cocoa in Colombia. *Phar. Jour. Trans.* [3], 970, 591.
- ANTHON. Preparation of cocoa butter. *Ann. Chem. Pharm.*, 24, 251.
- ASBORTH. Estimation of starch in cocoa and chocolate. *Rep. anal. Chem.*, 7, 99.
- ASSOCIATION OF SWISS ANALYTICAL CHEMISTS. Directions for the examination and judgment of samples of cocoa preparations. *Vierteljahresschrift u. d. Fortschr. auf dem Gebiete der Chemie d. Nahr.- u. Genussmittel*, 1890, 2, 171.
- BELL. Caffeine in cocoa. Analysis and adulteration of food, p. 85.
- BELOHOUBECK. An analysis of Van Houten's cocoa. *Casopsis českého lékarnictva*, 1888, 7, 311; *Rep. d. Chem. Ztg.*, 1888, 270.
- BELOHOUBECK. Soluble Dutch cocoa. *Böhm. pharm. Zeitsch.*, 7, 311; *Chem. Centrbl.*, 1890, 131.
- BENEDIKT. Analyse der Fette und Wachsarten. Berlin, 1886.
- BENSEMANN. Analysis of chocolate. *Ber. d. chem. Ges.*, 16, 856; *Rep. f. anal. Chem.*, 4, 213; *Ibid.*, 5, 178; *Zeitsch. f. anal. Chem.*, 24, 464.
- BENSEMANN. Estimation of cocoa in chocolate. *Ber. d. chem. Ges.*, 6, 856; *Chem. News*, 48, 305.
- BENSEMANN. Properties of cocoa butter. *Rep. f. anal. Chem.*, 4, 165; *Zeitsch. f. anal. Chem.*, 24, 628.
- BERNHARDT. Examination of cocoa and chocolate. *Zeitsch. Nahrungs- u. Hyg.*, 1890, 4, 121.
- BERNHARDT. Losses in the preliminary preparation of the bean for manufacture. *Chem. Ztg.*, 1889, 32.
- BOUDET and PELOUZE. Examination of cocoa butter. *Ann. Chem. Pharm.*, 29, 41.
- BJÖRKLUND. Ether-test for cocoa butter. *Zeitsch. f. anal. Chem.*, 3, 233; Benedikt, *Analyse der Fette und Wachsarten*.
- BOUSSINGAULT. Constituents of cocoa. *Compt. Rend.*, 1883, 96, 1395.
- BOUSSINGAULT. Cultivation of the cocoa tree. *Compt. Rend.*, 96, 1395; *Jour. Chem. Soc.*, 1883, 44, 933. A second article. *Ann. Chim. Phys.* [5], 28, 433; *Jour. Chem. Soc.*, 46, 202.
- BOUSSINGAULT. Examination of cocoa and chocolate. *Ann. Chim. Phys.* [5], 28, 433.
- BOUSSINGAULT. Properties of cocoa butter. *Ann. Chem. Pharm.*, 21, 200.
- BOUSSINGAULT. Percentage of theobromine in cocoa beans. *Vorträge u. Agricultur-Chemie*, 1872, 451. A second article, *Ann. Chim. Phys.* [5], 28, 433; *Chem. Ztg.*, 1883, 902.

- DELCHER. Historical and chemical researches on cocoa.
- DIETERICH. Specific gravity of cocoa butter. *Geschäftsber. d. Papier- u. chem. Fabrik zu Helfenberg*, 1883; *Zeitsch. f. anal. Chem.*, **23**, 567.
- DIETERICH. Chemical properties of cocoa butter. *Zeitsch. f. anal. Chem.*, **25**, 431.
- DUCLAUX. Copper in cocoa ash. *Bull. Soc. Chim. de Paris*, 1871, **16**, 33.
- ELSNER. Microscopical examination of cocoa. *Rep. anal. Chem.*, 1885, **5**, 128; *Chem. Ztg.*, 1885, 781.
- FILSINGER. Examination of chocolate. *Chem. Ztg. Rep.*, **13**, 309; *Chem. Centrbl.*, 1890, 130.
- FILSINGER. Modification of the ether test for cocoa butter. *Zeitsch. f. anal. Chem.*, **19**, 247; *Chem. Ztg.*, 1889, **13**, 309; Benedikt, *Analyse der Fette und Wacharten*.
- FISCHER. Constitution of theobromine. *Ann. Chem. Pharm.*, **215**, 253.
- FRÜHLING UND SCHULZ. An analysis of cocoa powder. *Correspondenz-Bl. d. Vereins analyt. Chem.*, 1880, 17.
- GALIPPE. Copper in cocoa ash. *Jour. de Pharm. et de Chim.* [5], **1**, 505.
- GLASSON. Formula of theobromine. *Ann. Chem. Pharm.*, **61**, 335.
- GÖSSMANN and SPECHT. Cocoa butter. *Ann. Chem. Pharm.*, **90**, 126.
- GRANDEAU. Analyses of cocoa husks. Dietrich u. König, *Zusammensetzung der Futtermittel*, p. 264.
- HAGER. Anilin test for the purity of cocoa butter. *Zeitsch. f. anal. Chem.*, **19**, 246; Benedikt, *Analyse der Fette und Wacharten*.
- HANAUSEK. Commercial varieties of cocoa beans. Hanausek's *Die Nahrungs- und Genussmittel aus dem Pflanzenreiche*.
- HARTWICH. Determination of starch in chocolate. *Jour. Chem. Soc.*, 1889, 192; *Chem. Ztg.*, **12**, 375.
- HEISCH. Analysis of cocoa. *Analyst*, **1**, 142.
- HEISCH. Analyses of cocoa beans. *American Chem.*, 1876, **76**, 930.
- HERBST. Examination of chocolate. *Rundschau*, 1882, 443; *Rep. d. anal. Chem.*, **2**, 236; *Zeitsch. f. anal. Chem.*, **22**, 278.
- HOLM. The harvesting and preparation of cocoa beans. *American Chem.*, **5**, 330; *Jsb. d. Chem.*, 1875, 1121.
- HUSEMANN. Solubility of theobromine. Husemann's *Pflanzenstoffe*, p. 822.
- JÜRGENSEN. Adulteration of cocoa and chocolate in Denmark. *Chem. Centralblatt*-1890 [2], **21**, 890.
- KELLER AND STRICKER. Solubility of theobromine. *Ann. Chem. Pharm.*, **118**, 151.
- KINGZETT. Composition of cocoa butter. *Phar. Jour. Trans.*, **8**, 412.
- LEGLER. A modification of Wolfram's method for the determination of theobromine. *Ber. d. chem. Ges.*, **15**, 2938; *Zeitsch. f. anal. Chem.*, **23**, 89.
- LEGLER. Microscopical examination of cocoa. *Rep. d. anal. Chem.*, **4**, 345; *Chem. Ztg.*, 1885, 105. A second article. *Ref. d. anal. Chem.*, **5**, 95; *Chem. Ztg.*, 1885, 679.
- MANSFELD. A modification of Asborth's method for the determination of starch in cocoa and chocolate. *Zeitsch. f. Nahrungsmitteluntersuch. u. Hygiene*, 1888, **1**, 2; *Chem. Ztg.*, **3**, 91.
- MANSFELD. Examination of cocoa preparations. *Zeitsch. d. allg. österr. Apotek.*, Ver., **44**, 329.
- MOSER. Analyses of cocoa husks. Dietrich und König, *Zusammensetzung der Futtermittel*.
- MULDER. Estimation of theobromine. König, *Chemie der Menschlichen Nahrungs- und Genussmittel*, 3 Aufl., Band 1, p. 1019.
- MÜNCH. Analysis of cocoa. *N. Jahrb. Pharm.*, **25**, 8; *Zeitsch. Chem.*, 1866, 191.
- MUTER. Analysis of cocoa. *Analyst*, 1879, 65.
- PAYEN. Detection of foreign starches in chocolate. *Jour. de Pharm. et de Chim.*, **41**, 367; *Zeitsch. f. anal. Chem.*, **2**, 444.

- PENNETIER. Estimation of starch in chocolate. *Jour. Pharm. Chim.* [5], 1887, **15**, No. 3.
- POHL. A method for the determination of melting points. *Wiener Academie, Berichte*, **6**, 587; *Benedikt, Analyse der Fette und Wacharten*.
- PORTELE. Analysis of cocoa husks. *Centrbl. f. Agrikulturchem.*, 1870, 304.
- REINSCH. Detection of flour in chocolate. *Fürther Gewerbe Ztg.*, 1868, 63; *Zeitsch. f. anal. Chem.*, **8**, 514.
- ROST VAN TONNINGEN. Analyses of the ash from various parts of the cocoa tree. *Rép. Chim. Appliqué*, **2**, 261; *Jadsb. d. Chem.*, 1859, 548.
- RÜDORFF. A method for the determination of melting points. *Jsb. d. Chem.*, 1872, 32.
- SALDAU. *Die Chokolade-Fabrikation*, 1881.
- SAMELSON. Adulteration of cocoa powder. *Chem. Ztg.*, 1888, **44**, 722.
- SALTSIEN. The action of iodine on the starch of cocoa. *Zeitsch. f. anal. Chem.*, **28**, 1.
- SCHAEGLER. *Die Technologie der Fette und Oele der Pflanzen- und Thierreichs*. Berlin, 1883.
- SCHMIDT. Caffeine in cocoa. *Arch. d. Pharm.*, **221**, 675; *Zeitsch. f. anal. Chem.* - **23**, 89.
- SCHROEDER. Untersuchungen über die Bestimmungen des Zuckers in den Cacao, waaren. *Zeitsch. f. angew. Chem.*, 1892, 173.
- SKALWEIT. Refractive index of cocoa butter. *Rep. d. anal. Chem.*, **6**, 181; *Zeitsch. f. anal. Chem.*, **28** 386.
- STENHOUSE. Cocoa butter. *Ann. Chem. Pharm.*, **36**, 56.
- STUTZER. Analysis of cocoa. *Russ. Zeitsch. f. Pharm.*, **21**, 724.
- STUTZER. Analysis of cocoa, partially deprived of fat. *Rep. f. anal. Chem.*, 1882, 88 and 161; *Hygiene-Bericht*, 1882-'83, **1**, 217.
- STUTZER. Neues aus der Röst-, Darr- und Trocknungs-Industrie. *Zeitsch. f. angew. Chem.*, 1891, 368.
- STUTZER, FASSBINDER, AND KLINKENBERG. Determination of nitrogen in cocoa. *Chem. Centrbl.*, 1882, 538; *Jsb. d. Chem.*, 1882, 1438.
- TRAUB. Composition of cocoa butter. *Arch. Pharm.* [3], **21**, 19.
- TRESCA. Manufacture of chocolate. *Les Mondes*, July 22, 1869.
- TREUMANN. Solubility of theobromine. *Arch. Pharm.* [3], **13**, 5; *Jsb. d. Chem.*, 1878, 872.
- TUCHEN. Dissertation über d. organ. Bestandtheile der Cacao. Göttingen, 1857.
- VALENTA. Solubility of cocoa butter in glacial acetic acid. *Dingl. polyt. Jour.*, **252**, 296; *Zeitsch. f. anal. Chem.*, **24**, 295.
- WARREN. Adulteration of chocolate. *Chem. News*, **62**, 99.
- WEIGMANN. Estimation of theobromine. *König, Chemie d. mensch. Nahrungs- u. Gennsmittel*, 3 Aufl., Band I, S. 1019.
- WEIGMANN. Microscopical examination of cocoa beans. *Rep. d. anal. Chem.*, 1885, 182.
- WIGNER. Nitrogenous constituents of cocoa beans. *Analyst*, **4**, 8.
- WOSKRESENSKY. Discovery of theobromine. *Ann. Chem. Pharm.*, **41**, 125.
- WOLFRAM. Determination of theobromine. *Jsb. d. k. chem. Centralstelle f. öff. Gesundheitspflege zu Dresden*, 1878; *Zeitsch. f. anal. Chem.*, **18**, 346.
- ZEDELER. Analysis of the ash of husked cocoa beans. *Ann. Chem. Pharm.*, **78**, 348; *Pharm. Centrbl.*, 1851, 716.
- ZIPPERER. Untersnch. ü. Cacao u. dessen Präparate, 1887.
- ZIPPERER. Estimation of theobromine. *König, Chemie der menschlichen Nahrungs- und Gennsmittel*, 3 Aufl., Band I, S. 1020.

BIBLIOGRAPHY OF METHODS FOR THE ESTIMATION OF TANNIN.

- ALLEN, A. H. Nearly all the methods worthy of detailed description are based on the precipitation of tannin by gelatin or its absorption by a gelatinous sub-

stance. Many of the methods appear very simple, but in practice offer very considerable difficulties, especially when gallic acid is present.

Commercial Organic Analysis, vol. III, Pt. 1, 109.

ALLEN, A. H. This writer states that a German commission recommends, in permanganate titrations, the addition of the solution 1 cc at a time instead of drop by drop. The results of the "1 cc method" differ considerably from those obtained by the "drop method." The latter method was employed by Neubauer and Oser in determining the reduction coefficients; hence Allen recommends it. At the reference cited, Procter points out the source of error in the "1 cc method."

Op. cit., page 116.

ALLEN, A. H. A method for tea assay based on the precipitation of tannin from a hot solution by a standard solution of lead acetate, employing ammoniacal ferricyanide in ascertaining the end reaction.

Chem. News, 29, 189; also Allen's Commercial Organic Analysis, vol. III, Pt. I, 123.

BARBIERI, J. Essentially the method of Carpené (*vide* Carpené, A., Ding. polytec. Journ., 216, 452); Ber. d. chem. Ges., 9, 78; also Zeitsch. f. anal. Chem., 16, 123.

BECKER. A volumetric method in which a solution of tannin is run into a hot solution of methyl-violet until the color is discharged. Zeitsch. f. anal. Chem., 25, 133.

BECKMAN, E. A volumetric method employing a solution of ammonio-ferrous sulphate, crystallized sodium acetate, and acetic acid for the removal of the tannin. The usual permanganate titrations are made before and after the removal of the tannin. Zeitsch. f. anal. Chem., 25, 527.

CARPENÉ, A. A volumetric method employing an ammoniacal solution of zinc acetate for the precipitation of the tannin. The precipitate is collected, washed, dissolved in dilute sulphuric acid, and titrated with permanganate of potassium. Ding. polytec. Journ., 216, 452; Zeitsch. f. anal. Chem., 15, 112.

CASALI, ADOLPHO. A volumetric method employing a standard nickel solution. A paper soaked in ferric chloride solution containing ferrous sulphate is employed as an indicator. Annali di Chim., 79, 65; also Chem. Ztg., 8, 1767; also Zeitsch. f. anal. Chem., 24, 272.

CECH. This author states that the presence of acetic acid, citric acid, tartaric acid, malic acid, cane sugar, dextrin, gum, fat, caffeine, or urea does not interfere in the estimation of tannin by permanganate, provided the solution is properly diluted. Zeitsch. f. anal. Chem., 7, 134.

COLLIN AND BENOIST. A volumetric method based on the precipitation of tannin by gelatin. A derivative of aniline added to the gelatin solution is used as an indicator. The tannate absorbs the coloring matter and the solution becomes colorless when the precipitation is complete. A standard tannin solution is employed in making up the gelatin solution. Mon. Scien., March, 1888, 364; Journ. Soc. of Dyers, 4, 62.

COMMAILLE. A volumetric method employing a standard solution of iodic acid in the presence of hydrocyanic acid. Zeitsch. f. anal. Chem., 3, 488.

CONNCLER AND VON SCHROEDER. An official report on the estimation of tannins, containing recommendations in regard to methods of analysis, strength of solutions, etc. Zeitsch. f. anal. Chem., 25, 121.

DARTON, N. H. Darton employed an ammonio-sulphate of copper solution for the precipitation of tannin. Extract the tea first with cold, then with boiling water. Treat the infusion with 25 cc dilute sulphuric acid (1 part acid to 10 parts water), filter, render filtrate slightly alkaline with ammonia, and again filter. Treat 100 cc this solution with an equal volume of ammonia sulphate of copper solution (1½ per cent copper salt), collect the precipitate. Titrate a known volume of the filtrate with permanganate for non-tannins. Wash

- the precipitate with ammonium carbonate solution, dry and weigh or ignite, moisten with nitric acid and again ignite and weigh. Weight, $\text{CuO} \times 1.034 =$ gallotannin. *Journ. Am. Chem. Soc.*, 1882, 4.
- DAVY, SIR H. This is the first method devised for the estimation of tannin, and consisted in titrating with a standard gelatin or glue solution. *Phil. Trans.*, 1803, 268.
- DURIEU, F. A method employing a standard calcium hypochlorite solution, under certain conditions, after the addition of ferric chloride. *Arch. d. Pharm.*, **22**, 523; *Zeitsch. f. anal. Chem.*, **24**, 273.
- EDER, J. M. A method for tannin of teas based on that of Fleck (*vide* Wagner's *Jsb.*, 1860, 531, or abstract in this bibliography). The tannin is precipitated from water solution, with copper acetate solution; the precipitate is collected, washed, and ignited, then moistened with nitric acid and again ignited. 1 gram $\text{CuO} = 1.3061$ grams tannin. *Zeitsch. f. anal. Chem.*, **19**, 106; *Ding. poly. Jour.*, 228, 81.
- FEHLING. An improvement of Davy's method, consisting in the addition of alum to the glue solution. *Jsb.*, Liebig and Kopp, 1853, 683; *Ding. poly. Journ.*, **130**, 53.
- FLECK. A method based on the precipitation of tannin by copper acetate. Wagner's *Jsb.*, 1860, 531.
- GAUTIER, A. A method employing carbonate of copper for the estimation of tannin in wines. *Zeit. f. anal. Chem.*, 1878, 222; *Jsb. d. Chem.*, 1878, 1088.
- GANTTER, F. A permanganate method. Ten cc of the bark solution are saturated with dilute sulphuric acid, heated to boiling point, then permanganate solution is added (3.988 grams permanganate of potassium per 1,000 cc) 1 cc at a time until the color disappears slowly; boil and then add the permanganate drop by drop. When a precipitate, not dissolved by hard boiling, forms, add 1 cc permanganate and boil until the color disappears. Add oxalic acid (7.951 grams per liter) until the precipitate dissolves and the solution becomes clear, then complete the titration with permanganate. From the total permanganate, less that required by the oxalic acid, calculate the tannin. *Zeitsch. f. angew. Chem.*, 1889, **57**, 577; *Jour. Anal. Chem.*, **4**, 83.
- GERLAND. A method based upon the precipitation of tannin by tartrate of antimony and potassium, in the presence of ammonium chloride. *Chem. News*, 1863, 54; *Zeitsch. f. anal. Chem.*, **2**, 419.
- GINTL, W. This chemist rejected Pribram's method on account of great variations in the results. *Zeit. Chem.*, 1868, 144; *Jsb. d. Chem.*, **21**, 888.
- GRASSI, E. A modification of Maumené's method. The tannin is precipitated by barium hydrate; the precipitate is washed with ammonium chloride or nitrate solution, neither of which dissolves the tannate of barium. This latter is dissolved in dilute sulphuric acid and titrated with standard permanganate. *Ber. chem. Ges.*, 1875, 257; *Jsb. d. Chem.*, 1875, 989.
- GUENEZ, E. A volumetric method employing a standard tartar emetic solution, with Poirier's green, 4 J. E., as an indicator. The tannin solution is kept at the boiling point during the titration. It is claimed that gallic acid offers no resistance in this method. *Compt. Rend.*, 1890, 532.
- HAMMER. This method employs a piece of animal skin to abstract the tannin from solution. Dr. A. B. Prescott (*Organic Analysis*, 473) says Hammer's method gives untrustworthy results. *Journ. f. prakt. Chem.*, **81**, 159; notes on this method, by Muntz and Ramspacher, *Compt. Rend.*, **79**, 380; also, *Zeitsch. f. anal. Chem.*, **13**, 462.
- HANDTKE. A method based on the reaction of ferric acetate with tannin. Standardize the iron solution by means of a tannin solution of known strength. In practice add the iron solution to the tannin solution until the precipitate settles and the liquid becomes clear. Method not applicable to coffees and teas. *Journ. f. prakt. Chem.*, **82**, 345; *Zeitsch. f. anal. Chem.*, **1**, 104.

- JOHANSON, E. A modification of Davy's method, employing 5 to 8 drops of a solution of chromic sulphate (one part in twenty-five parts water) per 100 cc of the glue solution. *Chem. News*, **50**, 176.
- JACKSON, R. This method employs lead carbonate as a precipitant of tannin. The percentage of tannin is calculated from the density of the solution before and after the precipitation. An error is possible from the partial solution of the lead by a free organic acid. *Chem. News*, **50**, 179; *Zeitsch. f. anal. Chem.*, **24**, 271.
- JEAN, F. A volumetric method based upon the fact that tannic and gallic acids combine with iodine, in the presence of sodium carbonate, in definite proportions. Filter paper covered with starch powder is used in testing for excess of iodine. The presence of gallic acid necessitates a second titration after the removal of the tannin by an appropriate precipitant. *Ber. d. chem. Ges.*, **9**, 730; *Zeitsch. f. anal. Chem.*, **16**, 123.
- JEAN, F. A colorimetric method, in which a tannin solution of known strength (1 per cent) is added to a ferric chloride solution containing free hydrochloric acid, until a piece of white paper placed under the beaker on black cloth is no longer visible. A similar titration is made with a solution of the sample to be assayed. Two titrations are necessary, one before and one after the removal of the tannin by gelatin. *Bul. Soc. chim. de Paris.*, **44**, 183; *Zeitsch. f. anal. Chem.*, **28**, 108.
- KATREINER. This chemist objects to the methods of Carpené and Barbieri, and states that the probabilities of error are great. *Zeitsch. f. anal. Chem.*, **18**, 112.
- LÖWE. In this method the infusion is evaporated with a few drops of acetic acid, and residue is then exhausted with alcohol, which dissolves the tannin only. The tannin is then estimated in the alcoholic solution. *Jsb. d. Chem.*, **18**, 743.
- LEHMAN. Titration with a glue solution containing ammonium chloride. *Jahresber. d. Chem.*, 1881, 1206; *Russ. Zeit. Pharm.*, 1881, 321.
- LÖWENTHAL, J. This method is one of the most important yet devised for tannin. It is given in considerable detail, since a modification of this method is usually employed in modern practice. See method as modified by Procter, Conneler, and Schroeder, page 890, this report.
- The method devised by Löwenthal is based upon the oxidation of tannin by permanganate of potassium. Four solutions are employed: (a) permanganate of potassium, standard solution; (b) 6 grams indigo, with 50 cc concentrated sulphuric acid per liter; (c) 25 grams white glue, dissolved in water and the solution saturated with sodium chloride, then made up to 1 liter; (d) a saturated solution of sodium chloride, with 25 cc sulphuric acid per liter. The sample is extracted in boiling water, the solution is cooled and made up to 1 liter. (1) 10 cc of this extract are diluted to 800 or 1,000 cc, 25 cc indigo solution added, and a titration is made with the permanganate. (2) 25 cc indigo solution are diluted to 800 or 1,000 cc. and titrated with permanganate. This burette reading is subtracted from that under (1) to obtain the net reading, i. e., the permanganate required for the tannin and gallic acid and other oxidizable matter in 10 cc of the extract. (3) 100 cc of the extract are now treated with 50 cc glue solution, and, after shaking, 100 cc salt solution are added. After several hours the solution is filtered. To 50 cc of the filtrate 25 cc indigo are added and another titration is made. Under these conditions, again determine the permanganate required for the indigo and deduct the burette, reading as before. In the titration under (3) a volume of the solution equal to 20 cc of the extract was taken; hence divide the net burette reading by 2. This gives the permanganate required to oxidize the substances other than tannin in 10 cc of the extract. Deducting this last amount of permanganate from that required for all the oxidizable matter in the extract, and the remainder is that required for the tannin in 10 cc extract.

The permanganate is standardized with oxalic acid. According to Neubauer, 63 grams crystallized oxalic acid correspond to 41.57 grams purified gallotannic acid. *Zeitsch. f. anal. Chem.*, **16**, 33; **20**, 91.

MACAGNO. A method based on the use of mercuric nitrate as a precipitant. *Ber. d. chem. Ges.*, **7**, 1, 360.

MAUMENÉ. A method for the estimation of tannin in wines. The tannin is precipitated by barium hydrate, and the precipitate after washing is dissolved in dilute sulphuric acid and titrated with permanganate. *Bul. Soc. Chim.* [2], **22**, 41; *Jsb. d. Chem.*, 1874, 1046.

MITTENZWEY. A method depending upon the absorption of oxygen by tannin. A portion of the substance is shaken in a graduated tube with a 30 per cent solution of potassium hydroxide. After standing twenty-four hours the tube is opened under water and the absorption noted. *Journ. f. prakt. Chem.*, **91**, 81.

MONIER E. Monier was the first to propose the use of permanganate of potassium in tannin estimations. He did not publish a definite method. *Compt. Rend.*, **46**, 577; *Ding. poly. Journ.*, **118**, 209.

MOULLADE, A. A modification of Jean's method for the estimation of tannin, employing iodine with sodium bicarbonate. Carbon bisulphide is used as an indicator. *Journ. de Pharm. et de Chim.* [5], **22**, 153.

MÜLLER, G. An improvement in Davy's method similar to that of Lehman. *Wagner's Jsb.*, 1858, 510; *Zeitsch. f. anal. Chem.*, **5**, 5.

OSTERMAYER. This chemist substitutes magenta for the rose aniline in Wagner's method. *Chem. News*, **40**, 181.

PAVESI and ROTONDI. A modification of Fleck's method, employing a solution of copper acetate with ammonium carbonate. The authors consider the precipitate to have the formula $C_{14}H_4Cu_2(NH_4)_2O_9 + H_2O$. *Gazz. Chim. Ital.*, 1874, 194; also *Ber. d. chem. Ges.*, 1874, 590.

PERSOZ, J. Persoz estimated the tannin, employing stannous chloride, by comparing the volume of the precipitate after setting aside from ten to twelve hours, with that obtained under the same conditions as to volume and time with a standard tannin solution. *Traité de l'Impression des Tissus*, **1**, 282.

PERRET, M. Perret employs an albumin solution as a precipitant of tannin, then removes the excess of albumin. The tannin is calculated from weight of the dried precipitate. *Bul. Soc. Chim. de Paris*, **41**, 32; *Zeitsch. f. anal. Chem.*, **24**, 273.

PRIBRAM. Based on the precipitation of tannin by lead acetate. The organic matter contained in the precipitate is termed "total astringent matter." *Jsb. d. Chem.*, (1866) **17**, 821; *Vierteljahresber. f. Chem.*, **15**, 520.

PROCTER, H. R. A modification of Löwenthal's method (see page 890, this report). A valuable improvement comprised in this modification is the use of powdered kaolin to facilitate the filtration after the addition of the gelatin. *Journ. Soc. Chem. Ind.*, **3**, 82.

PROCTER, H. R. The same as Hammer's method, except that hide powder is used instead of the animal skin. *Allen's Commercial Organic Analysis*, Vol. III, Pt. 1, 119.

PROCTER, H. R. A criticism of Gantter's method. Procter finds that the amount of permanganate consumed is materially influenced by the excess in which it is added and by the time of boiling. He concludes that by a rigid scheme of operation the new method might give concordant results, but at best such results could have but a comparative value. *Journ. Soc. Chem. Ind.*, **9**, 260; *Journ. Franklin Inst.*, **129**, 421.

PRUD'HOMME. A volumetric method employing a standard solution of calcium hypochlorite with an aniline color (methyl-orange) as an indicator. The color is discharged in the presence of unprecipitated tannin. *Bul. Soc. Chem.* [2], **21**, 169; *Jsb. d. Chem.*, 1874, 1034.

- RICHARDS and PALMER.** A volumetric method based upon the work of Gerland. A standard solution of tartrate of antimony and potassium is employed for the titration. A solution of acetate of ammonium is prepared for use as described below. In preparing this solution, saturate glacial acetic acid with stronger water of ammonia. Exhaust a weighed portion of the sample as usual with water, using such proportions to give approximately 0.3 to 0.9 gram tannin per 150 to 300 cc. Divide this solution into three or four aliquot parts. To one portion add the standard tartar emetic solution in slight excess, and to a second add less of the solution than is required to precipitate the tannin. To each portion add 1 cc ammonium acetate solution per 25 cc taken. After allowing time for the precipitate to settle, test a drop of the clear liquid on a porcelain plate with a drop of sodium thiosulphate. An excess of antimony is indicated by an orange-yellow precipitate. The tests of the first two aliquot parts, more tartar emetic being added to the second if necessary, indicate approximately the volume of the antimony solution required to precipitate the tannin, and the remaining aliquot part or parts serve to determine this volume with accuracy. The volume of the standard solution taken $\times .01 =$ tannin as digallie acid. Ammonium acetate acts similarly to the chloride in Gerland's method and prevents the precipitation of the gallic acid. *Am. Jour. Sci.*, **16**, 196 and 361; *Allen's Commercial Organic Analysis*, Vol. III, Pt. 1, 123; *Preseott's Organic Analysis*, 471.
- RISLER-BEUNAT, H.** A method based on the precipitation with stannous chloride. Extract 10 grams of tea thirty minutes with 500 cc water; wash the residue with about 500 cc boiling water; cool and complete the volume to 1 liter. Precipitate 100 cc of this extract with 100 cc stannous chloride solution containing 18 grams of this salt and 2 grams ammonium chloride per liter. Collect, wash, dry, and weigh the precipitate, then ignite with ammonium nitrate and again weigh. Calculate the resulting SnO_2 to SnO , and deduct this from the weight of the precipitate; the remainder is the weight of the tannin. *Zeitsch. f. anal. Chem.*, **2**, 287.
- SCHREINER, L.** An apparatus for the absorption of tannin by hide powder. This apparatus is so arranged that the first portions of the solution, which always dissolve some albumin from the powder, collect in a small bulb placed below by reason of their greater density. *Der Gerber*, **14**, 244.
- SCHULZE, F.** A volumetric method employing a gelatin solution standardized by means of pure tannin dissolved in a concentrated solution of chloride of ammonium. The gelatine is also dissolved in a concentrated solution of chloride of ammonium. In the titration, the standard solution is added until the precipitate formed will settle within half a minute, leaving the supernatant liquid clear. *Zeitsch. f. anal. Chem.*, **5**, 455.
- SCHMIDT, E.** A method employing a standard solution of lead acetate in dilute alcohol. A measured volume of the solution to be assayed is warmed to 60°C . and titrated with the lead solution, using potassium iodide as an indicator. *Bul. Soc. Chem.* [2], **21**, 256; also, *Jsb. d. Chem.*, 1874, 1035.
- SACKNER.** Sackner commenting on various methods for tannin considers that of Löwenthal the most accurate. *Wagner's Jsb.*, 1861, 625.
- SIMPKIN, S. J.** A volumetric method based on the oxidation of tannin by permanganate, and its precipitation by ammonium sulphate of copper. *Chem. News*, **32**, 11; also *Jsb. d. Chem.*
- TERREIL.** An improvement of Mittenzwey's method. Terreil states that 0.1 gram pure tannic acid will absorb 20 cc oxygen in twenty-four hours. *Ber. d. chem. Ges.*, **5**, 362; also, *Compt. Rend.*, **78**, 690; also, *Zeitsch. f. anal. Chem.*, **13**, 243.
- VILLON, M.** A method involving extensive calculations based on the specific gravities of the original solution, the precipitant, and the solution after filtering off the precipitate. Lead acetate with sodium acetate is the precipitant em-

- ployed. In the original article several of the constants for different tannins are given. *Chem. News*, **55**, 168.
- WAGNER. A volumetric method, employing a standard solution of cinchonine colored with rosaniline and acidulated with sulphuric acid. *Zeitsch. f. anal. Chem.*, **5**, 1.
- WARRINGTON. A volumetric method depending upon the direct precipitation of tannin by gelatin. A filter tube for testing is prepared by loosely introducing a piece of sponge into the end of a small tube. In filtering off a test sample immerse the sponge in the solution, and the clear liquid will rise into the tube. Test this filtrate with gelatin. *Phil. Mag.*, **31**, 150; also *Journ. Sci.*, 1848, **6**, 112.
- WEISS. Illustrations of apparatus for the continuous extraction of barks, etc. *Zeitsch. f. anal. Chem.*, **28**, 114.
- WILDENSTEIN, R. A colorimetric method, employing strips of filter paper of various shades and comparing them with strips of similar paper after dipping them into the infusion which had been previously treated with an iron salt. *Zeitsch. f. anal. Chem.*, **2**, 137.
- WOLFF, E. A method for the estimation of tannin in teas, employing copper acetate as a precipitant. The precipitate, after washing with boiling water, is dried and ignited, moistening with nitric acid. Weight obtained $\times 1.304 =$ gallo-tannin. *Krit. Blaett.*, **44**, 167; *Zeitsch. f. anal. Chem.*, **1**, 103.

ADDITIONAL REFERENCES TO THE LITERATURE OF TANNINS.

- BÜCHNER. Tannin estimations, criticisms on. *Jsb. d. Chem.*, **18**, 862 (1867); *Dingl. polyt. Jour.*, **184**, 250, 330.
- CARLES, P. Tannin, gelatin solution for estimating. *Chem. News*, **51**, 297; *Zeitsch. f. anal. Chem.*, **24**, 272 (1885).
- ETTI, C. Tannic acid, composition of. *Chem. Centrbl.*, 1880, 204; *Jsb. d. Chem.*, 1880, 898.
- FRANK. Tannins, estimation of. *Pogg. Ann.*, **43**, 161.
- FRAAS. Tannin, improvement on Müller's method. *Zeitsch. f. anal. Chem.*, **5**, 5; *Ergebnisse landwirthsch. und agri. chem. Versuche*, Munich, (1861), **3**, 41.
- FREDA, P. Tannic acid, constitution of. *Gazz. Chim. ital.* **8**, 9.
- GAUHE, F. Tannin, comparison of various methods. *Zeitsch. f. anal. Chem.*, **3**, 122.
- GRIESSMAYER, V. Tannin, detection of. *Zeitsch. f. anal. Chem.*, 1872, 43; *Jsb. d. Chem.*, 1872, 903.
- HALLWACHS, W. Tannin, criticisms on methods. *Zeitsch. f. anal. Chem.*, **5**, 231; *N. Jhrb. f. Pharm.*, Bd. **25**, 2, 68.
- HILL, ALFRED. Tannin, estimation of, in tea. *Analyst*, **6**, 95.
- HINSDALE, S. J. Tannin, colorimetric method for. *Chem. News*, **62**, 19.
- HLASIWETZ and MALIN. Tannin, note on error in estimating. *Dingl. polyt. Jour.*, **229**, 87; *Wien. Akad. Ber.*, 1867, 55, 19.
- HOOPER, DAVID. Tannin in Indian and Ceylon teas, on the. *Chem. News*, **60**, (1870), 311.
- JAHODA, RUDOFF. The variability of the percentage of tannin in some tanning materials. *Zeitsch. f. angew. Chem.* (1891), **4**, 104.
- KOKOSINSKI, E. Tannin, determination of in hops. *Ztschr. f. d. ges. Brauw.* (1890), **13**, 571; *Chem. Ztg. Rep.*, **15**, 3, 27.
- MUNTZ and RAMSPACHER. Tannin, estimation of. *Zeitsch. f. anal. Chem.*, **13**, 462; *Compt. Rend.*, **79**, 380.
- RENISCH, H. Tannin of the sumach. *N. Jhrb. d. Pharm.*, **25**, 82; *Zeitsch. Chem.*, 1866, 220; *Jsb. d. Chem.*, 1866, 695.
- SCHIFF, H. Tannic acid, constitution of. *Ber. d. chem. Ges.*, 1871, 231, 967; *Bull. soc. chim.* [2], **16**, 198.

- SCHIFF. Tannic acid, constitution and formula of. *Ber. d. chem. Ges.*, 1872, 291, 438; *Bull. soc. chim.* [2], **18**, 23.
- SCHIFF, H. Tannic acid, constitution of. *Berichte*, 1882, 2582; *Jsb. d. Chem.*, 1882, 915.
- SCHMIDT, E. Tannin, modification of Pribram's method. *Jsb. d. Chem.*, 1874, 1035; *Bull. soc. chim.* [2], **21**, 256.
- HENHOUSE. Tannin, kinds of. *Dingler's polyt. Jour.*, **165**, 150.
- SONNENSCHN. Tannin, action of, on Fehling's solution. *Analyst.*, **10**, 205.
- SIMAND, F. Gelatin, substitute for, in Löwenthal's process. *Analyst.*, **3**, 125.
- TRIMBLE, HENRY. Tannins, the. J. B. Lippincott Company, Philadelphia.
- ULBRICHT, R. Tannin, notes on Löwenthal's method. *Zeitsch. f. anal. Chem.*, **25**, 252; *Ber. d. chem. Gesell. zu Berlin*, **18**, 1116.
- VOGL, A. Tannin in the roots of plants. *Wien. Acad. Ber.*, **53**, 156; *Jsb., d. chem.* 1866, 690.
- WATTS. Tannin, criticisms on Fleck's method. *Jsb. d. Chem.* **19**, 887 (1868); *Pharm. J. Trans.* [2], **10**, 223.

CAFFETANNIC ACID.

- HLASIWETZ. Caffetannic acid, derivatives of. *Ann. Chem. Pharm.*, **142**, 219; *Bull. soc. chim.* [2], **9**, 122; *J. pr. Chem.*, **101**, 97.
- KRUG, W. H. The separation and estimation of caffetannic acid. Original article in this report, page 908.
- LIEBICH. Caffetannic acid, action of KOH on. *Jsb. d. Chem.*, 1849, 324; *Ann. Chem. Pharm.*, LXXI, 57; *Pharm. Centralhalle*, 1849, 855.

BIBLIOGRAPHY OF METHODS FOR THE ESTIMATION OF CAFFEINE.

- ALLEN, A. H. A thin dough is made of the powdered tea, caustic lime, and water and the whole allowed to stand several hours. It is now dried on the water-bath, extracted with benzole, the benzole evaporated and the caffeine recrystallized from water. *Chem. News*, **29**, 129, 140, 167, 189, 221; **30**, 2.
- BEEKMAN. The material is extracted with water containing a little sodium carbonate. The aqueous extract is mixed with an alum solution, the excess of alum precipitated by sodium carbonate and the precipitate filtered off. The filtrate and washings are evaporated to a syrupy consistence, made alkaline with sodium carbonate, and the caffeine extracted with ether. *Zeitsch. f. anal. Chem.*, **4**, 206.
- BLYTH, A. W. This method is based on the ease with which caffeine sublimes. The author proceeds as follows: From one to two grams of finely powdered tea are boiled with water for an hour in a flask connected with an upright condenser. The whole is transferred to a porcelain dish, about its weight of magnesia added, and the mass evaporated nearly to dryness. It is now transferred to an iron subliming plate and covered with a tarred glass funnel, the edge of which is accurately ground and which has a neck several inches long. The mixture is first heated to 110° C., then slowly raised to 200° C. and finally to 220° C. The caffeine sublimate is collected and weighed. *Analyst*, **2**, 39; also in *Foods: Their Composition and Analysis*, A. W. Blyth.
- CANADA, DOMINION OF. Ten grains of the finely ground sample are exhausted by repeated boiling with water, filtered, the filtrate made up to a definite volume, and an aliquot part precipitated with basic acetate of lead. The filtrate without any concentration is shaken with successive portions of chloroform. The chloroform is removed by distillation and the caffeine is weighed. Report on Adulteration of Food, Supplement III, Report of the Dept. of Inland Revenue, 1886.
- CAZENEUVE-CAILLON. Add to the tea four times its weight of boiling water and when the leaves become soft add an equal weight of slaked lime. Dry and extract

with chloroform. Distill off the chloroform and dissolve the residue in boiling water. Filter and wash with boiling water. Evaporate the filtrate and washings carefully on the water bath to obtain the caffeine. *Zeitsch. f. anal. Chem.*, **17**, 321.

CLAUS, C. The material is extracted with ether and the extract evaporated to one-third its original volume. This is now mixed with one-tenth of its volume of very dilute sulphuric acid, shaken in a separator, and the acid solution run off, successive portions of dilute acid being added until the ether is no longer bitter. To the united acid solutions magnesium oxide in excess is added, its whole mass dried, pulverized, and extracted with ether. The ethereal solution is evaporated and the resulting caffeine weighed. *Zeitsch. f. anal. Chem.*, **4**, 205.

COMMAILLE, A. Five grams of the powdered tea are mixed with 1 gram of calcined magnesia, dried twenty-four hours, and the caffeine extracted with three successive portions of chloroform. The chloroform extract is evaporated three times with boiling water. The aqueous extract is evaporated and the caffeine dried and weighed. *Ber. d. chem. Ges.*, **8**, 2, 1590.

EDER, J. M. Ten grams of tea are dried, powdered, and mixed with 3 grams anhydrous sodium carbonate and 3 grams lead oxide. The mixture is moistened with 10 grams of water, dried, powdered, and extracted with chloroform. The chloroform is distilled off and the caffeine weighed. *Zeitsch. f. anal. Chem.*, **19**, 371.

HEIJNSIUS, H. Separate the caffeine from tea by means of a subliming apparatus similar to that employed for benzoic acid. *J. f. prakt. Chem.*, **49**, 317; *Jsb. d. Chem.*, 1849, 382; *Pharm. Centralhalle*, 1850, 73.

IWANOW. Extract with dilute sulphuric acid; add an excess of magnesia to the acid extract; evaporate and extract with ether. This method gives results uniformly lower than any other method. *Zeitsch. f. anal. Chem.*, **4**, 207.

KREMEL, A. Estimation of caffeine in guarana. Ten grams of guarana are mixed with 100 cc 25 per cent alcohol in a flask and the whole weighed. After digesting for one to two hours on the waterbath, the loss in weight is made up with similar alcohol and 50 cc of the solution is (= 5 grams guarana) filtered off, mixed with calcium hydrate and dried. The finely powdered residue is extracted with chloroform, the chloroform distilled off, and the caffeine dried at 100° and weighed. *Arch. Pharm.* [3], **26**, 318; *Pharm. Post*, **21**, 101.

LIEVENTHAL, E. Extract the powdered tea with chloroform, filter, wash the residue with chloroform. Distill off the chloroform, extract the residue several times with boiling water, filter, and evaporate. The residue consists of caffeine. *Zeitsch. f. anal. Chem.*, **12**, 104 (*orig. Pharm. Zeit. f. Russland*, **11**, 369).

LÖSCH, A. Ten to 20 grams of tea are boiled twice with water, filtered, and the residue washed with boiling water until the filtrate runs colorless. The filtrate is mixed with magnesia equal to $1\frac{1}{2}$ times the weight of tea taken, and then evaporated to dryness on the waterbath. The residue is finely powdered and extracted with boiling chloroform. The chloroform is evaporated in a tared beaker, and the caffeine obtained dried at 100° and weighed. *Arch. Pharm.* [3], **25**, 497; *Jour. Chem. Soc.*, October, 1887, 1002.

MARKOWNIKOFF, W. Ten grams of tea are extracted with 500 cc of water, after having been mixed with 30 grams of magnesia. The extract is filtered, the filter being washed with hot water and the filtrate and washings mixed with a little magnesia and sand, then evaporated to dryness. The residue is extracted with hot benzole, the benzole distilled off, and the caffeine weighed. *Ber. d. chem. Ges.*, **9**, G1313.

PAUL, B. H. Author states that he has proved by experiment that chloroform can not be substituted for alcohol in determining caffeine in tea. By chloroform

he obtained 1.3 per cent caffeine from a tea known to contain 3.78 per cent. Boiling alcohol was then used and the rest of the caffeine was obtained. Experiments were made to determine whether caustic lime decomposed caffeine. By experiment he determined that caustic lime does not decompose caffeine under the conditions obtaining in analysis, but that lime exerts a restraining influence on the extractive power with chloroform. Complete extraction of caffeine was obtained with boiling chloroform when magnesia was used. *Pharm. J. Trans.*, March 1891, 882.

PAUL and COWNLEY. Five grams of powdered tea are moistened with hot water, well mixed with 1 gram of calcium hydrate, and the whole dried on the water bath. The dry residue is then transferred to a small percolating apparatus and extracted with strong alcohol. The clear liquor is evaporated to remove the alcohol and the remaining water solution, which amounts to about 50 cc, is mixed with dilute sulphuric acid which separates traces of lime and partially decolorizes the liquid. After filtering it is transferred to a separator and well shaken with chloroform, about 200 cc, in five to six successive portions. The united chloroform solutions are then placed in a separator and shaken with very dilute sodium hydrate. This renders the caffeine quite colorless. The chloroform is then distilled off and the caffeine weighed. *Phar. J. Trans.*, [3], 908, 417.

PATROUILLARD, C. Fifteen grams tea are extracted with boiling water until completely exhausted. The liquid is filtered, evaporated to the consistence of an extract, mixed with 2 grams calcined magnesia and 5 grams powdered glass, and dried. The residue is ground to powder, extracted three or four times with 60 cc of ether and this solvent then distilled off. The crystals of caffeine obtained are redissolved in chloroform and recrystallized. *Chem. Ztg.* No. 9, Feb. 26, 1880; *Chem. News*, 42, 259.

PELIGOT. Make an aqueous extract of the tea, precipitate with lead acetate, add ammonium hydrate, and heat. Filter, pass hydrogen sulphide into the liquid, and filter again. Evaporate to a proper consistency and allow the caffeine to crystallize. This chemist determined the caffeine in exhausted tea leaves by means of a standard tannin solution, but does not give the details of his method. *Zeitsch f. anal. Chem.*, 4, 207.

SCHWARZ, M. H. Exhaust the tea with boiling acidulated water, neutralize with lime, evaporate to dryness, and exhaust the residue with ether. Evaporate collect, and weigh the caffeine. *Bull. Soc. Chim. de Paris* Aug. 5, 1876, 3; *Chem. News*, 34, 94.

SMITH, E. D. Coarsely powdered coffee (0.65 gram) is mixed with 0.13 gram magnesia, boiled 15 minutes with 150 cc water, filtered, and the filtrate made up to 300 cc by percolation. The filtrate is evaporated to 20 cc and the residue treated with 120 cc strong alcohol, transferred to a filter, and washed with alcohol. The alcohol is then drawn off and the residue dissolved by gradual addition of small quantities of water. This water solution is extracted three times with 25 cc chloroform. The chloroform is distilled off and the caffeine dried and weighed. *Chem. Centrbl.* 1887, 1270, 1271; *Zeit. oesterreich. Apoth.*, Vcr., 41, 359.

SNYDER, HARRY. Author states that the albuminoids of tea are almost entirely insoluble in hot water. He extracts the powdered sample with four or five portions of hot water, collects the residue on a filter (*S. & S. No.* 589), and transfers filter and contents to a digestion flask, and determines the nitrogen by the Kjeldahl method. Total nitrogen in the sample (as determined by Kjeldahl method for alkaloids), less nitrogen of the insoluble portion, gives the alkaloidal nitrogen from which the caffeine may be determined. See Kozai's article on teas, which gives the distribution of the nitrogen, page 879, *Journ. Anal. Chem.*, 4, 443.

STAILLSCHMIDT. Extract the tea six hours with water; precipitate with basic lead acetate; decant and wash the precipitate by decantation. Remove excess of lead by H_2S ; filter off the PbS and evaporate the filtrate to a sirup. Extract this with hot benzole, which on evaporation will yield the caffeine. Poggendorf's *Annalen*, **112**, 441; *Chem. centrbl.*, 1861, 396.

THOMPSON. Exhaust the tea by boiling with water, remove the tannin by means of lead acetate, and free the filtrate from lead by means of hydrogen sulphide. The lead sulphide is filtered off and the caffeine precipitated in the filtrate by means of potassium carbonate. The caffeine is crystallized from an alcoholic solution to purify it. *Zeitsch. f. anal. Chem.*, 1872, 203; *Jsb. d. Chem.*, 1872, 924.

VITÉ, F. The author has made a critical study of the methods for the estimation of caffeine in teas. He adopted a modification of Hilger's method. Extract 5 grams tea, 3 times, one hour each, with successive portions water of about 300 cc.; evaporate the united extracts to one-fourth the original volume, and while still hot add fresh lead hydrate and coarse sand. Dry on water bath and extract with ehloroform. Collect the caffeine as usual and dry at a temperature not exceeding $100^{\circ}C$.—*Mitth. Lab. ang. Chem. Erlangen*, **3**, 131; *Chem. Centrbl.* (1890), **2**, 274; *Journ. Chem. Soc.* **140**, 372.

WAAGE. This chemist prefers Mulder's method modified as follows: Ten grams tea are exhausted by boiling four times with water, the exhaustion requiring about three hours. The decoction should amount to about two liters. It is evaporated to a syrupy consistence after filtering. The residue is mixed with 2 grams magnesia and 5 grams pure sand, dried, extracted in a Soxhlet apparatus with anhydrous chloroform, and this extract carefully evaporated. The caffeine is purified by dissolving in water, filtering, evaporating to dryness, drying two hours at $100^{\circ}C$., and weighing.—*Archiv d. Pharm*, 225, 443; *Zeitsch. f. anal. Chem.*, **28**, 257.

WEYRICH. This author has examined various methods of caffeine estimation and reports as follows:

Peligot's method is very unreliable and must be rejected, as the titration with tannin must of course be very uncertain and the tannin besides precipitates many other substances.

Zöller's method gives a very impure caffeine, and the long heating with a concentrated acid may cause decomposition of caffeine. The method is also quite complicated.

Löwenthal's method is uncertain, as the chloroform is unable to penetrate the tea sufficiently to dissolve all the thein. Besides it dissolves other substances.

The author finally recommends Mulder's method as being simple and accurate. Themagnesia in this method must be very finely pulverized, as the ether extraction may otherwise be incomplete. The mass previous to extraction must be pulverized in a warm mortar. *Zeitsch. f. anal. Chem.*, **12**, 104.

ZÖLLER. Extract the powdered leaves with quite concentrated sulphuric acid, dilute the extract with water, neutralize with lead hydrate, and evaporate to dryness. Extract the residue with 85 per cent alcohol, filter, and evaporate to dryness. Extract the caffeine from the residue by means of ether, distill off the ether, and weigh the caffeine. *Zeitsch. f. anal. Chem.*, **12**, 106.

ADDITIONAL REFERENCES TO THE LITERATURE OF CAFFEINE.

AUBERT. Caffein, per cent of in coffee. *Dingl. Polyt. Jour.*, **206**, 500; *Jsb. d. Chem.*, 1872, 805.

ATTFIELD, J. Caffeine in cola nuts. *Jsb. d. Chem.*, 1865, 632. *Pharm. J. Trans.* [2] **6**, 457.

BÖTTGER, R. Caffeine, detection of. *Zeitsch. f. anal. Chem.*, 1873, 442; *Jsb. d. Chem.*, 1873, 960.

- BÖTTGER, R. Caffein, reaction of. *Pol. Notizblätter*, 1873, 257; *Zeitsch. f. anal. Chem.* (1873), **12**, 442.
- Caffeine, influence of, on digestion. *Journ. Chem. Soc.*, 1889, 534.
- Caffeine oxalate. *Journ. Chem. Soc.*, 1889, 1018.
- Caffeine, oxidation of, with ozone. *Journ. Chem. Soc.*, 1889, 1017.
- Caffeine in coffee. *Pharm. Transactions*, 1887, 565.
- Caffeine, synthesis of. *Chem. News*, 1889, June 28, 305.
- COMMAILLE, A. Caffeine, estimation of in coffee. *Compt. Rend.*, **81**, 817-819.
- FISHER, E. Caffeine, derivatives of. *Jsb. d. Chem.*, 1881, 902; *Ber. d. chem. Ges.*, 1881, 637, 1905.
- FISHER, E. Caffeine, constitution and derivatives. *Ber. d. chem. Ges.*, 1882, **29**, 453; *Monit. scien.* [3], **12**, 381, 433.
- GRÈENE, F. C. Caffeine, estimation of, in Guarana. *Phar. J. Trans.* [3], **8**, 87.
- GROSSCHOPFF, C. Caffeine, preparation of. *Jsb. d. Chem.*, 1866, 470; *Arch. d. Pharm.* [2], **128**, 206.
- HECKEL and SCHLAGDENHAUFFEN. Caffeine in cola nnts. *Jsb. d. Chem.*, 1882, 1162; *Compt. Rend.*, **94**, 802.
- HILGER. Caffeine, determination of. *Zeitsch. f. anal. Chem.*, **28**, 257 (orig. *Arch. d. Pharm.*, **223**, 828).
- HINTERBERGER. Caffeine, compound of, with mercuric chloride. *Ann. Ch. Pharm.*, **78**, 201; *Jsb.*, 1851, 474.
- KOHL and SWOBODA. Caffeine, compound, with mercuric cyanide. *Wien. Acad. Ber.*, **9**, 252; *Ann. Chem. Pharm.*, **83**, 339; *Jsb. d. Chem.*, 1852, 549.
- LAFFAN, A. H. Caffeine, solubility in alcohol. *Phar. J. Trans.* [3], **11**, 902; *Jsb. d. Chem.*, 1881, 902.
- LEHMANN, J. Caffein, physiological action of. *Jsb. d. Chem.*, 1853, 751; *Ann. Chem. Pharm.*, **87**, 205, 275; *J. pr. Chem.*, **62**, 104.
- LOESCH. Caffeine, estimation of. *Chem. News*, **56**, 47.
- LUCHIN. Caffeine, reactions of. *L'orosi*, **8**, 110; *Arch. d. Pharm.* [3] **23**, 684; *Zeitsch. f. anal. Chem.*, **25**, 565.
- MALY and HINTEREGGER. Caffeine, constitution and derivatives. *Monatsh. f. Chem.*, 1882, 85.
- PAUL and COWNLEY. Caffeine, content of, in teas. *Centrbl.*, 1890, **2**, 491.
- PAUL and COWNLEY. Caffeine, estimation of, in tea. *Journ. Chem. Soc.*, **106**, 540.
- PAYEN. Caffeine cafeeo-tannate in coffee. *Ann. chim. phys.* [3], **26**, 108; *Jsb. d. Chem.*, 1849, 486.
- PELLETIER. Caffeine, isolation of pure. *Jour. Pharm.* [2], **12**, 229.
- PETRIK. Caffeine, per cent of, in tea. *Dingl. polyt. Jour.*, **218**, 220.
- PFAFF. Caffein, spec. grav. of. *Ann. Chem. Pharm.* **1**, 17.
- POLACCI, E. Caffeine, preparation of. *Jsb. d. Chem.*, 1857, 412; *Cimento*, **5**, 396.
- PUCCELLI, P. Caffeine, preparation from tea and coffee. *Arch. d. Pharm.* [2], **84**, 198.
- ROBIQUET, CAVENTON, and GAROT. Caffeine, isolation of pure. *Jour. Pharm.* [2], **2**, 234.
- ROCHLEDER. Caffeine, decomposition of. *Ann. Chem. Pharm.*, **69**, 120; *Jsb. d. Chem.*, 1849, 382.
- ROCHLEDER. Caffeine, decomposition of, by chlorine. *Wien. Acad. Ber.*, 1850, 96; *Pharm. Centrbl.*, 1851, 41; *Jour. pr. Chem.*, **51**, 398.
- ROSENGARTEN and STRECKER. Caffeine, formula of. *Ann. Chem. Pharm.*, **157**, 1; *Bull. Soc. Chim.* [2], **15**, 66.
- RUNGE. Caffeine, discovery of. *Schweigg. Jour. Chem. Phys.*, **31**, 308.
- SCHMIDT, E. Caffeine, literature of. *Ber. d. chem. Ges.*, **14**, 814.
- SCHMIDT and BIEDEMANN. Caffeine, derivatives of. *Ber. d. chem. Ges.*, 1881, 813.
- SCHULTZEN, O. Caffeine, decomposition products of. *Bull. soc. chim.* [2], **9**, 239; *Jsb. d. Chem.*, 1867, 516.

- SCHWARZENBACH. Caffeine, reaction of. *Chem. Centralbl.*, 1861, 989; *Zeitsch. f. anal. Chem.* **1**, 229.
- SPENCER, G. L. Estimation of caffeine. *Jour. Anal. Chem.*, **4**, October, 1890. See this report, page 889.
- STRAUCH, A. Caffeine, properties of. *Jsb. d. Chem.*, 1867, 808, 770. *Zeitsch. f. anal. Chem.*, **6**, 503.
- STRECKER. Caffeine, preparation of, from Theobromine. *Ann. Chem. Phar.*, **108**, 151; *Compt. Rend.*, **52**, 1210, 1268.
- TIEMANN, F. Caffeic and hydrocaffeic acids. *Ber. d. chem. Ges.*, 1878, 659.
- TILDEN, W. A. Caffein, iodine derivatives of. *Ann. Chim. Phys.* [4] **5**, 487; *Chem. Soc. Jour.* [2], **3**, 99; *Jour. pr. Chem.*, **96**, 371; **98**, 245.
- VERSMANN, H. J. Caffeine, preparation from coffee. *Arch. d. Pharm.* [2], **68**, 148; *Pharm. Centrbl.*, 1851, 889; *Jsb. d. Chem.*, 1851, 474.

BIBLIOGRAPHY OF THE LITERATURE OF CHICORY.

- ANDERSON, Th. Chicory, analysis of ash of. *Pharm. Centrbl.*, 1853, 525.
- CHEVALLIER. Chicory, method of detecting adulterations of. *J. Pharm.* [3], **16**, 50; *Dingl. polyt. Journ.*, **102**, 387.
- DRAPER, C. Chicory, detection of. *Philos. Magaz.*, **38** (No. 228), 104; *Zeitsch. f. anal. Chem.*, **7**, 388.
- FRANZ, A. Chicory, detection of. *Arch. Pharm.* [3], **8**, 298; *Jsb. d. Chem.*, 1876, 1021.
- HIEPE. Chicory, detection of. *Monit. Scient.* [3], **10**, 1339; *Jsb. d. Chem.*, 1880, 1222.
- HUSSON, C. Chicory, detection of. *Ann. Chim. Phys.* [5], **21**, 419-426; *Jour. of the Chem. Soc.*, **36**, 558.
- KRAUCH, C. Chicory and cereals, detection of. *Ber. d. chem. Ges.*, 1878, 277; *Jsb. d. Chem.*, 1878, 1163.
- KRAUCH, C. Chicory, detection of. *Biedermann's Centrbl.*, July, 1878.
- LEEBOY, J. R. Chicory, detection of. *Chem. News*, **30**, 243.
- LEEBOY, J. R. Chicory, colorimetric determination. *Chem. News*, **30**, 243.
- MAYER, A. Chicory, analyses of. *Biedermann's Centrbl.*, 1885, 828; *J. Chem. Soc.*, 1886, **50**, 388.
- MARQUIS. Chicory, adulteration of. *J. Pharm.* [3], **24**, 210; *Jsb. ii. d. Fort. der Chem.*, 1853, 752.
- PADÈ, L. Chicory, detection of. *Bull. Soc. Chim. de Paris*, 1887, **47**, 501; *Jsb. d. Chem.*, 1887, 258.
- PETERMANN, A. Chicory, analyses of. *Biedermann's Centrbl.* 1883, 843; *Jour. Chem. Soc.* **46**, 648.
- POLYTECHNISCHE GES. ZU BERLIN. Chicory, detection of. *Dingl. polyt. Journ.*, **211**, 78; *Jsb. d. Chem.*, 1874, 1043.
- PRUNIER. Chicory, detection of. *J. Pharm. Chem.* [5], **1**, 222; *Jour. Chem. Soc.*, 1880, 514.
- RIMMINGTON, F. M. Chicory, detection of, in coffee. *Analyst*, **6**, 2. *Pharm. J. Trans.* [3], **11**, 529; *Jsb. d. Chem.*, 1881, 1215.
- SMITH, ALBERT. Chicory, detection of. *Chem. News*, **34**, 283.
- SCHULZ, HUGO. Chicory, ash of, analysis of. *Z. des Ver. f. Rübenzuckerindustrie*, 1866, 435.
- WITTSTEIN, G. C. Chicory, detection of. *Am. Chemist*, **6**, 220.

APPENDIX B.

UNITED STATES TEA ADULTERATION LAW.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That from and after the passage of this act it shall be unlawful for any person or persons or corporation to import or bring into the United States any merchandise for sale as tea, adulterated with spurious leaf or with exhausted leaves, or which contains so great an admixture of chemicals or other deleterious substances as to make it unfit for use; and the importation of all such merchandise is hereby prohibited.

SEC. 2. That on making entry at the custom-house of all tea or merchandise described as tea imported into the United States, the importer or consignee shall give a bond to the collector of the port that such merchandise shall not be removed from warehouse until released by the custom-house authorities, who shall examine it with reference to its purity and fitness for consumption; and that for the purpose of such examination samples of each line in every invoice shall be submitted by the importer or consignee to the examiner, with his written statement that such samples represent the true quality of each and every part of the invoice, and accord with the specification therein contained; and in case the examiner has reason to believe that such samples do not represent the true quality of the invoice, he shall make such further examination of the tea represented by the invoice, or any part thereof, as shall be necessary: *Provided*, That such further examination of such tea shall be made within three days after entry thereof has been made at the custom-house: *And provided further*, That the bond above required shall also be conditioned for the payment of all custom-house charges which may attach to such merchandise prior to being released or destroyed (as the case may be) under the provisions of this act.

SEC. 3. That if, after an examination, as provided in section 2, the tea is found by the examiner not to come within the prohibition of this act, a permit shall at once be granted to the importer or consignee declaring the tea free from the control of the custom authorities; but if on examination such tea, or merchandise described as tea, is found, in the opinion of the examiner, to come within the prohibitions of this act, the importer or consignee shall be immediately notified, and the tea, or merchandise described as tea, so returned shall not be released by the custom-house, unless on reëxamination called for by the importer or consignee the return of the examiner shall be found erroneous: *Provided*, That should a portion of the invoice be passed by the examiner, a permit shall be granted for that portion, and the remainder held for further examination, as provided in section 4.

SEC. 4. That in case of any dispute between the importer or consignee and the examiner, the matter in dispute shall be referred for arbitration to a committee of three experts, one to be appointed by the collector, one by the importer, and the two to choose a third, and their decision shall be final; and if, upon such final reëxamination, the tea shall be found to come within the prohibitions of this act, the importer or consignee shall give a bond, with securities satisfactory to the collector, to export such tea, or merchandise described as tea, out of the limits of the United States

within a period of six months after such final reëxamination; but if the same shall not have been exported within the time specified, the collector, at the expiration of that time, shall cause the same to be destroyed.

SEC. 5. That the examination and appraisement herein provided for shall be made by a duly qualified appraiser of the port at which said tea is entered, and when entered at ports where there are no appraisers, such examination and appraisement shall be made by the revenue officers to whom is committed the collection of duties, unless the Secretary of the Treasury shall otherwise direct.

SEC. 6. That leaves to which the term "exhausted" is applied in this act shall mean and include any tea which has been deprived of its proper quality, strength, or virtue by steeping, infusion, decoction, or other means.

SEC. 7. That teas actually on shipboard for shipment to the United States at the time of the passage of this act shall not be subject to the prohibition thereof.

SEC. 8. That the Secretary of the Treasury shall have the power to enforce the provisions of this act by appropriate regulations.

Approved, March 2, 1883.

APPENDIX C.

ABSTRACTS FROM THE ITALIAN LAW (AUGUST, 1890) RELATING TO THE ADULTERATION OF FOODS.

[Translated from *Revue Internationale de Falsifications*, 15 Juin, 1891.]

XXI.—COFFEE.

ART. 155. It is forbidden to apply the name "coffee," or to sell under this name any substance, whether in the form of grains or powder, which does not consist exclusively of the product of the plant *Coffea Arabica*.

ART. 156. Under the following conditions, it is forbidden to sell genuine coffee beans:

- (a) When colored by means of objectionable or injurious substances;
- (b) When roasted and ground with spent coffee ("coffee grounds") or with foreign substances.

ART. 157. Coffee substitutes and mixtures must not contain any harmful substance, and can only be sold under a name indicating the nature and name of the substances entering into the preparation, and must never be sold under the name of genuine coffee. These brands or marks must be entered on the books of the dealer and upon the invoices, bills of lading, etc.

XXII.—TEA.

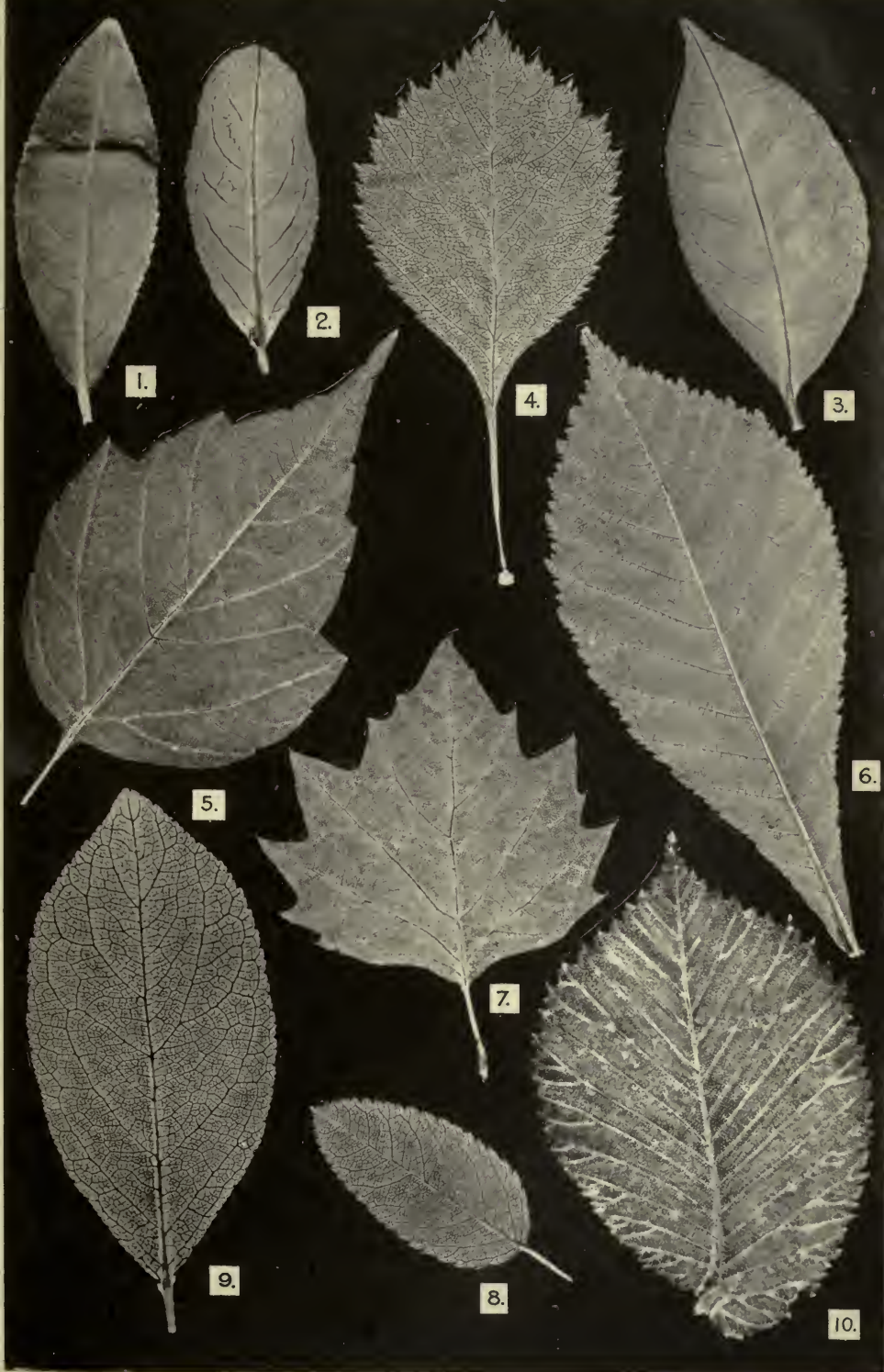
ART. 158. The sale is forbidden of tea (leaves of *Thea Chinensis*) artificially colored or sophisticated by means of the addition of foreign leaves or mineral matters; also the sale of exhausted or damaged tea leaves; and further, the sale is forbidden under the name "tea" of leaves coming from any other plant.

XXIII.—CHOCOLATE.

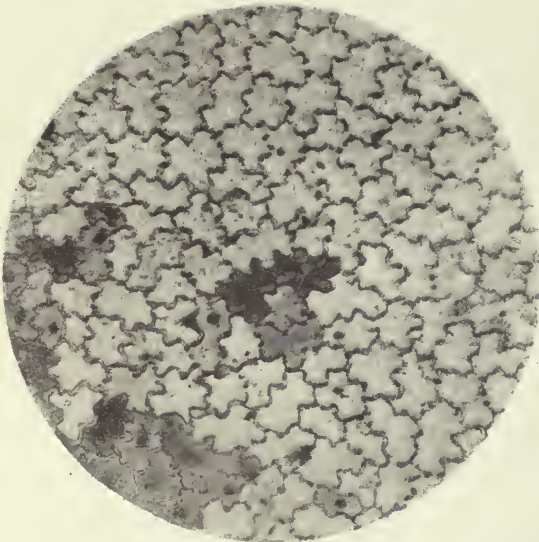
It is forbidden to sell chocolate (powder or the preparations of *Theobroma cacao* and sugar) sophisticated with lime, ocher, or other substances, mineral or vegetable, which are indigestible or harmful.

LIST OF LEAVES ILLUSTRATED BY PLATES XXXIX AND XL.

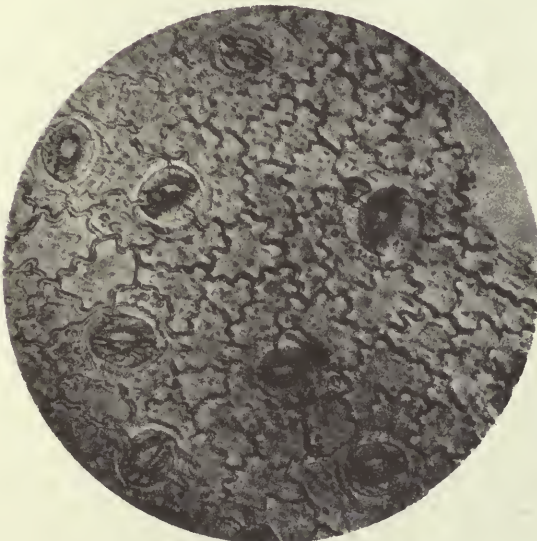
1. Tea.
2. Maté, or Paraguay tea (*Ilex Paraguayaensis*).
3. Camellia (*Camellia Japonica*).
4. Hawthorn.
5. Box Elder.
6. Horse Chestnut.
7. Sycamore.
8. Rose.
9. Plum.
10. Elm.
11. Ash.
- 12 and 13. Willow.
14. Beech.
15. Oak.
16. Missouri, or Golden Currant.
17. Ash.
18. Common Red Currant.
19. Birch.
20. Poplar.
21. Raspberry.
22. Jersey Tea (*Ceanothus Americanus*).



GENUINE TEA LEAVES AND POSSIBLE ADULTERANTS.



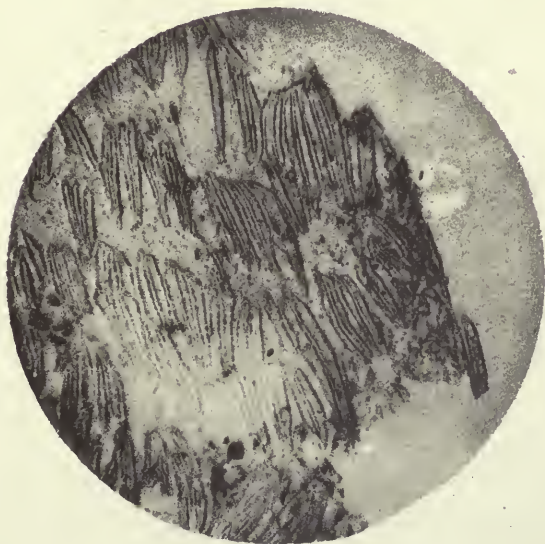
TEA LEAF $\times 115$
Upper surface of epidermis.



TEA LEAF $\times 115$
Lower surface of epidermis.



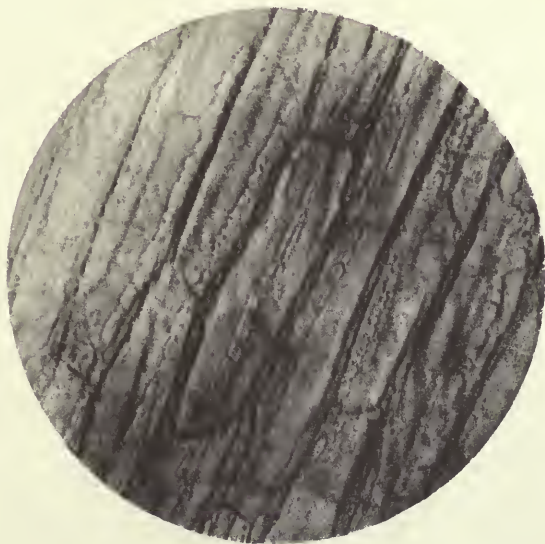
STONE CELL OF TEA LEAF x115



SEED COAT OF COFFEE x 55
"Coffee Flights."



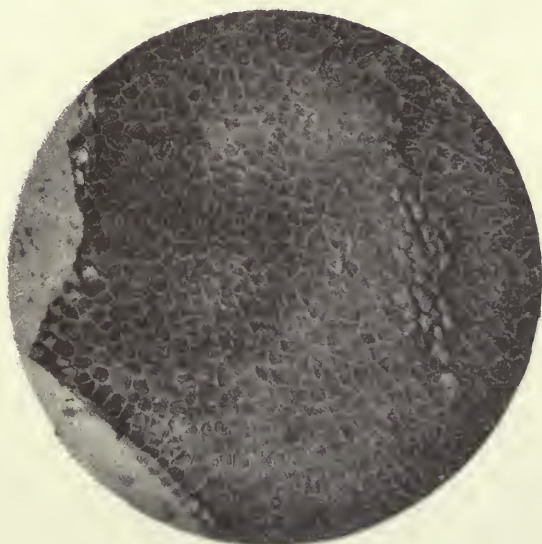
RAW COFFEE x 55
Cross section.



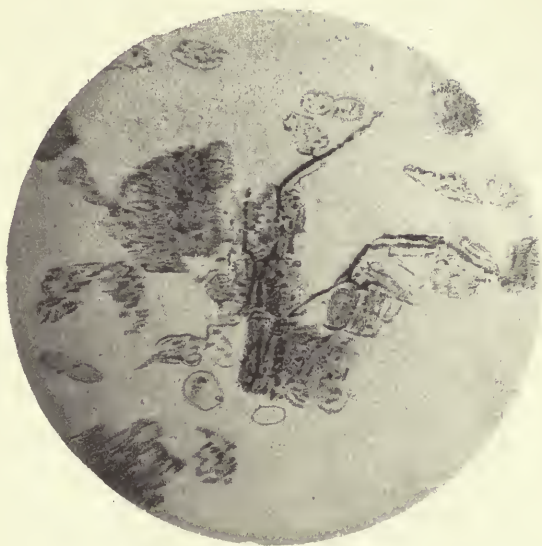
WHEAT BRAN x 115
Outer or longitudinal layer.



WHEAT BRAN $\times 115$
Second or transverse layer.



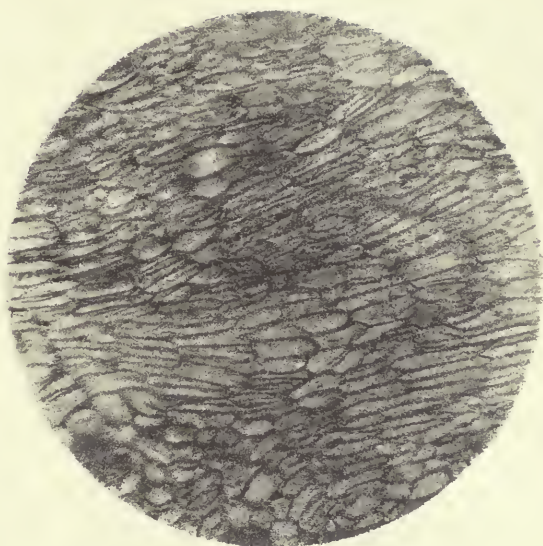
WHEAT BRAN $\times 55$
Gluten layer.



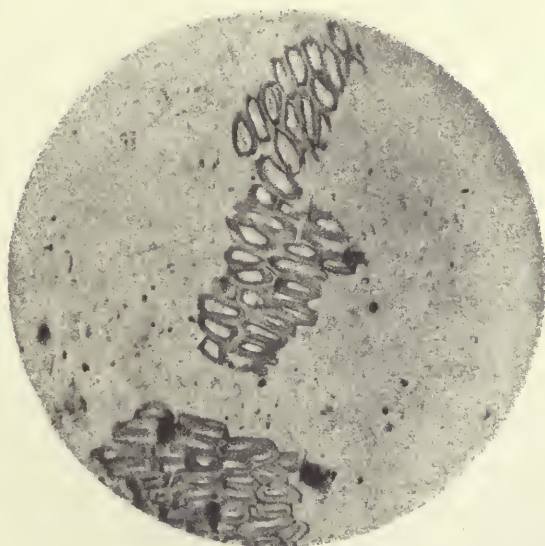
CHICORY (ROASTED) x95
Parenchyma cells and milk vessels.



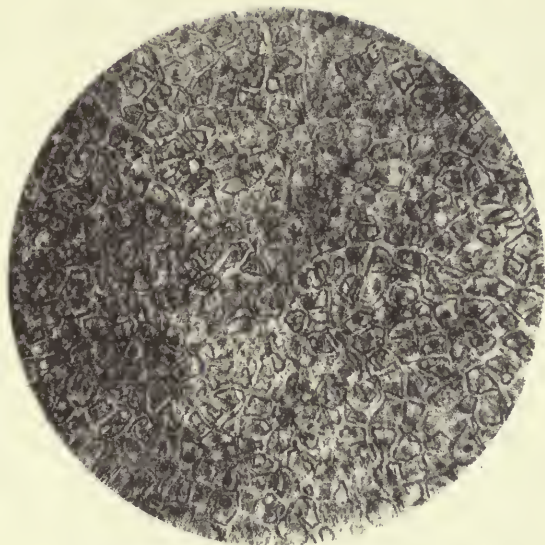
CHICORY (ROASTED) x95
Pitted cells.



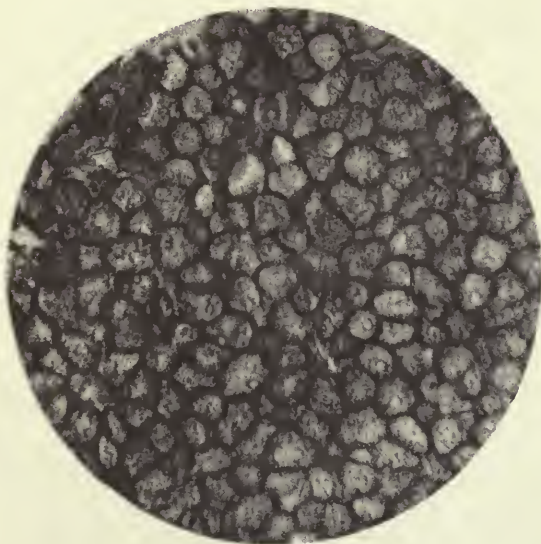
COCOA x 75
Epidermis of Husk.



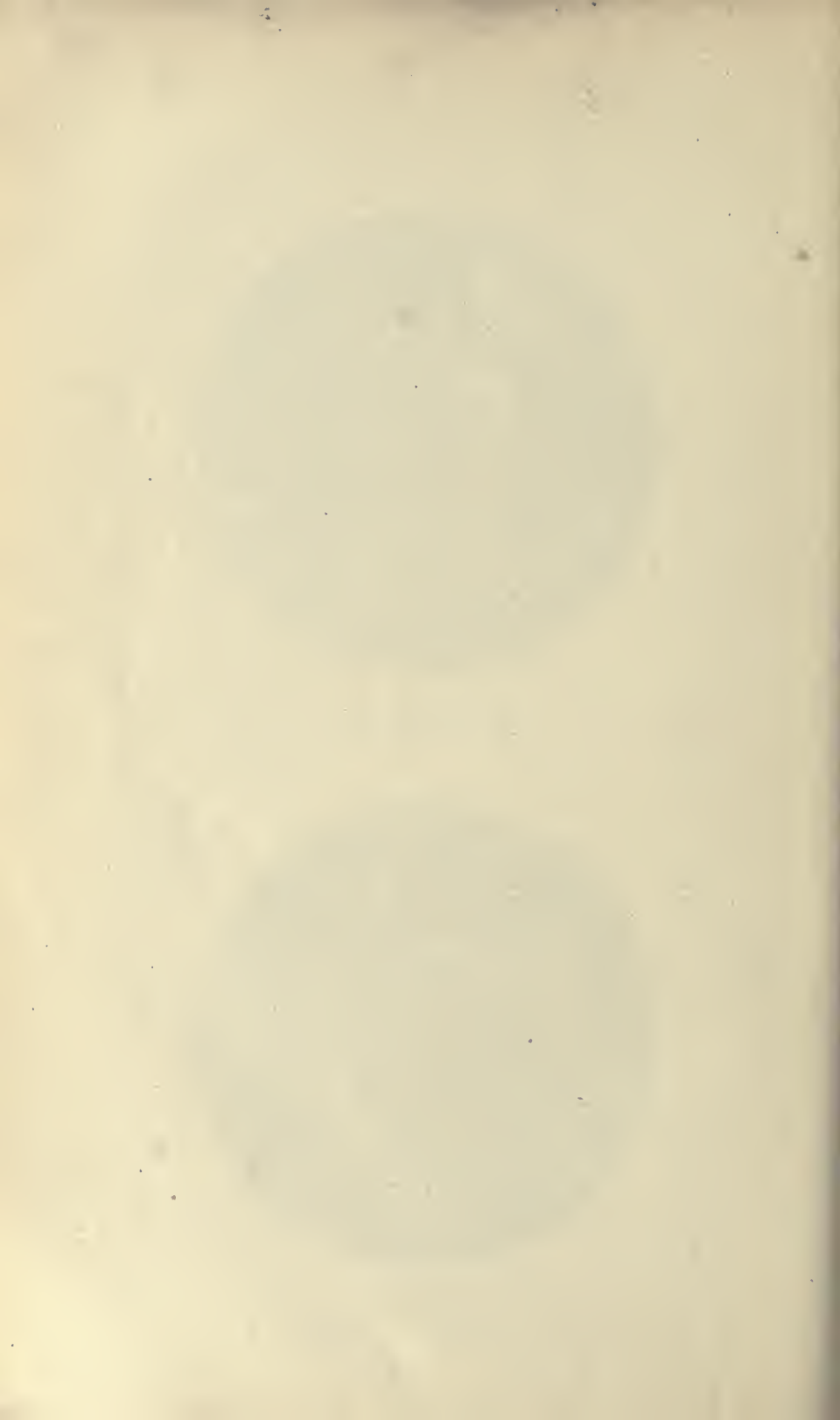
COCOA HUSK x 115
Characteristic thick-walled cells.



COCOA HUSK x115
Thin inner membrane.



COCOA x115
Section of cotyledon.



INDEX.

A.

	Page.
Acid, caffetannic	1004
Acorns, detection of	912
Adulteration of coffees, definition of the	908
foods, definition of	879
Italian law relating to	1013
Adulterations of teas	880
Albuminoid nitrogen, estimation of	908
Albuminoids of cocoa, food value of	939
Ammonia, detection of the use of, in the manufacture of cocoas	956
Analyses of cocoa beans, summary of, by various chemists	960
husks by various chemists	962
preparations by various chemists	959
summary of, by the Department of Agriculture	985
Analysts, general remarks to	892
Analytical methods for cocoa preparations	949
the constituents of teas	889
Ash, acid required to neutralize, from 2 grams of cocoa preparations	956
alkalinity of, in teas	891
determination of, in cocoas	956
insoluble in acid, estimation of, in teas	891
of cocoa beans and preparations, analysis of	969
cocoas, quantitative and qualitative examination of	950
soluble and insoluble, estimation of, in teas	891
maxima, minima, and mean percentage of, in teas	883
total, estimation of, in teas	891
soluble and insoluble of coffees	908
Association Swiss Analytical Chemists, report of	985
Astringents, foreign, in teas	884
Azo-colors, detection of	910

B.

Bibliography of the literature of tea, coffee, and cocoa	991
Black teas	876
Brazil coffee	900
Brick, particles of, detection of, in teas	886

C.

Cacao-masse	941
definition of	942
Caffeine, bibliography of methods for the estimation of	1004
detection of	905

	Page.
Caffeine, estimation of	889
in coffees	907
in coffees	904
presence of, in cocoa beans	939
Caffetannic acid	905, 1004
estimation of	908
Carbohydrates of coffee	905
Catechu, examination of teas for	885
Canna seed	912
Cereals, ash of	912
detection of	912
Ceylon coffee	900
Chicory, bibliography of the literature of	1009
detection of	910
Draper's method for the estimation of	911
formula for the quantitative estimation of, in coffees	911
milk vessels of	911
Premier's method for the estimation of	911
Chocolate a la noisette, definition of	942
Chocolate, definition of	942
Chocolates, preparation of	941
Cocoas and chocolates, recommendations in regard to the examination of	986
Cocoa, aroma of	941
astringent principle of	940
beans and preparations, analysis of, by Bensemann	967
analyses of, by various chemists	960
analyses of commercial preparations of	963
commercial preparations of	941
chemical constituents of	935
nonalkaloidal nitrogenous substances of	939
relations between the constituents of	968
butter, commercial value of	936
extraction of	936
properties of	936
effect of different processes of manufacture on the chemical constituents of	944
fat, physical and chemical constants for	938
properties of	936
fats, variations in the melting and congealing points of	987
food value of	989
gum, properties of	941
manufacture, losses in	934
microscopical examination of	956
powders, examination of, by Stutzer	943
soluble, definition of	942
preliminary processes of manufacturing	934
preparation of, for analysis	949
preparations	933
adulterations of	945
analyses of, by various chemists	962
in the Department of Agriculture	980
analytical methods for	949
bibliography of the literature of	991, 995

	Page.
Cocoa preparations, definition of, Swiss Association of Analytical Chem-	
ists	942
description of samples of	971
discussion of results of examination of	987
examination of, in the Department of Agriculture.	970
microscopic examination of	946
red	940
solubility of, in water	985
starch, measurements of the grains of	948
starch of	940
statistics of	935
summary of analyses of, by the Department of Agriculture	985
tree, description of	933
Coffee	899
ash of	904
average price of, from 1878 to 1890	900
beans, imitation, analyses of	919
seized by the French Government, composition of ..	920
Roumanian Government, composi-	
tion of	920
specific gravity of	919
size of	901
berries, sophisticated	918
bibliography of the literature of	991, 993
bought in the open market, description of	921
Brazil	900
Ceylon	900
chemical composition of	901
consumption, statistics of	900
extracts	929
presence of tin and copper in	929
facing of	909
fat	905
glazing of	913
imitation	915
Java	900
presence of pentose-yielding substances in	907
roasted, composition of	903
with sugar	914
without sugar	914
substitutes	914, 932
unroasted, composition of	902
West India	900
Coloring matter of cocoas	940
cocoa preparations, determination of	956
Copper, detection of, in teas	886
presence of, in coffee extracts	929
Crude fiber, estimation of, in coffees	908

E.

Exhausted leaves, detection of, in teas	882
Extract, half hour, Geissler's averages for	882
total, Geissler's averages for	882
of teas	891

	Page
Extracts of coffee	929
Ewell, Ervin E., report of, on cocoa preparations.....	933
F.	
Facing mixtures for coffees.....	909
of coffees	909
teas	880
detection of	881
objections to	887
Fat, cocoa, estimation of	950
qualitative examination of	951
determination of, in cocoa preparations	956
estimation of, in coffees	908
Fiber, determination of	958
in cocoa preparations	955
Fixed alkalis, detection of the use of, in the manufacture of cocoa preparations	956
Flour, detection of, in cocoa preparations	955
G.	
Geissler, Joseph F., analyses of genuine teas by	882
Glazing of coffees, objections to the	914
Glucose, commercial, determination of, in chocolates	954
Green coffees	927
teas	878
Ground coffees	927
adulterated	929
results of the examination of	928
Gum, determination of, in cocoa preparations	956
of cocoas, properties of	941
Gypsum, detection of, in tea facing	881, 885
I.	
Imitation coffee beans, analyses of	919
seized by the French Government, composition of	920
seized by the Roumanian Government, composition of	920
specific gravity of	919
coffees	915
composition of	916
circulars of dealers in regard to	917
detection of	920
India teas	878
Indigo, detection of, in facing	881
Iron, magnetic oxide of, detection of, in teas	886
metallic, said to be sometimes present in adulterated teas	885
salts, examination of teas for	885
Italian law relating to the adulteration of foods	1013
J.	
Java coffee	900
coloring of	900
K.	
Kunst Kaffee	914

L.		Page.
Leguminous seeds		912
Lie tea		886
detection of		886
M.		
Mangoldwurzel, detection of		911
Mannite, presence of, in coffees		905
Microscopic examination of cocoa preparations		946
Mogdad coffee		913
Moisture, determination of, in cocoas	950-956	
coffees		907
in teas, Geissler's averages for		882
Mussaenda coffee		913
N.		
Nitrogen, albuminoid, Stutzer's method for		890
determination of, in cocoa preparations		956
estimation of		890
O.		
Organic matter, water-insoluble, determination of		956
P.		
Plumbago, detection of, in facing		881
Prussian blue, detection of, in facing		881
R.		
Rio coffee		900
Roasted coffees		926
adulterants of		926
S.		
Sand, detection of, in teas		886
Sawdust		912
Soapstone, detection of, in tea facing	881, 885	
Soluble cocoa		945
Sophisticated coffee beans		918
Spent leaves, detection of, in teas		882
Starch, Allen's method for the detection of		912
average amount of, in cocoa and chocolate		986
determination of, in cocoa preparations		954
McElroy's method for the determination of		957
of cocoas		940
percentage of, in materials used in cocoa manufacture		958
Starches used as adulterants of cocoa		949
Stone cells, presence of, in tea leaves		884
Substitutes for coffee		914
Sugar and sirup, treatment of coffee with		913
determination of		957
estimation of, in cocoa preparations		954
Swiss Analytical Chemists' Association, report of		985
T.		
Tannin, bibliography of the methods for		997
determination of, in cocoa preparations		956

	Page.
Tannin, estimation of	890
maxima, minima, and mean percentages of, in tea	883
Tartaric acid, estimation of, in cocoas	941
Taylor, Dr. Thomas, method of, for preparing leaves for microscopic examination	884
Tea	875
adulterants, general remarks on	886
adulteration law, United States	1011
bibliography of the literature of	991, 992
general classification of	875
green	878
half-hour extract of	892
leaf, general examination of	884
leaves, stomata of	884
maxima, minima, and mean percentages of tannin in	883
theine in	883
statistics of the consumption of	875
Teas, adulterations of	880
analyses of	894
analytical methods for the constituents of	889
black	876
China, analyses of	888
classification of	888
description of samples of	892
estimation of insoluble-leaf of	891
foreign astringents in	885
leaves in	883
general statements concerning the constituents of	887
green and black, comparative analyses of	879
India	878
Japanese flat	878
method of judging the quality of	877
methods of manufacturing	876
proposed chemical method for estimating value of	888
qualitative examination of	896
table showing the condition of the leaves and foreign matters present	896
remarks on qualitative examination of	897
Theine, estimation of	889
maxima, minima, and mean percentages of, in tea	883
presence of, in every part of a theine-producing plant	884
sublimation test for	884
Theobroma cacao	933
Theobromine, properties of	937
Tin poisoning	930
presence of, in coffee extracts	929
Turmeric, detection of, in facing	881
W.	
Water, estimation of, in teas	891
insoluble organic matter of cocoas	985
Wax palm, fruit of, as a coffee substitute	915
West India coffee	900

