

U. S. DEPARTMENT OF AGRICULTURE.  
DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

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# FOODS

AND

# FOOD ADULTERANTS.

BY DIRECTION OF

THE COMMISSIONER OF AGRICULTURE.

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PART THIRD:

FERMENTED ALCOHOLIC BEVERAGES,  
MALT LIQUORS, WINE, AND CIDER.

BY

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WASHINGTON:  
GOVERNMENT PRINTING OFFICE.  
1887.





[BULLETIN No. 13.]

## PART 3.—FERMENTED ALCOHOLIC BEVERAGES.

### LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,  
*Washington, D. C., August 15, 1887.*

SIR: I have the honor to submit herewith that portion of the work on food adulteration which has been under my charge.

I take this opportunity for acknowledging my indebtedness to the following gentlemen, Messrs. Trescot, Fiske, Lengfeld, and Dugan—my fellow assistants in the division—by whom a large part of the analytical work was performed.

Respectfully,

C. A. CRAMPTON,  
*Assistant Chemist.*

Dr. H. W. WILEY,  
*Chief Chemist.*  
4450—No. 13, PT. 3—1



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PART I.

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MALT LIQUORS.

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## MALT LIQUORS.

The production of malt liquors in this country as an industry is second only in importance to the production of breadstuffs. Their consumption is steadily on the increase, as is also the amount consumed in proportion to other kinds of alcoholic beverages. The following tables are taken from recent statistics, compiled by the Bureau of Statistics, U. S. Treasury Department, from figures obtained from official sources:<sup>1</sup>

*Annual consumption of distilled and malt liquors and wines in the United States and the average annual consumption per capita of population during the years 1840, 1850, 1860, and from 1870 to 1886, inclusive.*

Year ending June 30—	Distilled spirits consumed.				Wines consumed.		
	Spirits of domestic product.		Imported spirits entered for consumption.	Total.	Wines of domestic product. <sup>2</sup>	Imported wines entered for consumption.	Total.
	From fruit.	All other.					
1840	<i>Pr. galls.</i>	<i>Pr. galls.</i>	<i>Pr. galls.</i>	<i>Pr. galls.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>
1840	(3)	40,378,690	2,682,794	43,060,884	124,734	4,748,362	4,873,096
1850	(3)	46,768,083	5,065,390	51,833,473	221,249	6,094,622	6,315,871
1860	(3)	83,904,258	6,064,393	89,968,651	1,860,008	9,199,133	11,059,141
1870	1,223,820	77,266,368	1,405,510	79,895,708	3,050,518	9,165,549	12,225,067
1871	2,472,011	59,842,617	1,745,028	64,059,661	4,980,783	10,853,280	15,834,063
1872	1,083,698	65,145,880	2,180,702	68,422,280	6,968,737	9,713,300	16,682,037
1873	2,965,987	62,945,154	2,125,998	68,037,139	8,953,285	9,893,746	18,847,031
1874	766,687	61,814,875	1,958,528	64,540,090	10,951,859	9,516,855	20,468,714
1875	1,757,202	62,668,709	1,694,647	66,120,558	12,954,961	7,036,360	19,991,330
1876	672,231	57,340,472	1,471,179	59,483,890	14,968,083	5,193,723	20,161,808
1877	1,527,141	57,016,248	1,376,729	59,920,118	16,942,592	4,933,738	21,876,330
1878	1,103,251	49,600,838	1,227,732	51,931,941	17,953,386	4,310,563	22,263,949
1879	1,021,708	52,003,467	1,253,300	54,278,475	19,845,113	4,532,017	24,377,130
1880	1,005,781	61,126,634	1,394,279	63,526,694	23,298,940	5,030,601	28,329,541
1881	1,701,206	67,426,000	1,479,875	70,607,081	18,931,819	5,231,106	24,162,925
1882	1,216,850	70,759,548	1,580,578	73,556,976	19,934,856	5,628,071	25,562,927
1883	1,253,278	75,508,785	1,690,624	78,452,687	17,406,028	8,372,152	25,778,180
1884	1,137,056	78,479,845	1,511,680	81,128,581	17,402,938	3,105,407	20,568,345
1885	1,468,775	67,689,250	1,442,067	70,600,092	17,404,698	4,495,759	21,900,457
1886	1,555,994	69,295,361	1,410,259	72,261,614	17,366,393	4,700,827	22,067,220

<sup>1</sup> Statements Nos. 32 to 50, inclusive, of the Quarterly Report No. 2 series 1886-'87, of the Chief of the Bureau of Statistics, Government Printing Office, 1887.

<sup>2</sup> Product less exports.

<sup>3</sup> Included with "All other."

Annual consumption of distilled and malt liquors and wines, &amp;c.—Continued.

Year ending June 30—	Malt liquors consumed.			Total consumption of wines and liquors.	Total consumption per capita of population.			
	Malt liquors of domestic product. <sup>1</sup>	Imported malt liquors entered for consumption.	Total.		Distilled spirits.	Wines.	Malt liquors.	All wines and liquors.
1840	23,162,571	148,272	23,310,843	71,244,817	2.52	0.29	1.36	4.17
1850	36,361,708	201,301	36,563,009	94,712,333	2.23	0.27	1.58	4.08
1860	100,225,879	1,120,790	101,346,699	202,374,461	2.86	0.35	3.22	6.43
1870	203,743,401	1,012,755	204,756,156	296,876,931	2.07	0.32	5.30	7.69
1871	239,888,137	1,299,996	241,138,127	321,031,851	1.62	0.40	6.09	8.11
1872	268,357,983	1,910,933	270,298,916	355,403,233	1.68	0.41	6.65	8.74
1873	298,519,675	2,177,587	300,697,262	387,581,432	1.63	0.45	7.27	9.29
1874	297,519,981	2,001,084	299,521,065	384,529,860	1.51	0.48	6.99	8.98
1875	292,961,047	1,992,110	294,953,157	381,065,045	1.50	0.45	6.71	8.66
1876	306,852,467	1,483,920	308,336,387	387,982,085	1.32	0.45	6.83	8.60
1877	303,854,988	1,072,679	304,926,667	386,723,115	1.29	0.47	6.58	8.34
1878	317,136,597	832,755	317,969,352	392,165,242	1.09	0.47	6.68	8.24
1879	343,724,971	880,514	344,603,485	423,261,090	1.11	0.50	7.05	8.66
1880	413,208,885	1,011,280	414,220,165	506,076,400	1.26	0.56	8.26	10.08
1881	442,947,664	1,164,505	444,112,169	538,882,175	1.37	0.47	8.63	10.47
1882	524,843,379	1,536,601	526,379,980	625,499,883	1.39	0.48	9.97	11.81
1883	549,616,338	1,881,002	551,497,340	655,728,207	1.45	0.48	10.18	12.11
1884	588,005,609	2,010,908	590,016,517	691,653,443	1.46	0.37	10.62	12.45
1885	594,063,095	2,068,771	596,131,866	688,632,415	1.24	0.38	10.41	12.06
1886	640,746,288	2,221,432	642,967,720	737,296,554	1.24	0.38	11.18	12.62

1 Product less exports.

NOTES.—(1) The data as to product of domestic liquors and wines for 1840, 1850, and 1860 were derived from the Census. (2) The consumption of imported liquors and wines for 1840, 1850, and 1860 is represented by the net imports. (3) The production of domestic wines, from 1870 to 1885, has been estimated by the Department of Agriculture; by Mr. Charles McK. Leoser, president of Wine and Spirit Traders' Society, New York, and other well-informed persons, and the amount stated as consumed represents the production minus the exports. (4) The consumption of domestic spirituous and malt liquors, from 1870 to 1886, was obtained from the reports of the Commissioner of Internal Revenue. (5) In computing the quantity of sparkling and still wines and vermouth in bottles, 5 so-called quart bottles are reckoned as equivalent to the gallon. (6) The consumption of distilled spirits as a beverage is estimated to be about 90 per cent. of the product consumed for all purposes.

This table shows admirably the rapid increase, especially in the last ten years, of the consumption of malt liquors, and the relative decrease in the consumption of the stronger alcoholic beverages. Thus it will be seen that in 1840 the amount of malt liquor consumed per capita was a little over one-half the amount of distilled liquor consumed; while in 1886 it was *nine times as much*. The amount of distilled liquor consumed per capita has diminished during the twenty-six years to one-half, while the amount of malt liquor consumed has increased very nearly seven times; or, in other words, the malt liquors have been driving out the distilled at the rate of about .05 gallons per capita each year, and supplanting it at the rate of about .38 gallons per capita.

The average quantity consumed annually for the last three years was 609,705,367 gallons, of which 2,100,370 gallons were imported. Taking this as a basis, Mr. F. N. Barrett, in the publication above mentioned, estimates the amount expended for beer per annum at \$304,852,683, placing the cost to the consumer at 50 cents per gallon. The cost to the consumer of the total quantity of liquors per annum he places at \$700,000,000.

That there is still opportunity for increase in the consumption of malt liquors in the United States will be seen from the following compara-

tive tables, from which it appears that while the United States quite holds her own in the quantity of distilled liquors consumed, she is still far behind the other great nations in the consumption of the milder alcoholic liquors.

*Comparative summary of the consumption per capita of population in the United States, the United Kingdom, France, and Germany, of distilled spirits, wines, and malt liquors during each year from 1881 to 1885, inclusive.*

[From original official data.]

Years.	Distilled spirits.				Wines.				Malt liquors.			
	United States.	United King-dom.	France.	Germany.	United States.	United King-dom.	France.	Germany.	United States.	United King-dom.	France.	Germany.
1881.....	Galls. 1.39	Galls. 1.00	Galls. .94	Galls. 1.32	Galls. .48	Galls. .43	Galls. 18.52	Galls. ( <sup>1</sup> )	Galls. 9.97	Galls. 33.90	Galls. ( <sup>1</sup> )	Galls. 22.35
1882.....	1.45	1.07	1.08	1.14	.48	.41	27.10	( <sup>1</sup> )	10.18	33.65	( <sup>1</sup> )	22.45
1883.....	1.46	1.03	1.24	1.19	.37	.40	30.75	( <sup>1</sup> )	10.62	33.13	( <sup>1</sup> )	22.45
1884.....	1.24	1.05	1.25	1.11	.38	.39	30.67	( <sup>1</sup> )	10.44	32.72	( <sup>1</sup> )	23.19
1885.....	1.24	1.01	1.32	1.14	.38	.37	36.88	( <sup>1</sup> )	11.18	32.79	( <sup>1</sup> )	23.78

<sup>1</sup> No data.

NOTE.—The years referred to are, for France and Great Britain, calendar years; for the United States, the five years ending June 30, 1886; for Germany in the case of beer, the five years ending March 31, 1885, and in the case of spirits the five years ending March 31, 1881, these being the latest years for which data were obtainable.

It is hardly necessary, after the above showing, to dwell upon the importance of this article of daily consumption, or the necessity of a thorough acquaintance with its manufacture, composition, and the nature and extent of its adulterations. There is no beverage that compares with it in the amount consumed by the people except water, and possibly milk. But little supervision has been exercised over its manufacture and sale, except the rigorous enforcement by the Government of its demands for a share in the profits of its manufacture.

#### THE PROCESS OF BREWING.

Brewing, or the art of preparing an alcoholic drink from starchy grains by fermentation, is of very ancient origin. It was practiced by the Egyptians, and the Greeks and Romans learned the art from them. Herodotus speaks of the Egyptians making wine from corn, and it was undoubtedly practiced by the Greeks in the fifth century before Christ, as the use of malt beverages is mentioned in the writings of Æschylus and Sophocles, poets of that period. It is also mentioned by Xenophon, 400 B. C. The Romans are also supposed to have derived a knowledge of the art from the Egyptians, and Pliny and Tacitus both speak of its use among the Gauls and Germans of Spain and France.

It is supposed that the art was introduced into Britain by the Romans and acquired from the natives by the Saxons. According to Verstigan "this excellent and healthsome liquor, beere, anciently called ale,

as of the Danes it yet is, was of the Germans invented and brought into use." Ale-houses are mentioned in the laws of Ina, king of Wessex, A. D. 680. Ale-booths were regulated by law A. D. 728.

The art of producing an alcoholic drink from starchy seeds seems to have been nearly as extensively known and practiced among the various nations of the earth as the less complex operation of preparing a fermented liquor from the juice of fruits and plants containing sugar. Thus the Kaffre races of South Africa are said to have prepared for many years a malt liquor from the seeds of the millet (*Sorghum vulgare*) going through all the processes of germinating the seed, extracting the malt, and fermenting the wort. In the north of Africa another seed is used. The Chinese prepared the drink called sam shee from rice.

The process of brewing consists of two distinct operations: the malting and the brewing proper. In fact the two operations are frequently separated, many small breweries buying their malt ready prepared. When kept dry it retains its qualities for an indefinite period and is handled as an article of commerce.

#### MALTING.

The object of this operation is the germination of the grain, and the consequent formation of the ferment diastase, which shall subsequently, under the proper conditions, perform its specific function of converting the starchy portions of the grain into saccharine or fermentable matter. Barley is the grain used almost exclusively for this purpose, its advantages having been recognized even by the Egyptians; they seem to be principally of a physical character, consisting of the firmness of the kernel, and the hard husk, which freely allows the entrance of water, but prevents the passage of starch or insoluble matter.

The operations through which the grain is successively passed are called, technically, steeping, crushing, flooring, and kiln-drying. In the first it is spread out in large vats, covered with water, and allowed to steep several days. When it has become softened, the water is run off and the swollen grain is subjected to a slight degree of heat, which causes it to germinate. This is the second operation. The operation of flooring has for its end the regulation of the germination of the grain, and the time when it has progressed sufficiently is judged by the length which has been attained by the aerospire or plumule. This is variously given as from two-thirds to seven-eighths the length of the grain. The sprouted grain is now spread out in the malt kilns and heat applied, while a current of air circulates about it. After the moisture is driven off, which is done at a low temperature, about 90° F., the heat is raised, and finished at from 125° to 180° F., according to the grade of malt required, the difference between pale, amber, and brown malt being due simply to the temperature at which they are kiln-dried. This last operation serves not only to drive off the moisture, but also stops ger-

mination by destroying the vitality of the germ, and fits it for keeping. It also probably develops the flavor by the formation of a minute quantity of empyreumatic oil in the husk.

The rootlets and germs are removed in this process by the turning and stirring of the grain. The water which is used in the process of steeping the grain is an important factor in the production of good malt, and the preference of brewers for hard lime waters for this purpose has been shown by recent experiment to be rational, for it is found that when barley is steeped with distilled water, a very putrescible liquor is obtained charged with albuminous matter, while if a hard water is used these matters remain in an insoluble condition in the grain.

Chemically considered a good malt should not contain more than 5 per cent. of water, and the soluble extract should constitute about 70 per cent. of the weight of the malt and should have a good diastatic action on starch mucilage. The determination of the acidity is important in determining the keeping qualities of the beer which is to be brewed from it, and should not be over .3 per cent. (calculated as lactic).

The following analyses by O'Sullivan show the composition of pale malt:

	(1)	(2)
Starch .....	44.15	45.13
Other carbohydrates (of which 60-70 per cent. consist of fermentable sugars). Inulin (?) and a small quantity of other bodies soluble in cold water.....	21.23	19.39
Cellulose matter .....	11.57	10.09
Fat .....	1.65	1.96
Albuminoids.....	13.09	13.80
Ash .....	2.60	1.92
Water .....	5.83	7.47
	100.12	99.76

#### BREWING.

Brewing proper includes a number of distinct operations, such as grinding and mashing the malt, boiling and cooling the wort or infusion, fermenting it, and clearing and racking the beer. In the process of mashing takes place the conversion of the starch into fermentable sugar, mainly maltose, by the action of the diastase. Two methods are used for extracting the soluble matter from the malt, called *infusion* and *decoction*, respectively; the former is the method most in use in England, the latter in Germany and France. The wort prepared by infusion contains less dextrin and more albuminoid matter than that prepared by decoction; the beers from the former are stronger in alcohol, but not so good in keeping qualities.

A good wort should give no blue color with iodine, showing the complete conversion of all the starch, and should contain a large percentage of maltose, which should constitute about 70 per cent. of the extract.

After the mashing process comes the *boiling* of the wort, which is begun as soon as it is drawn off from the exhausted malt and continued for one to two hours. This prevents the formation of acid, and serves to extract the hops, which are added at this stage of the process. The boiling of the wort with hops serves not only to impart to it the desired hop flavor, but also to partially clarify it by precipitating some albuminous matter by means of the tannin in the hops, and to enhance its keeping qualities. To this end larger quantities of hops are used for beers intended for exportation or long keeping.

The wort is now ready to be submitted to the most important operation of all—fermentation—which calls for very careful supervision on the part of the brewer.

#### FERMENTATION.

After the wort has been boiled with hops it is cooled as rapidly as possible, to prevent the formation of acid, usually effected by means of artificial refrigerating apparatus; it is then ready for the addition of the yeast.

There are two distinct methods of fermentation in use, called by the Germans *Ober- und Untergärung*, and by the French *fermentation haute* (top fermentation) and *basse* (bottom fermentation). The former is carried on at a comparatively high temperature, the action is rapid, and the yeast with the impurities is carried to the surface of the liquid; in the latter method the temperature is kept low, the fermentation goes on slowly, and the yeast and impurities sink to the bottom. The second method is often called the Bavarian method, as it seems to have originated there, and is used exclusively in that country. It is generally preferred in Germany and France, while in England and this country the upward clearing method appears to be more in vogue.

The nature of the fermentation depends greatly upon the character of the yeast used, for Pasteur's experiments have shown that yeast from upward-fermented beer tends to produce the upward fermentation, while yeast from bottom-fermented beer produces the bottom fermentation. The purity of the yeast used is of the very first importance in the production of good beer. Many experiments have been made with the end in view of producing a perfectly pure yeast, which should contain only the yeast ferment proper, and thus produce a beer of good flavor and keeping properties, free from diseased or acid ferments.

#### PURE YEAST.

The production of pure yeast for brewing purposes has been put on a practical basis of late years through the scientific researches of Dr. E. C. Hansen, of the Carlsberg Institute, in Copenhagen. He succeeded in producing a pure yeast cultivated from a *single cell*. He was able to differentiate in this way six different species or varieties of *saccharomyces*, several of which may usually be found in an ordinary brewery

yeast. These different varieties have been shown to produce beers differing not only in coloring, flavoring, facility of separation of the yeast, &c., but also in chemical composition.

In a recent address before the Society of Chemical Industry by G. H. Morris,<sup>1</sup> a résumé is given of the work done in this direction by Hansen and others.

Dr. Morris states that the employment of the pure yeasts is coming very largely into use in the beer-drinking countries of the Continent, and has met with favor from some of the most noted brewing technologists, such as Jacobson, Aubry, März, and Lintner, the latter of whom sums up the question in the following statements:

- (1) By contamination with so-called wild yeast an otherwise normal brewery yeast can be rendered incapable of producing a beer of good flavor and with good keeping qualities.
- (2) A contamination with wild yeasts may be produced by the dust of the air during summer and autumn, by the malt, or other sources.
- (3) By employing Hansen's method of pure cultivation and analysis it is possible to obtain from a contaminated yeast a good brewery yeast in a state of purity.
- (4) Yeast cultivated in a state of purity possesses in a marked degree the properties of the original yeast before contamination as far as concerns the degree of alteration of the flavor and keeping qualities of the beer.
- (5) There exist different varieties of normal bottom yeast (*S. cerevisiae*), each with special properties which, like the peculiarities of species, are maintained constant.

The use of this yeast has not yet extended to England, although experiments on an industrial scale are now being carried on at Burton-on-Trent with different species of pure yeast.

The chemical characteristics of beer made from the different species of pure yeast have been investigated by Borgmann,<sup>2</sup> who analyzed samples of beer produced from two species of pure yeast, each cultivated from a single cell and the beer fermented under comparable conditions. The analysis gave the following results:

	Beer prepared with—	
	Yeast No. 1 gives in 100 cc.	Yeast No. 2 gives in 100 cc.
Alcohol.....	4.13	4.23
Extract.....	5.35	5.84
Ash .....	.20	.25
Fatty acid (as lactic) .....	.086	.144
Glycerol.....	.109	.137
Phosphoric acid .....	.0775	.0828
Nitrogen.....	.071	.0719

From these numbers, which are the means of many determinations, the analyst concludes that the different yeasts produce beers which differ in chemical composition. He also finds that the proportion of alcohol

<sup>1</sup> Jour. Soc. Chem. Ind., 1887, p. 113.

<sup>2</sup> Zeit. Anal. Chem. 25, 532.

to glycerol is different from that formed with other beers. From analyses of other beers he finds that the proportion is—

	Alcohol.	Glycerol.
Maximum .....	100	5.497
Minimum .....	100	4.14

while with the Carlsberg pure yeast the proportion is—

	Alcohol.	Glycerol.
No. 1 .....	100	2.63
No. 2 .....	100	3.24

#### BELGIAN BEERS.

The method of brewing pursued in Belgium would seem the most unscientific known, still the Belgium beers are largely consumed in Europe. No yeast whatever is used, but the wort is left to ferment of itself, as it were, standing in the vats at a low temperature, until fermentation is provoked by germs that have accidentally fallen into it, or which may have found their way in during the process of manufacture. The action is naturally very slow, requiring sometimes several years for its completion, and a considerable formation of acid takes place, which is a predominant constituent in this class of beers.

#### CLARIFYING, STORING, AND PRESERVING.

The treatment of malt liquors after the process of fermentation is complete is very diverse, according to the kind of liquors it is intended to produce, the length of time it is to be kept, &c. The problem of clarifying and preserving the beer is very simple of solution if it has been properly and carefully brewed, for then it is easily cleared and keeps well; but where the reverse is the case it is necessary to make use of various clarifying and preserving agents, and here comes in the delicate question of the proper agents to use, which will perform this duty and still introduce no objectionable constituents into the drink.

The discussion of this question comes properly under the head of adulterations, and will be considered later on. As clarifying agents may be mentioned gelatine, tannin, Iceland moss, and flaxseed, and as mineral coagulating agents phosphate of lime, and alum.

Formerly beer was stored in casks or vats in cool cellars for a long period, to allow it to age or ripen, especially in Germany, whence came the name of "lager" beer, but the aim of the brewer at the present day is to produce an article fit for the market in as short a time as possible and thus turn his capital often and keep step with the rapid pace of modern business industry, so that the name of lager beer is rather a misnomer.

## COMPOSITION OF MALT LIQUORS.

The composition of malt liquors varies greatly according to the materials used, the method of brewing, the season, and the use for which it is intended.

Malt liquors, properly so called, should be made only of malted barley, hops, yeast, and water, but the use of other materials as substitutes for the first three ingredients has extended so greatly in countries where their use is not prohibited that it is difficult to define what a beer really is.

Modern beer has been defined as a "fermented saccharine infusion to which some wholesome bitter has been added."

Its chemical composition is very complex, the principal constituents being alcohol, various sugars and carbohydrates, nitrogenous matter, carbonic, acetic, succinic, lactic, malic, and tannic acids, bitter and resinous extractive matter from the hops, glycerine, and various mineral constituents, consisting mainly of phosphates of the alkalies and alkali earths.

## VARIETIES.

The names given to different kinds of malt liquors relate to various attributes, as the country where they were produced, as English, German, Bavarian beer, &c., or to the peculiarities in the method of brewing, &c. Thus, *porter* is simply a beer of high percentage of alcohol, and made from malt dried at a somewhat high temperature, which gives its dark color; *ale* is a pale beer, likewise of high attenuation and made of pale malt, with more hop extract than porter. *Stout* has less alcohol and more extract and still less hops than porter. These terms are used chiefly with reference to English malt liquors. The terms used for German beers, such as *Erlanger*, *Münchener*, &c., are for the most part names of places and are applied to beers made in imitation of the beers originally brewed in those cities. *Export* beer is beer that is specially prepared with a view to long-keeping qualities.

The following table gives some recently published and very complete analyses of beers made by C. Graham: <sup>1</sup>

<sup>1</sup> Jour. Soc. Chem. Ind.

## Composition of various typical beers (Graham).

	Non-volatile.	Volatile.*						
	Maltose.	Dextrose.	Albuminoles (Wanklynnized).	Acetate and succinate acids.	Alcohol.	Total nitrogen (by soda Lime).	Specific gravity of beer.	Original gravity of wort.
			Ash, coloring matter, and hop extract (by difference).	Total solid matters.			Ratio of maltose to dext.	Ratio of solids to alcohol.
Burton ales:								
Mild	2.13	3.64	0.26	0.18	0.53	6.74	0.01	6.78
Pale	1.75	2.48	0.21	0.14	0.55	5.13	0.02	5.37
Bitter	1.62	2.60	0.16	0.17	0.87	5.42	0.01	5.44
Mild X.	1.87	1.88	0.20	0.14	1.30	5.39	0.04	4.60
XXX.	2.88	2.04	0.30	0.10	1.48	6.80	0.02	6.30
A. K. bitter	0.81	0.75	0.21	0.14	0.85	2.76	0.06	4.69
Somersashire ale two years old	1.54	2.48	0.42	0.64	0.94	6.02	0.07	5.50
Scotch export, bitter.	1.62	2.50	0.30	0.09	0.70	5.21	0.16	5.00
Dublin stout, XXX.	3.45	3.07	0.26	0.17	1.76	8.11	0.01	5.50
Dublin stout, XXX.	5.35	2.69	0.43	0.25	1.40	9.52	0.04	6.78
German beer:								
Vienna lager	1.64	2.74	0.36	0.13	1.12	5.90	0.02	4.69
Pilsen lager	0.69	2.65	0.20	0.09	0.59	4.22	0.02	3.29
Munich lager	1.57	3.15	0.40	0.14	1.82	7.08	0.01	4.75
Rivoli (sold in London)	1.75	2.15	0.17	0.12	1.39	5.58	0.02	5.31
Bohemian (average)	0.57	2.14	0.26	0.09	0.82	3.81	0.06	3.97
Davarian (average)	0.87	2.26	0.26	0.09	0.87	4.17	0.14	4.14

\* Water not determined.

The following is taken from the report of the Municipal Laboratory Paris for 1885, and gives the composition of the principal beers sold in Paris.<sup>1</sup>

*Analyses of beers made in Municipal Laboratory of Paris in 1881.*

No.	Density.	Per cent. by volume of alcohol.	Grams per liter.						Polarization.	Remarks.
			Extract.	Sugar.	Dextrin.	Albuminoid.	Ash.	Acidity as $H_2SO_4$ .		
1.....	1.020	6.1	64.20	12.50	34.10	3.04	2.28	.....	+15°. 76	Good.
2.....	1.020	6.2	65.00	15.00	40.60	3.86	2.28	.....	+16°. 25	
3.....	1.019	6.2	63.10	13.17	28.20	.....	.....	.....	+ 5°. 88	
4.....	1.021	5.5	65.70	12.35	31.80	.....	2.33	.....	+ 7°. 38	
5.....	1.017	6.2	56.20	13.00	.....	.....	2.39	.....	+14°. 50	
6.....	1.022	5.0	75.90	11.62	47.16	.....	2.32	.....	+ 7°. 50	
7.....	1.024	5.6	79.25	17.32	32.91	.....	2.32	1.47	+17°. 48	Contained salicylic acid.
8.....	1.027	6.1	62.25	11.16	51.00	.....	2.09	.68	+15°. 34	Good.
9.....	1.030	5.8	98.36	23.92	36.43	.....	2.93	1.47	+23°. 52	
10.....	1.019	5.2	65.53	12.56	.....	.....	2.04	1.12	+14°. 20	
11.....	1.020	5.7	65.50	15.83	.....	.....	1.58	1.96	+18°. 05	
12.....	1.017	6.5	58.72	11.58	.....	.....	1.64	2.45	+15°. 17	
13.....	1.020	4.0	58.42	10.01	.....	.....	1.94	2.45	+12°. 21	
14.....	1.020	5.7	63.40	8.30	.....	.....	.....	.....	+ 6°. 27	
15.....	.....	5.8	53.48	14.29	.....	.....	2.70	.....	+14°. 50	Contained salicylic acid.
16.....	1.013	5.9	59.59	9.81	.....	.....	2.90	1.21	+12°. 41	Good.
17.....	1.014	5.9	60.03	9.79	.....	.....	2.95	1.12	+11°. 54	
18.....	1.019	5.3	48.90	8.28	.....	.....	2.85	2.03	+ 7°. 74	
19.....	.....	7.3	78.45	16.54	.....	.....	4.30	.....	+20°. 00	
20.....	.....	4.7	58.71	12.23	.....	.....	2.90	1.04	+20°. 68	
21.....	.....	3.4	27.45	.....	.....	.....	.95	1.36	+ 5°. 60	Passable.
22.....	.....	3.8	64.49	11.91	.....	.....	2.35	.86	+13°. 12	Good.

General averages of 135 samples of beer, analyzed in 1882: Alcohol, 4.25 per cent. by volume; extract, 52.06 grams per liter.

The following, taken from the same source, is interesting as showing an average of the composition of beers manufactured in various countries. It is taken from a very large number of collated analyses.

*Average of the content of alcohol, extract, and ash in various beers for export and preservation.*

	Per cent. alcohol by volume.			Per cent. extract.			Per cent. ash.		
	Min.	Max.	Mean.	Min.	Max.	Mean.	Min.	Max.	Mean.
<i>French beers.</i>									
Strasburg.....	4.0	5.8	4.7	4.0	5.6	4.65	0.30	0.35	0.32
Lille.....	4.0	4.2	4.1	4.0	5.3	4.65	.....	0.35	.....
Paris.....	3.5	3.5	3.5	4.0	8.0	6.00	.....	.....	.....
Nancy, Tourtel, Tartenville, Vittel, Vezelise, Toul-	5.0	6.0	5.6	4.0	7.6	5.70	0.10	0.35	0.20
Lyon.....	5.0	.....	.....	5.0	.....	.....	.....	.....	.....
<i>German beers.</i>									
Saxony.....	2.08	6.90	3.70	4.40	7.4	5.8	0.18	0.45	0.25
Bavaria.....	3.00	8.30	4.50	3.90	11.3	7.2	0.13	0.47	0.29
Hanover, Holstein, Pomerania.....	3.93	4.81	4.20	5.07	6.7	5.9	0.25	0.26	0.25

<sup>1</sup> Documents sur les falsifications des matières alimentaires, et sur les travaux du laboratoire municipal, deuxième rapport, Paris, 1885.

*Average of the contents of alcohol, extract, and ash in various beers, &c.—Continued.*

	Per cent. alcohol by volume.			Per cent. extract.			Per cent. ash.		
	Min.	Max.	Mean.	Min.	Max.	Mean.	Min.	Max.	Mean.
<i>Austrian beers.</i>									
Vienna, Moravia .....	3.00	4.50	3.5	5.00	8.0	6.1	0.18	0.28	0.20
Bohemia .....	3.29	4.59	3.6	4.10	5.9	4.7	0.17	0.28	0.20
<i>English beers.</i>									
Ale .....	5.0	8.5	6.2	4.8	14.0	6.6	-----	-----	-----
Porter .....	4.0	6.9	6.4	5.9	7.4	6.5	-----	-----	-----
<i>Belgian beers.</i>									
Lambick .....	4.5	7.7	6.02	2.07	5.6	3.7	0.30	0.35	0.32
Faro .....	2.5	4.9	4.15	2.90	5.1	4.2	0.29	-----	-----
Bière d'orge .....	3.0	4.9	4.35	2.70	4.5	3.4	0.29	-----	-----
Uxter .....	2.7	3.2	3.00	4.00	5.0	4.4	-----	-----	-----
Bières blanches .....	2.2	4.4	-----	3.00	5.0	4.0	-----	-----	-----
Bières diverses .....	3.5	8.4	5.80	3.10	8.0	5.5	-----	-----	-----

#### COMPOSITION OF AMERICAN BEER.

But very little work has been done on American beers; they seem to have shared with other dietary articles the general indifference of the American public to the composition of their food and drink.

A very extensive series of analyses was made in the State of New York in 1885, under the authority of the State Board of Health, by Dr. F. E. Englehardt, and outside of this I have been able to find very few published analyses of American beers.

Dr. Englehardt's analyses were made upon a very large number of samples, 476 in all, which were collected from all over the State, and were intended to furnish a good average representation of the beer retailed in the State. The samples included various kinds of malt liquor, porters, ales, and a weak beer sold under the name of weiss beer. Unfortunately no arrangement of the analyses was made with a view to showing the composition of various kinds, as the examination was made principally with reference to the adulteration, so all varieties are tabulated together. The following averages I have had compiled from his table by the Statistical Division of this Department, only excepting a few samples which he has indicated as being imported:

*Average composition of American malt liquors, as shown by analyses made for New York State Board of Health by F. E. Englehardt, Ph. D.*

Kind.	Specific gravity.	Alcohol	Extract.	Ash.	Phosphoric acid.
		by weight.			
Lager, 172 samples .....	1.016	3.754	5.864	.259	.0964
Ale, 199 samples .....	1.013	4.622	5.423	.307	.0832
Porter, 70 samples .....	1.015	4.462	6.003	.345	.0942
Weiss, 28 samples .....	1.006	1.732	2.356	.189	.0491

The maximum and minimum content of alcohol, extract, and ash in the same samples is as follows :

Kind.	Maximum.			Minimum.		
	Alcohol by weight.	Extract.	Ash.	Alcohol by weight.	Extract.	Ash.
Lager . . . .	7.061	9.647	.412	.677	3.655	.172
Ale . . . .	8.994	9.501	.552	2.410	2.705	.197
Porter . . . .	6.695	11.783	.557	1.671	2.843	.170
Weiss . . . .	3.179	4.143	.468	.755	1.277	.069

These analyses show great lack of uniformity of composition in the different varieties of malt liquor, but it should be remembered that the samples were collected with a view to ascertaining the extent of adulteration, and many samples were found to be sophisticated in one way or another. Especially in the case of the content of ash the average of these samples does not give the average composition of American beers, for many of these ashes were found to consist principally of salt.

Following is the average of nineteen analyses made by the same chemist for the New York Board of Health in 1882 :

Specific gravity . . . .	1.0162
Alcohol by weight . . . .	2.78
Extractive matter . . . .	6.047
Sugar . . . .	1.521
Free acid calculated as lactic . . . .	.189
Ash . . . .	.305
Phosphoric acid . . . .	.105

The following analyses of four samples of beer sold in Indianapolis, Ind., were made by Mr. J. N. Hurty :<sup>1</sup>

	Specific grav- ity.	Extract.	Sugar.	Dextrin.	Albuminoids.	Bitter matter.	Acidity calen- lated as acetic.	Alcohol.
Milwaukee . . . .	1.0174	7.312	1.895	3.880	.037	1.530	.159	5.350
Lieber's . . . .	1.0220	5.988	3.126	2.644	.016	.202	.281	9.900
Maus's . . . .	1.0180	6.330	4.060	2.060	.006	.118	.309	5.384
Schmidt's . . . .	1.0172	5.816	3.440	2.283	.014	.074	.080	4.640
Average . . . .	1.0180	6.361	3.130	2.719	.018	.481	.207	6.341

The analyst does not state whether the percentage of alcohol is by weight or volume, but on account of its being so high, presumably it is the latter.

*Analyses made in 1873 of New York beers for the "Moderation Society" by Professor Doremus.<sup>1</sup>*

Specific grav- ity.	Alcohol.		Extractive matter.		Water.		Non-nitrogen- ized.	Albuminoids.	Ash.	Phosphoric acid.		Sugar.		Adulterations or deleteri- ous matter.
	Pr. ct.	Pr. ct.	Per cent.	Per cent.	Per cent.	Per cent.				Per cent.	Per cent.	Per cent.	Per cent.	
1.0280	4.00	8.5215	87.40	7.1605	0.8750	0.2970	0.1890	0.1300		2.2105				None.
1.0315	2.00	8.4580	89.50	7.0695	0.8750	0.3425	0.1710	0.1610		2.2034				None.
1.0175	4.60	6.5570	88.80	5.4080	0.7000	0.3050	0.0810	0.1775		1.2285				None.
1.0275	2.80	8.3410	88.80	7.3530	0.6125	0.2675	0.1080	0.0575		2.7826				None.
1.0330	3.40	-----	86.60	8.9035	0.6300	0.3740	0.0900	0.1200		3.0250				None.
1.0210	4.60	6.8280	88.50	5.7170	0.6375	0.3385	0.1350	0.1875		2.6472				None.
1.0250	2.50	6.9740	90.40	5.8565	0.6650	0.2905	0.1620	0.0875		0.9726				None.
1.0180	2.80	6.8600	90.30	5.6705	0.6375	0.4620	0.1625	0.0625		1.0338				None.
										Phos. acid.	Malt- ose.	Dex- trose.	Dex- trine.	
1.0150	3.10	5.1840	91.60	4.0960	1.6120	0.2500	0.2230	0.1000	0.5470	0.5090	2.6260			None.
1.0125	5.20	5.4660	89.20	4.1940	0.7870	0.2590	0.2250	0.1000	0.3120	1.1220	2.3010			None.
1.0155	4.30	6.4040	89.60	4.6870	0.8400	0.2410	0.2700	0.1120	0.2040	1.0580	2.6980			None.
1.0120	5.20	5.0740	89.60	3.7620	0.7700	0.2720	0.2700	0.1170	0.7540	0.3910	1.9670			None.
1.0150	4.60	6.4260	88.80	5.6680	0.4530	0.2100	0.1620	0.0850	0.6890	0.8450	3.5760			None.
1.0150	4.60	6.1080	89.20	4.7000	0.8570	0.2950	0.3150	0.1200	0.0950	0.2060	2.3750			None.

*Analyses made by Professor Eglehardt for same society.*

Specific grav- ity.	Alcohol.		Extractive or solid matter.	Water.		Maltose sugar.	Dextrine.	Albuminoids.	Ash.	Lactic acid.	Phosphoric acid.	Adulterations or deleteri- ous matter.
	Per cent.	Per cent.		Per cent.	Per cent.							
1.0145	4.25	5.750	90.00	1.420	2.745	0.599	0.153	0.302	0.093	None.		
1.0150	3.70	5.670	90.60	1.426	2.680	0.677	0.174	0.279	0.107	None.		
1.0156	3.70	5.770	90.50	1.590	2.510	0.706	0.150	0.309	0.107	None.		
1.0134	4.25	5.350	90.30	1.285	2.563	0.319	0.212	0.310	0.318	None.		
1.0197	3.50	6.470	89.90	1.434	3.150	0.869	0.150	0.321	0.076	None.		
1.0187	3.70	6.462	89.70	1.563	3.303	0.760	0.202	0.311	0.078	None.		
1.0120	4.10	4.297	90.60	0.913	2.037	0.624	0.150	0.321	0.076	None.		
1.0175	4.30	6.586	89.00	1.435	3.141	0.657	0.123	0.337	0.097	None.		
1.0174	4.20	0.380	89.40	1.448	3.004	0.810	0.212	0.330	0.098	None.		
1.0162	4.25	6.209	89.50	1.485	3.033	0.700	0.212	0.352	0.098	None.		

The tables furnished above constitute about all the analyses of American beer I have been able to find in the literature I have access to. Probably more have been published in the trade journals.

#### ANALYSES OF BEERS BY THE UNITED STATES DEPARTMENT OF AGRICULTURE.

The analyses made by this Department comprise 32 samples, this being about all the different brands and varieties of beers of domestic manufacture obtainable in Washington. The investigation was made principally with a view to ascertain the extent and nature of their adulteration, if any, and especially the use of antiseptic and preservative agents. As a basis for determining adulteration, however, it is necessary to know the normal or average composition, so a fairly com-

plete analysis of all samples examined has been made. The intention of the investigation was not so much to make a very extensive series of analyses as to establish definite methods of analysis for the guidance of analysts of state boards of health or similar bodies, whose province it is more especially to investigate the extent of adulteration prevailing in their States by the examination of large numbers of samples.

#### SAMPLES.

The malt liquors used as samples were all purchased in Washington, D. C., and included the various popular brands made in Milwaukee, Cincinnati, Philadelphia, New York, &c., which are sold all over the country, as well as the product of the few local brewers. Some were obtained from wholesale dealers, but the majority were purchased in retail saloons and groceries, without statement of the purpose for which they were intended. All the draft beers were obtained in this way.

A few English and German beers and ales were analyzed for comparison.

Designation.	Manufactured in—	Number of analysis.	Specific gravity.	Alcohol by weight.	Oligesimal gravity.	Asp.	Dextrin.	Reducing sugar.	Attribumolids.	Phosphate acids.	Carboxylic acids.	Remarks.	
Lager beer, bottled.	Milwaukee, Wis. ....	4500	4.28	4.39	4.18	1.0505	.196	1.10	.511	.057	.411		
Export beer, bottled.	do .....	4801	2	0.1040	4.42	5.5	5.40	1.0537	2.63	.400	.057	Salicylated.	
Lager beer, bottled.	Alexandria, Va. ....	4802	3	4.55	5.71	5.71	1.0517	.369	1.06	.056	.300	Salicylated.	
Lager beer, bottled.	Washington, D. C. ....	4803	4	0.1043	4.18	5.24	5.05	1.0532	2.04	.681	.074	0.91	0.480
Lager beer, bottled.	Cincinnati, Ohio. ....	4804	5	0.1010	5.53	6.94	4.55	1.0528	0.08	.650	.059	.415	
do .....	Saint Louis, Mo. ....	4805	6	0.1018	4.40	5.6	6.15	1.0530	.240	0.94	.225	.513	.073
do .....	Philadelphia, Pa. ....	4806	7	0.1047	4.29	5.39	5.22	1.0549	.241	1.16	2.54	.667	.071
do .....	do .....	4807	8	0.1047	4.35	5.47	5.09	1.0549	.272	1.37	2.50	.558	.078
do .....	do .....	4808	9	0.1081	5.21	5.63	5.94	1.0609	.231	2.14	2.57	.521	.219
do .....	do .....	4810	10	0.0241	3.84	4.78	7.05	1.0601	.222	2.81	3.09	.519	.078
do .....	do .....	4811	11	0.1032	4.36	5.47	4.63	1.0539	.265	1.17	2.52	.636	.046
do .....	do .....	4812	12	0.1046	4.29	5.39	5.18	1.0545	.236	1.22	2.21	.669	.044
do .....	do .....	4813	13	0.1069	4.63	5.78	5.86	1.0607	.235	2.37	2.20	.456	.074
do .....	do .....	4814	14	0.1037	4.71	5.86	4.91	1.0535	.263	1.10	2.40	.619	.068
do .....	do .....	4815	15	0.1040	4.30	5.39	4.83	1.0538	.292	1.49	1.45	.613	.087
do .....	do .....	4816	16	0.1081	4.86	4.85	5.62	1.0565	.312	1.52	2.59	.619	.067
do .....	do .....	4817	17	0.1078	4.28	5.39	6.64	1.0527	.182	2.17	2.75	.463	.067
do .....	do .....	4818	18	0.0203	4.68	5.86	5.82	1.0650	.222	2.51	2.58	.675	.046
do .....	do .....	4819	19	0.0059	6.24	7.74	3.46	1.0647	.401	0.39	.90	.531	.232
do .....	do .....	4820	20	0.0095	5.66	7.09	4.42	1.0633	.309	0.49	.20	.500	.117
do .....	do .....	4821	21	0.1047	6.13	7.66	5.90	1.0728	.371	0.57	.276	.763	.151
do .....	Boston, Mass. ....	4822	22	0.0077	5.30	6.63	3.94	1.0557	.328	1.06	1.62	.536	.107
do .....	Dresden. ....	4823	23	0.0036	5.38	6.71	3.05	1.0543	.162	0.69	1.36	.268	.045
do .....	Bavaria. ....	4824	24	0.1017	3.86	4.86	6.24	1.0533	.190	0.71	3.22	.419	.085
do .....	Milwaukee, Wis. ....	4825	25	0.1050	4.59	5.71	5.38	1.0581	.194	1.87	2.46	.425	.071
do .....	Philadelphia, Pa. ....	4826	26	0.1071	5.25	6.55	6.02	1.0668	.331	1.40	2.80	.569	.034
do .....	England. ....	4827	27	0.1025	6.92	8.63	6.02	1.0638	.322	1.00	1.71	.721	.037
do .....	do .....	4828	28	0.0209	6.89	8.10	5.19	1.0781	.412	0.92	1.99	.382	.441
do .....	do .....	4829	29	0.1083	4.22	5.32	5.88	1.0736	.482	2.88	7.02	.763	.166
do .....	do .....	4843	30	0.1083	4.22	5.32	5.84	1.0587	.203	1.78	3.12	.413	.052
do .....	do .....	4844	31	0.1083	4.16	5.24	5.88	1.0562	.224	1.82	3.04	.406	.071
do .....	do .....	4845	32	0.1087	5.06	6.32	6.26	1.0660	.346	1.75	2.87	.550	.074
		.....	1.0161	4.63	5.79	5.53	1.0597	.279	1.65	2.33	.563	.077	Average (28 samples) .398

In the averages the five samples of foreign beer were omitted.

## METHODS OF ANALYSIS.

In the work on malt liquors an endeavor has been made to simplify the analyses as much as possible, and various methods have been tried with this end in view. The various processes given are believed to be the best possible for combining rapidity of execution with sufficient accuracy of results.

The necessary determinations may be conveniently divided into two classes :

1. The analysis of the sample proper, comprising determination of the density, alcohol, extract or total solids, original gravity, saccharine matter, albuminoids, free acid (fixed), free acid (volatile), ash, glycerine, phosphoric acid, and carbonic acid.

2. The processes for the detection of adulteration, comprising a search for substitutes for malt, substitutes for hops, preserving agents (salicylic acid, borax, sulphites), and mineral additions.

## ALCOHOL.

The estimation of alcohol in beers and wines is generally made in one of two ways, either by direct distillation and determining the alcohol in the distillate, or, indirectly, by evaporating the alcohol from a sample and obtaining the per cent. from the difference in specific gravity of the sample before and after the alcohol has been driven off. Authorities differ as to the accuracy of the indirect method, some even holding it to give better results than the direct estimation. It is generally recommended to use both methods, as the one gives a check over the other, and it is very easy to carry both on together, as the same sample which is used for the distillation can be used for the determination of the density of the de-alcoholized solution, provided no tannin is used. I much prefer the distillation method, and have adopted the results by it in the tables.

I have almost invariably found that during the distillation a precipitation of flocculent albuminous matter takes place in the flask, evidently bodies which are rendered insoluble, either by the evaporation of the alcohol or acetic acid, or by the heating of the solution, and it would seem obvious that this separation of solids from the solution would vitiate more or less the results by the indirect method.

For the distillation method 100cc. of the sample, freed from carbonic acid by shaking, are measured out, rinsed into a flask with about 50cc. water, the latter connected with a Liebig's condenser, and 100cc. distilled off. The sample and distillate should be measured at the same temperature. The specific gravity of the distillate is then obtained by means of an accurately tared pycnometer, preferably one carrying a thermometer, so that the weight may be taken at exactly 15.5° C. The per cent. of alcohol in the distillate is then obtained by reference to a table giving the per cent. of alcohol in solutions of different specific

gravities, of which tables Helmer's are the best in use. This per cent. multiplied by the specific gravity of the distillate, and the result divided by the specific gravity of the original sample, gives the per cent. of alcohol by weight contained in the latter. The accuracy of the operation is increased by weighing the sample taken, and also the distillate. Then the weight of the distillate multiplied by the per cent. of alcohol corresponding to its specific gravity as found in the table, and the result divided by the weight of the sample taken, gives the per cent. of alcohol by weight contained in the latter.

For the indirect method it is necessary to estimate accurately the specific gravity of the original beer, thoroughly freed of carbonic acid by shaking and standing; then 100cc. or any convenient quantity is measured out, evaporated to half its bulk, cooled, and made up to its original volume with water, taking care to have the solution at the same temperature as the sample when first measured. The specific gravity of the de-alcoholized liquor is taken. Then the specific gravity of the original sample divided by the specific gravity of the de-alcoholized solution gives the specific gravity of the alcohol evaporated, from which figure the per cent. of alcohol is ascertained by reference to the table. The same sample which is used for distilling can be very conveniently used for this determination as well.

## Hehner's alcohol table.

Specific gravity at 15.5° C.	Alcohol by weight.	Alcohol by volume.	Specific gravity at 15.5° C.	Alcohol by weight.	Alcohol by volume.	Specific gravity at 15.5° C.	Alcohol by weight.	Alcohol by volume.	Specific gravity at 15.5° C.	Alcohol by weight.	Alcohol by volume.
1.0000	0.00	0.00	0.9999	0.05	0.07	0.9929	4.06	5.08	0.9839	8.71	10.82
8	0.11	0.13	8	0.16	0.20	8	4.12	5.16	8	8.79	10.91
7	0.16	0.20	7	0.21	0.26	6	4.19	5.24	7	8.86	11.00
6	0.21	0.26	6	0.26	0.33	5	4.25	5.32	6	8.93	11.08
5	0.26	0.33	5	0.31	0.38	5	4.31	5.39	5	9.00	11.17
4	0.32	0.40	4	0.37	0.44	4	4.37	5.47	4	9.07	11.26
3	0.37	0.46	3	0.42	0.53	2	4.44	5.55	3	9.14	11.35
2	0.42	0.53	1	0.47	0.60	1	4.56	5.71	2	9.21	11.44
1	0.47	0.66	0	0.53	0.66	0	4.63	5.78	0	9.36	11.52
0	0.53								0	11.61	14.82
0.9989	0.58	0.73	0.9919	4.60	5.85	0.9819	9.43	11.70	0.9779	14.91	18.36
8	0.63	0.79	8	4.75	5.94	8	9.50	11.79	8	15.00	18.48
7	0.68	0.86	7	4.81	6.02	7	9.57	11.87	7	15.03	18.58
6	0.74	0.93	6	4.87	6.10	6	9.61	11.96	6	15.17	18.68
5	0.79	0.99	5	4.94	6.17	5	9.71	12.05	5	15.25	18.78
4	0.84	1.06	4	5.00	6.24	4	9.79	12.13	4	15.33	18.88
3	0.89	1.13	3	5.06	6.32	3	9.86	12.22	3	15.42	18.98
2	0.95	1.19	2	5.12	6.40	2	9.93	12.31	2	15.50	19.08
1	1.00	1.26	1	5.19	6.48	1	10.00	12.40	1	15.58	19.18
0	1.06	1.34	0	5.25	6.55	0	10.03	12.49	0	15.67	19.28
0.9979	1.12	1.42	0.9909	5.31	6.63	0.9839	10.15	12.58	0.9769	15.75	19.39
8	1.19	1.49	8	5.37	6.71	8	10.23	12.68	8	15.83	19.49
7	1.25	1.57	7	5.44	6.78	7	10.31	12.77	7	15.92	19.59
6	1.31	1.65	6	5.50	6.86	6	10.38	12.87	6	16.00	19.68
5	1.37	1.73	5	5.56	6.94	5	10.46	12.96	5	16.08	19.78
4	1.44	1.81	4	5.62	7.01	4	10.51	13.05	4	16.15	19.87
3	1.50	1.88	3	5.69	7.09	3	10.62	13.15	3	16.23	19.96
2	1.56	1.96	2	5.75	7.17	2	10.69	13.24	2	16.31	20.06
1	1.62	2.04	1	5.81	7.25	1	10.77	13.34	1	16.38	20.15
0	1.69	2.12	0	5.87	7.32	0	10.85	13.43	0	16.46	20.24
0.9969	1.75	2.20	0.9809	5.94	7.40	0.9829	10.92	13.52	0.9755	16.85	20.71
8	1.81	2.27	8	6.00	7.48	8	11.00	13.62	0.9750	17.25	21.19
7	1.87	2.35	7	6.07	7.57	7	11.08	13.71	0.9745	17.67	21.69
6	1.94	2.43	6	6.14	7.66	6	11.15	13.81	0.9740	18.08	22.18
5	2.00	2.51	5	6.21	7.74	5	11.23	13.90	0.9735	18.46	22.61
4	2.06	2.58	4	6.28	7.83	4	11.31	13.99	0.9730	18.85	23.10
3	2.11	2.62	3	6.36	7.92	3	11.38	14.09	0.9725	19.25	23.58
2	2.17	2.72	2	6.43	8.01	2	11.46	14.18	0.9720	19.67	24.08
1	2.22	2.79	1	6.50	8.10	1	11.54	14.27	0.9715	20.08	24.58
0	2.28	2.86	0	6.57	8.18	0	11.62	14.37	0.9710	20.50	25.07
0.9959	2.33	2.93	0.9889	6.64	8.27	0.9819	11.60	14.46			
8	2.39	3.00	8	6.71	8.36	8	11.77	14.56			
7	2.44	3.07	7	6.78	8.45	7	11.85	14.65			
6	2.50	3.14	6	6.86	8.54	6	11.92	14.74			
5	2.56	3.21	5	6.93	8.63	5	12.00	14.84			
4	2.61	3.28	4	7.00	8.72	4	12.08	14.93			
3	2.67	3.35	3	7.07	8.80	3	12.15	15.02			
2	2.72	3.42	2	7.13	8.88	2	12.23	15.12			
1	2.78	3.49	1	7.20	8.96	1	12.31	15.21			
0	2.83	3.55	0	7.27	9.04	0	12.38	15.30			
0.9949	2.89	3.62	0.9879	7.33	9.13	0.9809	12.46	15.40			
8	2.94	3.69	8	7.40	9.21	8	12.54	15.49			
7	3.00	3.76	7	7.47	9.29	7	12.62	15.58			
6	3.06	3.83	6	7.53	9.37	6	12.69	15.68			
5	3.12	3.90	5	7.60	9.45	5	12.77	15.77			
4	3.18	3.98	4	7.67	9.54	4	12.85	15.86			
3	3.24	4.05	3	7.73	9.62	3	12.92	15.96			
2	3.29	4.12	2	7.80	9.70	2	13.00	16.05			
1	3.35	4.20	1	7.87	9.78	1	13.08	16.15			
0	3.41	4.27	0	7.93	9.86	0	13.15	16.24			
0.9939	3.47	4.34	0.9869	8.00	9.95	0.9799	13.23	16.33			
8	3.53	4.42	8	8.07	10.03	8	13.31	16.43			
7	3.59	4.49	7	8.14	10.12	7	13.38	16.52			
6	3.65	4.56	6	8.21	10.21	6	13.46	16.61			
5	3.71	4.63	5	8.29	10.30	5	13.54	16.70			
4	3.76	4.71	4	8.36	10.38	4	13.62	16.80			
3	3.82	4.78	3	8.43	10.47	3	13.69	16.89			
2	3.88	4.85	2	8.50	10.56	2	13.77	16.98			
1	3.94	4.93	1	8.57	10.65	1	13.85	17.08			
0	4.00	5.00	0	8.64	10.73	0	13.92	17.17			

## EXTRACT OR TOTAL SOLIDS.

This can also be obtained either directly, by weighing or measuring a sample into an open dish, driving off the moisture at 100° C. until a constant weight is obtained, or indirectly, by calculation from the specific gravity of the de-aleoholized solution. For the direct estimation a small quantity should be weighed out; 10 grams is quite sufficient, and 5 grams gives still better results. This is allowed to run out into a thin film on the bottom of a shallow dish having an area of several square inches. In this shape it is very readily dried to a constant weight at 100° C., while if a larger quantity is taken this becomes quite difficult, and it is necessary to use a higher temperature.<sup>1</sup>

In case the indirect method is used the per cent. of extract may be obtained from one of the various tables in use or by dividing the difference between the specific gravity and 1.000 by the factor 3.86. The result obtained is the number of grams of solid matter in 100cc. of the beer, and must be divided by the specific gravity of the original beer to get the exact per cent.

## ORIGINAL GRAVITY.

The "original gravity" of beer is the specific gravity of the wort from which it was made, before fermentation. This is ascertained by computation from data given by the aleoholic content and the malt extract contained in the de-alcoholized liquid. The specific gravity of the aleoholic distillate (or the specific gravity of the alcohol evaporated, if the indirect method is used) when subtracted from 1.000 gives a number called the "spirit indication." The degrees of gravity lost are then ascertained by reference to the table given below. The degrees found are added to the specific gravity of the de-alcoholized beer, and the number thus obtained is the original gravity.

The following table was calculated by Graham, Hofmann, and Redwood from actual experiments on malt worts fermented under normal conditions:

<sup>1</sup> The following duplicate determinations show very satisfactory agreement in the results obtained by using a small quantity for drying:

	No. 4800.	No. 4801.	No. 4804.	No. 4817.
1.....	4.25	5.40	4.52	4.67
2.....	4.16	5.40	4.58	4.62

Degrees of spirit indication.	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
0	....	.3	.6	.9	1.2	1.5	1.8	2.1	2.4	2.7
1	3.0	3.3	3.7	4.1	4.4	4.8	5.1	5.5	5.9	6.2
2	6.6	7.0	7.4	7.8	8.2	8.6	9.0	9.4	9.8	10.2
3	10.7	11.1	11.5	12.0	12.4	12.9	13.3	13.8	14.2	14.7
4	15.1	15.5	16.0	16.4	16.8	17.3	17.7	18.2	18.6	19.1
5	19.5	19.9	20.4	20.9	21.3	21.8	22.2	22.7	23.1	23.6
6	24.1	24.6	25.0	25.5	26.0	26.4	26.9	27.4	27.8	28.3
7	28.8	29.2	29.7	30.2	30.7	31.2	31.7	32.2	32.7	33.2
8	33.7	34.3	34.8	35.4	35.9	36.5	37.0	37.5	38.0	38.6
9	39.1	39.7	40.2	40.7	41.2	41.7	42.2	42.7	43.2	43.7
10	44.2	44.7	45.1	45.6	46.0	46.5	47.0	47.5	48.0	48.5
11	49.0	49.6	50.1	50.6	51.2	51.7	52.2	52.7	53.3	53.8
12	54.3	54.9	55.4	55.9	56.4	56.9	57.4	57.9	58.4	58.9
13	59.4	60.0	60.5	61.1	61.6	62.2	62.7	63.3	63.8	64.3
14	64.8	65.4	65.9	66.5	67.1	67.6	68.2	68.7	69.3	69.9
15	70.5	71.1	71.7	72.3	72.9	73.5	74.1	74.4	75.3	75.9

A content of acetic acid above 0.1 per cent. somewhat vitiates the results given by the table, and a correction is necessary. The manner of making this correction may be ascertained by consulting Allen,<sup>1</sup> or any of the standard works, which give also tables for the calculation of the actual weight of malt used in the wort.

Below is given a table showing the results obtained from the samples analyzed, by the direct and indirect methods :

*Comparison of direct and indirect methods of estimating alcohol and extract in malt liquors.*

Serial No.	Number of analysis.	Alcohol di-rect.	Alcohol by calculation.	Extract di-rect.	Extract by calculation.	Original gravity by distillation.	Original gravity by evapo-ration.
		Per ct.	Per et.	Per ct.	Per ct.	Per ct.	Per ct.
4800.....	1	4.28	4.33	4.18	4.56	1.0505	1.0506
4801.....	2	4.42	.....	5.40	6.03	1.0573	.....
4802.....	3	4.55	4.51	5.71	6.44	1.0607	1.0596
4803.....	4	4.18	3.96	5.05	5.52	1.0533	1.0516
4804.....	5	5.53	5.39	4.55	5.03	1.0628	1.0616
4805.....	6	4.40	4.08	6.15	6.44	1.0590	1.0562
4806.....	7	4.29	4.08	5.22	5.67	1.0549	1.0531
4807.....	8	4.35	3.84	5.09	5.54	1.0549	1.0507
4808.....	9	4.52	4.39	5.94	6.64	1.0609	1.0594
4810.....	10	3.84	3.67	7.05	7.82	1.0601	1.0584
4811.....	11	4.36	4.08	4.63	5.29	1.0539	1.0516
4812.....	12	4.29	3.90	5.18	5.57	1.0545	1.0513
4813.....	13	4.63	4.33	5.86	6.32	1.0607	1.0576
4814.....	14	4.71	4.57	4.91	5.62	1.0585	1.0569
4815.....	15	4.30	3.90	4.83	5.39	1.0538	1.0506
4816.....	16	3.86	.....	5.62	.....	.....	.....
4817.....	17	4.28	3.90	4.64	6.36	1.0577	1.0545
4818.....	18	4.68	4.33	6.82	7.24	1.0650	1.0613
4819.....	19	6.24	5.83	3.46	4.17	1.0647	1.0616
4820.....	20	5.66	5.39	4.42	4.90	1.0633	1.0611
4821.....	21	6.13	5.77	5.90	6.33	1.0728	1.0697
4822.....	22	5.30	5.51	3.94	4.54	1.0587	1.0665
4823.....	23	5.38	5.07	3.05	3.25	1.0543	1.0520
4824.....	24	3.86	3.43	6.24	6.63	1.0553	1.0517
4825.....	25	4.59	4.20	5.38	5.80	1.0581	1.0545
4826.....	26	5.25	4.76	6.02	6.55	1.0669	1.0623
4827.....	27	6.92	6.42	5.55	6.09	1.0781	1.0741
4828.....	28	4.89	4.39	8.19	8.83	1.0736	1.0683
4842.....	29	4.22	3.49	5.88	6.31	1.0570	1.0509
4843.....	30	4.22	3.37	5.84	6.23	1.0567	1.0493
4844.....	31	4.16	3.37	5.88	6.26	1.0563	1.0494
4845.....	32	5.06	4.14	6.26	6.69	1.0660	1.0576
Average .....		4.73	4.41	5.40	5.94	1.0600	1.0569

<sup>1</sup> Commercial Organic Analysis, 2d edit., Vol. I.

## SACCHARINE MATTER.

The saccharine matter in beer consists principally of maltose and dextrin, with probably a small proportion of dextrose. The greater part of the entire extract is composed of these different carbohydrates. The proportion of maltose to dextrin is of considerable importance in judging of the quality of a beer. The dextrin contributes to the "fullness" (vollmündigkeit) of the taste, and a larger proportion of dextrin to extract makes a beer of good "body." The method of brewing is said to have an important influence upon the relative proportions of maltose and dextrin. The determination of the percentages of these sugars is, therefore, quite an important one as showing the nature and quality of the sample, though not of much utility in detecting adulterations. The usual method is to estimate the maltose by Fehling's solution, and the dextrin, if it is reported at all, is obtained by difference from the total solids. So little is known of the saccharine bodies already existing in the grain and of the products of the conversion of starch into sugars, that their separate estimation is rather unsatisfactory.<sup>1</sup> The dextrin may be determined directly by precipitating with alcohol, washing and weighing. The results are sufficiently accurate for commercial purposes, according to J. West Knights,<sup>2</sup> who employed it upon worts.

Graham estimates the maltose and dextrin in beer worts by the use of Fehling's solution before and after inversion.<sup>3</sup> The cupric oxide reducing power is determined gravimetrically, 10cc. are measured out and diluted to 100cc.; 20cc. of this solution are used to 30cc. Fehling's solution. The weight of CuO obtained, multiplied by 0.7314 gives the amount of maltose in the quantity of diluted liquid employed. The maltose having been determined, 10cc. of the wort are mixed with 3cc. of sulphuric acid, diluted to 100cc. and inverted by heating to 100° C., for 3 to 4 hours in a flask furnished with a long tube. The volume of the solution is again made up to 100cc., 10cc. carefully measured or weighed, neutralized with sodium carbonate, and the reducing power determined by heating with Fehling's solution, in the same way as before. The percentage of dextrin is then calculated as follows: Multiply half the weight of CuO, obtained by the action of Fehling's solution on 2cc. of the original wort by 1.72, and subtract the product from the CuO obtained from the inverted solution (= 1cc. of the original). The difference multiplied by 40.8 gives the *grams* of dextrin in 100cc. of the original wort.

This method was applied to the samples analyzed with very unsatisfactory results. In some cases the sum of the maltose and dextrin determined in this way exceeded the amount of total extract, while in other cases the per cent. of dextrin was a minus quantity. This method was therefore abandoned. The results given were obtained by

<sup>1</sup> Recent work on this problem has been published by O'Sullivan, *Jour. Chem. Soc.*, Jan., 1886, p. 58.

<sup>2</sup> Allen's *Organic Analysis* 1, 274.

<sup>3</sup> *Analyst* 7, 211.

the following method taken from Allen.<sup>1</sup> The maltose was estimated by Fehling's solution, volumetrically, by the method in use in this laboratory for the determination of cupric oxide reducing power.<sup>2</sup> The polarization is then observed in the original beer, the clarification being accomplished by means of lead acetate. The reading is taken on the circular scale, and constitutes the total circular rotation. The number of grams of maltose in 100cc. of the beer having been ascertained from the results obtained with Fehling's solution, it is multiplied by 2.78, which gives the rotation due to maltose; this result is deducted from the total rotation, which gives the rotation due to dextrin. The angle found, divided by 3.86 or multiplied by .259, gives the grams of dextrin in 100cc of the solution. These figures are based on the assumption that the polariscope used is one in which monochromatic light is employed, and that the liquid is observed in a tube 200 millimeters in length. By dividing the grams per 100cc. by the density of the beer, the actual percentage of maltose and dextrin will be ascertained.

As considerable interest is attached to the nature of the polarizing bodies in malt liquors, I append the polarization given by the samples examined. It is a very easy and satisfactory determination to make, the beers being readily clarified by acetate of lead, giving bright, clear solutions. The figures given are in divisions of the cane-sugar scale, and for the normal beer, the dilution of one-tenth incident upon the addition of the lead being corrected by reading in a 220-millimeter tube; the instrument employed was a Laurent polariscope, in which monochromatic light is employed.

Serial number.	Normal polarization.	Serial number.	Normal polarization.
4800	○	4817	77.6
4801	42.3	4818	79.4
4802	61.3	4819	18.7
4803	66.6	4820	45.8
4804	33.9	4821	57.2
4805	52.5	4822	42.8
4806	74.4	4823	33.1
4807	60.4	4824	82.5
4808	50.4	4825	68.6
4810	74.8	4826	69.7
4811	93.0	4827	47.8
4812	47.5	4828	87.6
4813	55.8	4842	75.4
4814	72.2	4843	78.2
4815	57.5	4844	78.8
4816	45.5	4845	74.8
	65.8		

The polarization was in all cases right-handed.

#### ALBUMINOID MATTERS.

The albuminoids were determined by weighing 10 grams of the beer into a schälchen, evaporating to dryness, and burning with soda lime in the usual way. The nitrogen found  $\times 6.25$  is given as the per cent.

<sup>1</sup> Page 275.

<sup>2</sup> Fully described in Bulletin No. 15, page 32.

age of albuminoids. Graham determines the nitrogen by "Wanklyuizing" the beer as in water analysis. The determination of the nitrogenous matters in beer is important in enabling the analyst to form an opinion on the question whether substitutes for malt were used in its manufacture, as saccharine matter derived from sources other than grain have little nitrogenous content. Too large an amount of albuminous matter is injurious to the keeping qualities of the beer.

The Kjeldahl<sup>1</sup> method, which was originally devised by its inventor for application to the determination of nitrogen in beers and worts, may also be used.

#### FREE ACIDS.

The ideas of chemists in regard to the nature of the acidity of normal beer have undergone considerable change in the last two or three years. It was formerly considered to be principally due to the presence of lactic acid, with a small quantity of succinic and other acids, but is now considered to be due, for the greater part, to acid phosphates. Acetic acid is present only to a very limited extent in normal beer, its presence in any considerable quantities being proof of the "souring" of the beer. Ott<sup>2</sup> has shown the difficulty of ascertaining the exact point of neutralization in beer, as by the addition of alkali to the acid phosphates the reaction becomes "amphoteric" from the simultaneous formation of both primary and secondary phosphates, and the establishment of the point of neutralization by the reaction with litmus paper is very difficult. No better means of determining acidity in beer has been proposed, however, and I have used the ordinary method of adding standard alkali until a drop placed on neutral litmus paper produces no alteration of color. 50cc are conveniently taken for this determination, freed from carbonic acid and titrated with dezinormal alkali. The acidity can be given directly as cubic centimeters of normal alkali required for 100cc. of beer, or reckoned as lactic acid. The volatile acids, when it is necessary to determine them separately, as in the case of soured beer, may be best estimated by distillation in a current of steam, as described under wine, all methods for their estimation by difference by evaporating the beer to dryness and titrating the residue having been shown to be faulty.

The Bavarian chemists,<sup>3</sup> at their last meeting in 1886, adopted the figure of 3cc. normal alkali for 100cc. beer as a maximum limit for a normal beer.

#### ASH.

The ash may best be determined by burning the residue from 100 cc. of beer at a very low red heat in a muffle. The ash obtained

<sup>1</sup> Zeit. Anal. Chem. 1883, 366, for reference to the modifications of the method, see Bulletin No. 12, U. S. Depart. Agriculture, Division of Chemistry, p. 55.

<sup>2</sup> Zeit. Anal. Chem. 24, 132.

<sup>3</sup> Ber. ü. d. finnste Ver. der Freien Verein Bay. Ver. d. angewandten Chem. zu Würzburg, Berlin, 1887.

should be subjected to a qualitative examination, with a view to ascertaining if any mineral substances have been added to the beer.

#### GLYCERINE.

The estimation of glycerine is a troublesome and unsatisfactory determination to make, and as the information obtained in the case of beer is not sufficient to repay the labor of the analysis, except in special cases, I have omitted it.

The following method is used by the Bavarian chemists: 50cc. of beer are treated with about 3 grams of quicklime, evaporated to a sirup, then mixed with about 10 grams coarsely powdered marble or sand, and brought to dryness. The entire dried mass is transferred to an extraction apparatus, and extracted for six or eight hours with not over 50cc. strong alcohol. To the slightly colored extract is added an equal volume of water-free ether, and the solution after standing a short time is poured into a weighed flask, or filtered through a small filter, which is afterwards washed with a little ether-alcohol. After the evaporation of the ether and alcohol the residue is dried in the air bath at  $100^{\circ}$  to  $105^{\circ}$  C. in a loosely-closed flask, until the losses in weight are constant. With beers that are very rich in extract, the ash-content of the glycerine may be determined and deducted from the total weight.

The methods recently published for the estimation of glycerine by its conversion into carbonic acid by sulphuric acid and bichromate of potash have been utilized for its determination in fermented liquors by Legler<sup>1</sup> and promise to prove more expeditious and exact than the old methods.

#### PHOSPHORIC ACID.

The phosphoric acid was determined by means of a standard solution of uranium acetate, except in the case of a few very dark-colored samples, when the analysis was made gravimetrically from the ash by precipitation with ammonium molybdate, in the usual way.

#### CARBONIC ACID.

Most investigators have given very little attention to the determination of the carbonic acid in beer, regarding it as of little importance in forming an estimate of the quality of the sample examined. The practical consumer, however, is of quite a different opinion and condemns immediately a beer which is "flat" or insufficiently carbonated, however worthy it may be in other respects. The reason for its unimportance as a determination is found in the difficulty of the accurate estimation in the beer as supplied to the customer. The usual method of determining it is to measure or weigh out a convenient quantity of the beer into a flask, connect the latter either with an absorbing apparatus for

<sup>1</sup> Rep. Anal. Chem. 47 Analyst, 1887, 14. See further under wine.

the estimation of the  $\text{CO}_2$  direct; or with a suitable apparatus for the retention of water, thus estimating it by loss or indirectly, its liberation from the beer being accomplished by the aid of heat. But this manner of manipulation gives simply the amount of carbonic acid capable of being held in solution by a liquid of the density and temperature of the beer when it was measured out, supposing it to have been fully charged previously. The excess of gas above the saturation point, however, which is held by the beer so long as it is kept under pressure, or at a low temperature, escapes as soon as the pressure is removed and gives the beer its "head," which is so desirable a qualification. This excess of gas soon passes off, but during this short interval the beer is drank. The problem is to estimate the carbonic acid just as it exists in the beer as it is consumed. This is a difficult matter with beer contained in casks or kegs, though it might be done by drawing the sample off by gas-tight connections as in gas analysis. But where the beer is furnished in stoppered bottles it is an easy matter and furnishes a most valuable index as to the freshness and proper preparation of the beer. Where secondary fermentation or souring has set in, there will be an excess of carbonic acid and the beer will have become cloudy.

Where there is a good content of carbonic acid, but the acidity of the beer is very low, the indications are that bicarbonate of soda has been added.

Hassall speaks of the estimation of  $\text{CO}_2$  in bottled aerated waters, the gas being drawn off by means of a champagne tap, and Dr. Wiley<sup>1</sup> has estimated the  $\text{CO}_2$  in koumiss in that way, using a calcium chloride tube for retention of the water carried off by the gas, and estimating it by difference, the whole bottle being weighed. In applying this form of apparatus to beer considerable difficulty was experienced on account of the viscosity of the liquid; the bubbles formed were so tenacious that when the bottle was connected directly with a calcium chloride or sulphuric acid tube, the latter would become filled with the beer in a very short time. This difficulty was obviated by the use of the form of apparatus shown in the accompanying figure, devised by Mr. T. C. Trescot and myself.

The cork of the bottle is pierced with a champagne tap, and this is connected with an Erlenmeyer flask, in the broad bottom of which the bubbles are broken and not allowed to pass beyond it; next comes a U tube filled with sulphuric acid, then a calcium chloride tube, then a soda lime tube to absorb the dried  $\text{CO}_2$ . The bottle of beer is placed in a convenient vessel—an empty ether can with the tap cut away answers admirably, as shown in the cut—which is nearly filled with cold water. After the apparatus is connected the tap is opened slowly and the gas allowed to flow through the apparatus; when it ceases to flow spontaneously a burner is placed under the can and the tempera-

<sup>1</sup> Am. Chem. Jour. 1885. Ann. Rept. U. S. Dept. Agr'l., 1885, p. 118.

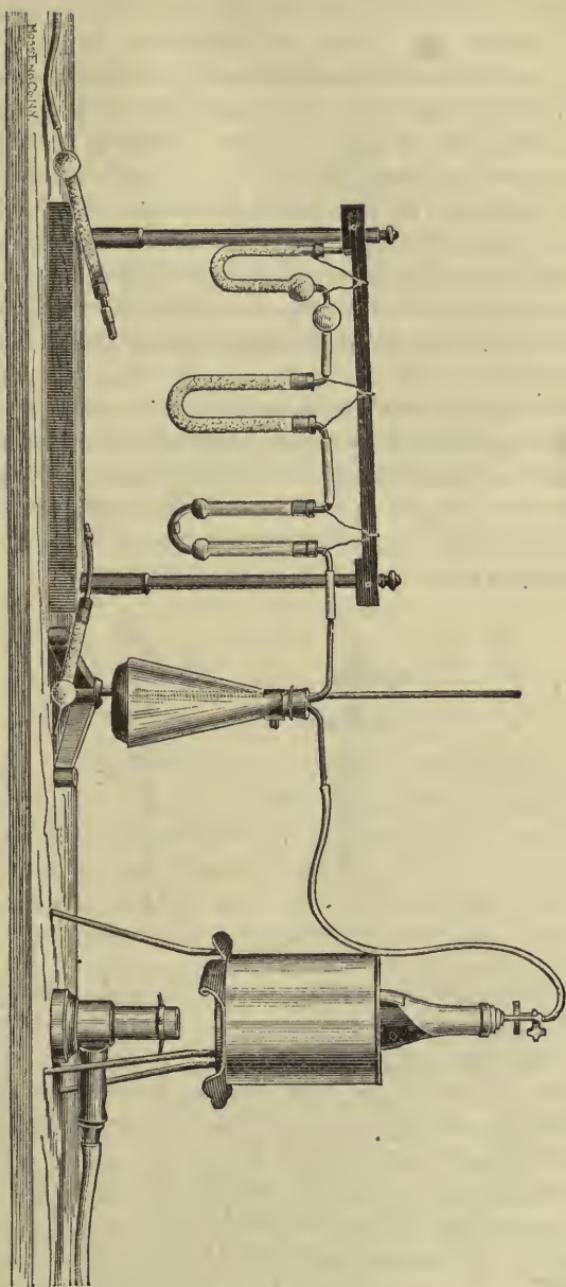


FIG. 14.

ture gradually raised until it reaches  $80^{\circ}$  C., beyond which it should not be carried. By holding it at this temperature for about half an hour and taking the bottle out of the can and shaking it occasionally all the  $\text{CO}_2$  may be driven off. Then the tube from a suction pump is connected with a calcium chloride tube and this in turn with the soda lime tube. The valve of the tap is then closed, the latter removed from the bottle, connected with a soda lime tube, and by gradually opening the valve, a stream of air, from which the  $\text{CO}_2$  has been removed, is drawn through the apparatus by the pump, so that all the  $\text{CO}_2$  contained in it is drawn into the soda lime tube and absorbed; the increase in the weight of the latter, of course, gives the weight of  $\text{CO}_2$  in the beer. The quantity of the beer is measured or weighed, that which has been carried into the Erlenmeyer flask being added, and the per cent. ascertained.

In the case of large bottles it may be found necessary to add a second soda lime tube, and it is best to use fresh soda lime for each determination. It was found necessary to modify the champagne taps, as the thread with which they are provided cuts the cork too much and allows the escape of the gas. Accordingly this thread was turned off entirely, leaving a smooth tube, as shown in the figure.

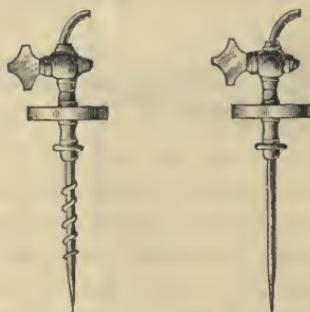


FIG. 15.

This can be forced through the cork with little difficulty, and allows no leakage of gas. It was also found necessary to have the cocks reground to make them fit accurately.

The patent rubber-stopper beer bottles presented a difficulty to this method of analysis, as it was impossible to make a gas-tight connection with them without the escape of the confined gas. In these the gas was estimated by loss of weight, calcium chloride and sulphuric acid tubes and connections being weighed with the bottle on a large balance, the bottle opened and connection made as quickly as possible, the gas driven off as before, and the loss ascertained by weighing the entire apparatus again. It is necessary to raise the heat very gradually to prevent the filling up of the tubes with the beer carried over by the bubbles. There is a slight error from the watery vapor which escapes with the pressure of gas when the bottle is opened, and altogether the determination is not so satisfactory as with the cork-stoppered bottles.

The percentage of CO<sub>2</sub> in the keg beers was not determined. For convenience of inspection the results of the determinations made are given again below:

Number.	Percent. CO <sub>2</sub> .	Number.	Percent. CO <sub>2</sub> .
4800	.411	4817	.629
4801	.300	4818	.344
4802	.489	4820	.503
4803	.415	4821	.397
4804	.328	4827	.441
4805	.471	4828	.592
4806	.717	4843	.242
4807	.219	4845	.265
4808	.324		

Nos. 4801-4803 and 4806 had rubber stoppers, the rest cork. No. 4068 was evidently in a state of after-fermentation, cloudy, and acid; excluding that analysis, the 16 others gave an average of .398 per cent. Most authorities give an average of .1 to .2 per cent. in beer. In Nos. 4801 and 4804 duplicate analysis were with different bottles of the same lot, with the following results:

	1.	2.
No. 4801 .....	.294	.307
No. 4804 .....	.329	.327

#### DETECTION OF ADULTERATION.

Probably there is no one article of daily consumption that has been so often subject to suspicion of adulteration or sophistication as beer. Its complex composition and peculiar nature have deceived people into making all sorts of charges against its purity, but experience has failed to establish the truth of by far the greater majority of these charges, and the facts of many published analyses show that it is as free from adulteration as most other articles of consumption, and more so than some. Here comes in the question, so difficult to answer in this country, of what constitutes adulteration or sophistication of an article of food? The definition of what shall constitute a pure malt liquor is hard to settle. Even in Europe, where a much stricter supervision is kept over foodstuffs than here, the definition varies widely. In Bavaria, where more beer per capita is consumed than in any other country, the laws limit the materials from which it is made to barley, malt, hops, yeast, and water, while in England the comprehensive definition has been given to beer as being "a fermented saccharine infusion to which a wholesome bitter has been added."<sup>1</sup>

#### SUBSTITUTES FOR MALT.

A great deal has been said, pro and con, on the subject of the propriety of the use of other matter than malted barley as a source of

<sup>1</sup> Blyth.

saccharine material for brewing purposes. There may be said to be three ways of substituting saccharine material. First, other grain may be used for malting; second, unmalted starchy matter, that is whole grain, may be added to the malt before it is mashed, the latter being diluted as it were, for the diastase in the malt has converting power sufficient for considerably more starch than is contained in itself; third, the saccharine matter may be supplied already converted, as in commercial starch-sugar, or glucose, cane sugar, inverted cane sugar, &c. Of these different substitutes the third class is probably the more objectionable, as beer brewed from such saccharine matter is lacking in various constituents derived from the grain, which are important additions to its nutritive power, namely, the phosphatic salts and the nitrogenous bodies.

In much the same way would bread made from starch alone be lacking in nutritive value.

There is no way of determining directly or absolutely that a beer has been brewed partially from glucose, but it may be inferred from its small content of those constituents which are contained in malt, but not in glucose, such as phosphoric acid and albuminoids, and the existence in the ash of large proportions of such salts as are known to form a large part of the ash of commercial starch-sugar, as sulphates. König gives .05 per cent. of phosphoric acid as the lowest limit for a beer containing 5 per cent. of extract or over.

The association of Bavarian chemists depends on the estimation of the nitrogen for the detection of the use of malt substitutes, and establishes the minimum of .65 per cent. of nitrogen (4 per cent. of albuminoids) in the extract. It is very evident that these figures are too high for American beers; only two of the samples examined, Nos. 4821 and 4823, contain less than .05 per cent. of phosphoric acid, and these are both imported beers; while the average content of the samples of American beer is .077. Not a single one of the samples contains as low as .65 per cent. of nitrogen in the extract, most of them containing about 1 per cent., while some give over 2 per cent. Dr. Englehardt's samples show a still higher average per cent. of phosphoric acid. Unfortunately there was no determination of the albuminoids in his samples. Yet it is a well-known fact that very few beers are made in this country without more or less malt substitution. Nothing can settle this point and enable the analyst to decide positively whether malt substitutes have been used until a standard is established by the analysis of a large number of samples known to be brewed from pure malt alone.

#### SUBSTITUTES FOR HOPS.

The nature of the bitters used in beer has long been the target towards which public suspicion is directed, and nearly every substance known possessing a bitter taste has been enumerated among the adulterations of beer, from poisonous alkaloids, such as strychnin and pic-

rotoxin, to harmless or quasi-harmless bitter roots and woods, such as quassia, gentian, &c. Complete and exhaustive schemes of analysis have been compiled, such as Dragendorff's, Ender's, &c., for the detection and isolation of such foreign bitters. Either these methods of investigation are faulty or difficult of manipulation, or the use of foreign bitters is very much less prevalent than is generally supposed, for the cases where such bitters have been detected and isolated are very scarce in chemical literature. In fact, Elsner, a German authority on food adulterations, goes so far as to say that there has never been a case where the existence of a foreign bitter in a malt liquor has been proven with certainty. This is going too far, of course, for picrotoxin and picroic acid have undoubtedly been found in beers, and probably more cases of such adulteration would occasionally have been discovered were it not for the difficulty of the analysis and the small quantity of matter required for imparting a bitter taste. But there is probably much less of this hop substitution than the space given it in works on the subject would indicate. Hops not only give the bitterness to beer but also impart to it its peculiar aroma, and enhance its keeping qualities, and unless it were at a time when they were very dear it would hardly pay the brewer to sacrifice the good flavor and keeping qualities of his beer in order to save a few cents a pound in his bitters.

It is stated by authorities on the subject that the bitter matter of hops is precipitated by acetate of lead, while with *all* hop substitutes the filtrate from the lead precipitate retains its bitter taste. The excess of lead should be precipitated by sulphureted hydrogen before the filtrate is tasted for bitterness. I examined qualitatively by this test all the samples analyzed and found them all free from foreign bitters according to it, with one exception, No. 4811, which contained a bitter other than hops, though not in sufficient quantity to admit of its separation and identification. All the samples except Nos. 4801, 4811, and 4815 gave a plainly perceptible odor of hops in the distillate.<sup>1</sup>

#### PRESERVING AGENTS.

We come now to what I consider the most important sophistication of beer at the present day and the most reprehensible and most deserving of repressive legislation. The use of artificial preserving agents not only introduces foreign matters into the beer which are more or less injurious, according to the nature of the material used, but also serves to cover up and hide the results of unskillful brewing or unfit materials; giving to the public for consumption a liquor, that, if left to itself under natural conditions, would have become offensive to the senses and putrid with corruption long before it was offered for sale.

The only means of preservation allowed by the authorities in Germany and France is the process called, from the name of its author, "Pasteurization." This process is entirely rational and commendable,

<sup>1</sup> See Appendix A.

as it conduces to the preservation of the beer by destroying the germs of unhealthy ferment, not by simply paralyzing their activity as antiseptics do, and moreover it introduces no foreign constituents into the beer. Liquid carbonic acid is also coming into use in some of the larger Continental breweries.

Other preservative agents extensively employed at the present day are salicylic acid, bisulphite of lime, and boracic acid.

#### SALICYLIC ACID.

Salicylic acid ( $C_7H_6O_3$ ) was first prepared by Piria and Ettling by oxidizing salicyl aldehyd, which had previously been obtained from various vegetable sources. It was afterwards obtained from oil of wintergreen, which is nearly pure methyl salicylate, a constituent also of many other essential oils. Its artificial production from phenol (carbolic acid) was discovered by Kolbe and Lautermann in 1860 but was not put into practical use until 1874, when Professor Kolbe succeeded in producing it at a moderate cost. It is now prepared almost exclusively in this way, the cheapness of the method having driven out of the market that which is prepared from oil of wintergreen.

By this process sodium carbolate is treated with dry carbonic acid gas and distilled at a rather high temperature, when one-half of the phenol combines with the sodium, forming sodium salicylate, while the other half is distilled over. The residue is decomposed with hydrochloric acid, the salicylic acid filtered off and washed, and purified by recrystallization or sublimation. The purest salicylic acid is obtained by dialysis, by which all the tarry matters can be separated. It is composed of long acicular crystals, having a peculiar, pungent, sweetish taste. It is irritating to the mucous membrane of the nasal passages and produces sneezing when handled.

The extended use of salicylic acid it owes to its property of arresting the action of ferment. This property has been extensively investigated and it is unnecessary to go into the subject further here.<sup>1</sup>

In medicine, besides its use externally as an antiseptic, it is administered very extensively internally, its chief application being as a remedy for acute rheumatic fever. Its physiological action is given as follows in the United States Dispensatory, fifteenth edition, page 101:

When salicylic acid is given to man in doses just sufficient to manifest its presence, symptoms closely resembling those of cinchonism result. These are fullness of the head, with roaring and buzzing in the ears. After larger doses, to these symptoms are added distress in the head or positive headache, disturbances of hearing and vision (deafness, amblyopia, partial blindness), and excessive sweating. According to Reiss (*Berliner Klin. Wochenschrift*, 1875, p. 674) decided fall of temperature, without alteration of the pulse, also occurs; but this is denied by other observers. The actions upon the system of the acid and of its sodium salts (also ammonium salt, Martenson, *Petersab. Med. Zeitschrift*, 1875, p. 343) appear to be identical, and, as sev-

<sup>1</sup> For recent information on this subject reference may be made to a paper by A. B. Griffiths, Chemical News 53, 28.

eral cases of poisoning with one or other of these agents have occurred, we are able to trace the toxic manifestations. Along with an intensification of the symptoms already mentioned there are ptosis, deafness, strabismus, mydriasis, disturbance of respiration, excessive restlessness passing into delirium, slow laboring pulse, olive-green urine, and involuntary evacuations. In some cases the temperature has remained about normal, but in others has approached that of collapse. The respiration seems to be characteristic, it being both quickened and deepened, often sighing. Sweating is usually very free, and the urine early becomes albuminous. Various local evidences of vaso-motor weakness may supervene, such as rapidly-appearing bed-sores at points subjected to pressure, and transitory dark colored maculae on various parts of the body. In several cases death was probably produced by the acid, although there is scarcely one instance which is beyond doubt.<sup>1</sup> In certain cases the mental disturbance has been strangely prolonged, lasting for eight days. In some instances it is cheerful, in others melancholic in type. It is stated that upon drunkards the acid acts very unfavorably, violent delirium being an early symptom of its influence.

By the same authority the dose of salicylic acid to be employed in cases of acute rheumatism is given as one dram (3.9 grams) in twenty-four hours. It is excreted chiefly by the kidneys and may be detected in the urine very soon after its ingestion. Authorities in therapeutics warn practitioners of medicine against its administration to patients whose kidneys are known to be diseased, and of late years the opinion has been growing among physicians that it has a very irritating action upon these organs, many preferring the alkaline treatment of rheumatic fever on this account.

#### USE AS A PRESERVATIVE.

The "salicylic-acid question," as it is called, has received a great deal of attention for several years in Europe, and much has been written, pro and con, on the question of the propriety of its use as a preserving agent in articles of food and drink. In France its use as a preservative in any form of food or drink was forbidden by ministerial decree on the 7th of February, 1881. This decree was based upon a decision of the consulting committee of hygiene that its constant use was dangerous to health.

In Germany its use is prohibited, except in beers intended for export to other countries where its use is allowed.

Its prohibition in France called forth a great deal of opposition, and experiments were made and published, which were intended to show that its constant use in small doses exerted no injurious influence upon the system. Kolbe himself made experiments upon himself and his assistants by taking doses of .5 to 1.0 gram daily for several days, and found no appreciable ill effects to follow its use.<sup>2</sup> Whether such ex-

<sup>1</sup> In the case recorded in the *Virginia Medical Monthly*, June, 1877, forty-eight grains of the acid were taken in four hours. The symptoms were violent vomiting, headache, total unconsciousness, with stertorous breathing. Death occurred forty hours after the first dose.

<sup>2</sup> *Jour. prak. Chem.* 13, 106. Reference may be made to similar experiments, as follows: *J. A. Barral, Jour. de l'Agriculture*, 1832, 69. *M. Blas, Bull. de l'Acad. Royale de Méd. de Belgique*. Bd. 12, No. 9.

periments suffice to prove its harmlessness when used for many years and without regard to age, sex, or personal idiosyncrasy is still an open question. A most interesting and exhaustive discussion of the reasons for and against its use can be found in the report of the fourth meeting of the "Independent Union of the Bavarian Representatives of Applied Chemistry, at Nürnberg, 7th and 8th August, 1885,"<sup>1</sup> when this body refused, with but one dissenting voice, to grant its sanction to the proposed use of salicylic acid in beer in the quantity of .05 grams to the liter. Certainly no one would deny the advisability of at least restricting the amount to be used of so powerful an agent. In an article of daily consumption, and in consideration of the prevalence of kidney disease<sup>2</sup> at the present day, it is a matter worthy of grave consideration, whether it would not be more prudent to forbid its use altogether. At all events, beer in which it is used should be sold under its proper designation as "salicylated beer." It would certainly be of interest to the physician, who prescribes beer as a tonic to a weak convalescent invalid, to know if he were giving at the same time not inconsiderable doses of a strong therapeutic agent, expressly contra-indicated perhaps, in the case he has on hand.

The following amounts of salicylic acid were found in various articles of diet by Ch. Girard, director of the Municipal Laboratory, in 1881<sup>3</sup>:

Wine contained in the liter, 1.95, 1.60, 1.48, 1.41, 1.35, 0.81, and in one case even 3.50 grams salicylic acid.

Sirup contained in the liter, 0.5 to 1.50 grams.

Beer contained in the liter, 0.25 to 1.25 grams.

Milk contained in the liter, 0.25 to 1.85 grams.

It will be noticed that in one case of wine mentioned it contained in one liter the full therapeutic dose for twenty-four hours.

In this country but little attention seems to have been given to the use of salicylic acid as a preservative. In the investigation made by the New York State Board mentioned above, no search was made for it, or, in fact, for any other preservative. In the last year the municipal boards of New York and Brooklyn seem to have been taking cognizance of its extensive use, as is shown by the following extract from a paper read by Dr. Cyrus Edson, of the New York Board of Health, before the New York Society of Medical Jurisprudence and State Medicine, November 12, 1886:<sup>4</sup>

Within the past few months I have been confronted with a subject the importance of which to the community is very great. I have already touched upon it. It is the use of salicylic acid, a food preservative. Many, if not all, the manufacturers of preserved foods are adding small amounts of this substance to their goods to prevent loss by decomposition. The amount used is probably between one-third of a grain and a grain to the pound, and in the case of wines and beer, to which it is also added

<sup>1</sup> Published by Drs. A. Hilger and R. Kayser, Berlin, 1886.

<sup>2</sup> The most common form is popularly known as "Bright's disease."

<sup>3</sup> Pharm. Cent. 22, 296.

<sup>4</sup> American Analyst 1887, p. 7.

to prevent over-fermentation, from a grain to 3 grains to the pint in quantity. The French authorities, as I have said, believe that the use of salicylic and boric acids tends to irritate delicate digestive organs, and to also irritate the kidneys, through which they are eliminated under their own forms. Though I have talked with a number of scientific gentlemen in this country, few are willing to go into court and swear that this is also their opinion. It must not be lost sight of, however, that a person might at a meal take several articles of diet, each containing that which if taken alone would be a harmless dose, but taken together, and possibly for a considerable time, would prove highly injurious. The only safe way is to discard all additions to food which may possibly become a source of damage.

The following is of interest as establishing a precedent for the condemnation of articles containing salicylic acid:<sup>1</sup>

Dr. Cyrus Edson, of the Board of Health, condemned and seized on November 11, 5,280 gallons of artificial wine in the possession of a Front street merchant. Dr. Edson reported the following as the process of manufacturing the stuff: "Dried fruits, such as raisins, currants, and peaches, are macerated with water, to which a certain amount of sugar is added. The mixture is then fermented, and when fermentation is considered sufficiently advanced it is checked by the addition of salicylic acid, sufficient being added to act as a preservative and prevent further fermentation. The so-called wine is then clarified, flavored, and colored to cause it to resemble port, claret, or any desired kind of wine. The object of the sophistication is to imitate and undersell natural native wines. The use of salicylic acid as a preservative is forbidden in France, as the French authorities consider it detrimental to health." I have consulted a number of noted chemists in this city as to their opinion concerning its use and nearly all unhesitatingly condemn it, holding that depressing effects in the nervous system would follow the daily use of the acid in small doses. It is my opinion and the opinion of Drs. J. B. Isham and J. B. Linehan, whom I have called on to assist in condemning and seizing the so-called wine, that the adulteration is a dangerous one and likely to cause sickness. The amount of acid used is about four and a half grains to the pint."

Recent information in regard to the status of the question in France, together with a very strong argument in favor of prohibiting entirely the use of this preserving agent, may be found in the following recommendation by Dr. Bartley:<sup>2</sup>

Dr. Bartley, the chief chemist of the Brooklyn Board of Health, has sent a communication to Health Commissioner Otterson, of that city, on the subject of poisonous beer adulterations, and strongly recommending that the Board take action against it. Dr. Bartley says: "During the year 1855 I had the honor to call the attention of the Department to certain abuses in the manufacture of lager beer, so called, in the course of which I said that 'the most important adulterations discovered, from a sanitary point of view, were yeast and sodium bicarbonate.' Since that time I have from time to time inspected the breweries of the city. As this beverage has become so largely used by families, it is now kept in bottles by many grocers. All brewers are compelled to manufacture an article for the use of bottlers. This is prepared with more care, in most cases, on account of the longer time it is to be kept before using. Unless it be thoroughly cured and well cleared the beer will often spoil before it is consumed, by a process of fermentation or putrefaction. To avoid the necessary care in the manufacture and the keeping of the beer for a longer time, it has become a practice among brewers to add salicylic acid to prevent this fermenta-

<sup>1</sup> American Analyst 1856, p. 416.

<sup>2</sup> American Analyst 1857, April 1.

tation after the beer is sent out. I have for some time been aware of this use of salicylic acid, as well as its addition to wines, canned fruits, cider, milk, and other goods, for the purpose of preserving them from fermentation. In fact, the use of this acid is coming into such general use in foods that it is becoming an important sanitary question as to its effects upon health when used in small quantities for a long time. There can be no doubt that in large quantities it acts very injuriously both upon the digestive processes and the kidneys. In its elimination the kidneys not rarely become acutely congested or even inflamed, giving rise to acute Bright's disease. Although a potent remedy in the treatment of acute rheumatism, it is not suitable for long administration, owing to the above injurious action. It requires the addition of from eight to ten grains of free salicylic acid to one gallon of beer in order to prevent the growth of ferments. If bicarbonate of soda has also been added, the quantity of acid necessary to be added is much more. Three grains have recently been found in each pint of wine. Assuming that the smallest effective proportion of the acid is used, viz., ten grains to the gallon, there are many persons in this city who take no inconsiderable amount of this drug every day of their adult lives. The salicylic acid of the market is prepared from carbolic acid, and is frequently contaminated with a small proportion of this very poisonous agent. This is a well known fact to all pharmacists. The sanitary question, then, rests upon the question of the action of small and long continued doses of salicylic acid, possibly contaminated with carbolic acid."

"Upon this point I think it necessary to quote here but one of many authorities. In 1881, and again in 1883, upon the recommendation of the Central Committee of Hygiene, the French Government prohibited the sale of articles of food containing salicylic acid. As protests were made against this legislation, and as these protests had led to contradictory judicial decisions, the above committee requested the opinion of the French Academy of Medicine. A commission created by that body of savants has recently made a report (*Bulletin de l'Acad. de Med.*, Paris, 1886, T. XVI., pp. 583 et seq.), from which the following is an extract: 'While in persons of good health the prolonged use of such small quantities of salicylic acid as would be contained in articles of food or drink treated with this substance is probably not injurious to health, it may nevertheless produce very decided disorders of health in certain persons, and especially in the aged and in those who have a tendency to diseased kidneys or dyspepsia. Salicylic acid and its salts are eliminated by the kidneys. They tend somewhat to check the action of the digestive ferments contained in the saliva, enteric juice, and pancreatic fluid, and hence to delay digestion; hence it is easy to understand that they may aggravate digestive or renal troubles.'

"The report closes with the recommendation that the addition of salicylic acid or its compounds, even in small amounts, to articles of food or drink shall be absolutely prohibited by law. This commission found by their investigation that the quantities added to wine were about six grains to the gallon, and to beer from twelve to fifteen grains."

"From the facts here stated I am of the opinion that it is time that the addition of salicylic acid to articles of food received a check at the hands of sanitary authorities. I have made examinations of several different kinds of bottled beers manufactured and sold in this city, and have found a number of them to contain salicylic acid. The list examined contained some of the Western beers, which were also found to contain it. I would respectfully recommend that some action be taken by this Department towards the prohibition of this injurious adulteration.

"Respectfully,

"E. H. BARTLEY, M. D., *Chief Chemist.*"

## SALICYLIC ACID IN SAMPLES EXAMINED BY THIS DIVISION.

Out of thirty two samples analyzed by this Division I found seven to contain salicylic acid in sufficient quantities to admit of qualitative proof, or nearly one fourth of the entire number analyzed. The serial numbers of these beers corresponding to those in the large table on page 282 are as follows: 4801-3-5-6-17-23-25. These were all bottled beers, one being an imported (Kaiser) beer. None was found in any of the draft beers. Of the nineteen samples of American bottled beers, six contained salicylic acid, or nearly one third. These included the product of some of the largest breweries in the country, beers that are used to a very large extent all over the United States. Whether the acid is added in the breweries where the beer is made, or whether it is used by the local bottlers, I am unable to decide. In one case I found it in the beer sold here under the brand of a large Western brewery, and sent direct to the same brewery for another sample, which gave no test for the acid; unfortunately I cannot be sure in this case that the firm in question did not know the purpose for which the sample was intended.

## DETECTION AND ESTIMATION OF SALICYLIC ACID.

Fortunately we have a particularly delicate and characteristic test for this substance, by means of which its presence can be detected in the minute quantity of 1 part to 100,000. This is the well-known characteristic violet color it gives with ferric salts. The test can sometimes be applied directly in the case of very clear beers, but in most samples it is obscured, either by the original color of the solution, or by the color produced by the combination of the iron with other constituents of the liquid. There are various procedures given for the separation of the salicylic acid from these constituents, so as to apply the test to it when in a pure state. Blas<sup>1</sup> has investigated various methods, including the previous precipitation of the beer with lead acetate, and the removal of the excess of lead with sulphuric acid;<sup>2</sup> shaking out the acidified solution with ether, evaporation of the ether and testing of the residue; treatment with bone-black, washing out the latter with alcohol and applying the test to the alcoholic solution;—none of which methods, he thinks, gives so good results as the application of the test to the urine of a person who has drank some of the beer in question. By this peculiar method of making the human body a medium of separation, he claims to make the test five times as delicate as where it is applied directly to the beer. Boruträger<sup>3</sup> arrived at similar conclusions, and I can testify myself to its superior delicacy to the direct application to the beer. Muter<sup>4</sup> was probably the first to use dialysis for the separation, also recommended by Aubry.<sup>5</sup> Portele<sup>6</sup> precipitates the tannin, &c., by a solution of gelatine and shakes up the filtrate with ether. Weigert<sup>7</sup> uses

<sup>1</sup> Jour. prak. Chem., 19, 43.

<sup>5</sup> Zeit. Anal. Chem. 1880, 105.

<sup>2</sup> E. Robinet, Compt., rend. 84, 1321.

<sup>6</sup> Weinlanbe 1879, 389.

<sup>3</sup> Zeit. Anal. Chem. 1881, 87.

<sup>7</sup> Zeit. Anal. Chem. 1880, 45.

<sup>4</sup> The Analyst 1, 193.

amyl alcohol as a solvent, as follows: "50cc. wine<sup>1</sup> (or beer) are shaken up with 5cc. amyl alcohol in a small flask for a few minutes and allowed to separate. The clear amyl alcohol is then drawn off and an equal quantity of alcohol added, with which it makes a colorless solution. To this solution is added a few drops of dilute solution of chloride of iron, which produces the violet color."

The use of chloroform as a solvent is recommended by the German Imperial Commission for the establishment of methods for wine analysis.<sup>2</sup>

The Paris Municipal Laboratory allows the choice of four different methods for applying the test in case the ether extract does not afford satisfactory results:

- (1) By adding a small quantity of a dilute solution of chloride of iron to precipitate the tannin, and subsequent extraction with ether.
- (2) By precipitation of the tannin with gelatine or albumen.
- (3) By treatment with ether in the ordinary way and after the ether is evaporated the residue is again treated with a few cubic centimeters of perfectly pure benzine, the solution evaporated, and the residue from this evaporation subjected to the test.
- (4) By treatment with pure chloroform.

The union of Bavarian chemists has<sup>3</sup> adopted the method of Röse<sup>4</sup> who uses a mixture of equal parts of ethylic and petroleum ether for extracting the beer or wine, as follows: "50cc. of the beer are shaken up in a separatory funnel with equal parts of ether and petroleum ether, after acidulating with 5cc. of dilute sulphuric acid. The separation follows very quickly, when the watery part is allowed to flow through the stop cock and the ethereal is poured out through the neck into a small dish. After the ether has been evaporated, and also the greater part of the petroleum ether except a few cubic centimeters, 3-4 cc. of water are brought into the still warm dish. This is well stirred, a few drops of a very dilute solution of chloride of iron added, and the whole filtered through a moistened filter, which allows of the passage only of the watery part of the solution. On the addition of the chloride of iron the petroleum ether solution assumes a deep yellow color, due to certain compounds of the iron with the resin of the hops. In the absence of salicylic acid the filtrate is nearly water-clear with a slight tinge of yellow; if present, even in traces, the solution takes on the well known violet color.<sup>5</sup>

To test the efficiency of these various methods two samples of a very dark beer known to be free from salicylic acid were taken, and to one was added salicylic acid in the proportion of .05 grams to the liter, and

<sup>1</sup> Some of the processes given are intended for wine, but are equally applicable to beer, milk, fruit juices, &c.

<sup>2</sup> Die Weinanalyse, Kommentar, u. s. w.

<sup>3</sup> Op. cit.

<sup>4</sup> Arch. f. Hygiene. Analyst, 1886, 133.

<sup>5</sup> The same test is given by H. Tasse, Bull. de la Soc. de Chim. de Paris, 46, No. 12.

to the other .005 grams. These two samples were then subjected to treatment by eight different methods, as follows :

- (1) Extracted with chloroform.
- (2) Extracted with ether simply.
- (3) Extracted with amyl alcohol.
- (4) Extracted with ether after previous precipitation with lead subacetate.
- (5) Extracted with ether after previous precipitation with gelatine.
- (6) Extracted with ether after previous precipitation with ferric chloride.
- (7) Extracted with equal parts of ethylic and petroleum ether.
- (8) Extracted with ether, the solution allowed to evaporate spontaneously, and the residue extracted with benzine.

These tests, tried on the beer containing .05 grams to the liter, gave results which ranged them in the following order of succession, according to the strength and brilliancy of the test: 8, 5, 7, 6, 2, 1, 4, 3. In methods 5 and 6 the length of time required to filter the solutions constitutes an objection to their use. The same tests applied to the beer containing .005 grams to the liter gave the test only in the case of Nos. 8 and 7, the former being the better of the two. The same series of tests were applied to a red wine, with very similar results, so I have adopted method No. 8 in all the work done on beers and wines, and would recommend either that or No. 7, both of which give a perfectly bright, water-clear solution, in which the slightest tinge of violet color is plainly visible.

#### QUANTITATIVE ESTIMATION.

While the qualitative determination of salicylic acid is so delicate and easy of execution, the quantitative estimation is unfortunately a tedious and unsatisfactory operation, in the small quantities in which it is found. This is an argument in favor of the entire prohibition of its use in foods in preference to the restriction of the quantity to be used.

The determination may be made by the same method as described above for the qualitative test, simply making the extraction complete. One hundred cubic centimeters of the beer or wine are taken, acidified with a few drops of hydrochloric acid, and extracted with three successive portions of ether of 50cc. each; these are mixed and the whole allowed to evaporate spontaneously. The residue from this evaporation is heated for an hour on the water bath to drive off volatile acids, and treated with 150cc. of pure benzine, which is allowed to stand in contact with the residue for twenty-four hours, when it is drawn off carefully and the residue again treated with 50cc. of benzine, which is added to the first portion. This 200cc. of benzine is then made up to 500cc. with absolute alcohol, and titrated directly with a decinormal solution of soda, previously standardized by operating upon a similar mixture.

The acid may also be extracted by treatment with successive portions of chloroform, which is carefully decanted, the solution evaporated, and the crystallized salicylic acid weighed.<sup>1</sup>

A. Rémont<sup>2</sup> published in 1881 a method for the colorimetric estimation of salicylic acid, which was modified by Pellet and De Grobert<sup>3</sup> in the following manner:

A series of uniform test-tubes are prepared about 20cm. in height and 15mm. in caliber, to which are added, successively, 1cc., 0.75cc., 0.5cc., 0.4cc., 0.3cc., 0.2cc., 0.1cc. of a solution of 1 gram of salicylic acid in 1 liter of distilled water, and the volume in each tube brought to 10cc. with distilled water. To the first tube is added three drops of a dilute solution of ferric chloride (1005 to 1010 specific gravity), in the second and third two drops, in the others one drop. The last tube may be simply stirred with a glass rod which has been dipped into the iron solution. One hundred cubic centimeters of the beer or wine to be tested are now taken and shaken up with 100cc. of ether, and five drops  $H_2SO_4$  of 30° B., allowed to stand, carefully separated, and this operation repeated twice. The ether is quickly evaporated off in the water bath, the residue brought into a porcelain evaporating dish of about 6-8 cm. diameter, the flask washed out with a few cc. of ether and the dish placed in an air bath heated to about 50° C. to drive off the ether completely; 1.5cc. of a solution of soda is then added, of such strength that 10cc. contain 0.4 grams  $Na_2O$ , which is sufficient to saturate about 0.2 grams of salicylic acid, equal to a content in the sample of 2 grams per liter. If the residue is still acid after this addition, it is due to acetic acid. By evaporating to dryness this excess can be driven off while the salicylic acid is retained, as it is able to displace acetic acid from its salts. The residue is now treated with five drops of  $H_2SO_4$  of 30° B., and then with 20cc. of benzine, and the whole filtered; 10cc. of the filtered benzine solution is brought into a test-tube of similar dimensions to those mentioned above; 10cc. distilled water and one or two drops of the dilute ferric chloride solution added, and the contents well shaken. If salicylic acid is present it is all taken up by the lower watery portion and the color may be compared with that of the standard tubes. If it agrees in intensity with one of these, the calculation is very simple. Suppose, for example, it agrees with the fourth tube, which contains in the 10cc. of liquid 0.0004 grams salicylic acid, then the 10cc. of benzine solution also contained 0.0004 grams, and the 20cc. from which it was taken contained 0.0008 grams in 100cc. of the wine, or .008 grams to the liter. The authors of this method found on applying it to wine to which a known quantity of salicylic acid had been added, that only 93 per cent. of the amount added was found, so their results were divided by 93 on that basis. With this modification they obtained from two samples of

<sup>1</sup> These two methods are employed in the Municipal Laboratory of Paris.

<sup>2</sup> Jour. Pharm. Chim. [5], 4, 34, Chem. Cent., 1881, 773.

<sup>3</sup> Compt. Rend. 93, 278, Chem. Cent., 1881, 711.

wine to which had been added, respectively, 0.084 and 0.126 grams per liter 0.084 and 0.123 grams.

M. Rémont<sup>1</sup> also devised, in 1882, what might be called an empirical method for the estimation of the quantity of salicylic acid added to wine or beer, which would doubtless prove very convenient in case its use were limited by law to a certain definite quantity, as follows:

In a liquid, similar to that which is to be tested, is dissolved a known quantity of pure salicylic acid; of this standard liquid 50cc. are taken and well shaken with 50cc. of ether, and allowed to separate; 25cc. of the ether are taken in a flat dish and subjected to evaporation at a temperature below boiling, in the presence of 10cc. of water; when the ether has disappeared, the water is poured into a graduated cylinder and its volume made up to 25cc. with the washings of the dish. This solution contains the same proportion of salicylic acid as the standard; 10cc. of the liquid to be analyzed are then taken and shaken with 10cc. of ether; 5cc. of the clear ether are taken, evaporated with 2cc. of water, and the residual liquid made up to 5cc. with the washings of the dish, as above.

Two tubes, each graduated to 30cc., are taken, and into one is introduced 5cc. of the standard aqueous solution as obtained above, and into the other the 5cc. obtained from the sample for analysis. To each tube is added the same quantity of a 1 per cent. solution of ferric chloride, avoiding an excess. The comparison of the two tubes may then be made, and the process can be made quantitative by diluting with water, or other colorimetric methods.

The author insists upon the necessity of taking, as a means of comparison, a liquid of the same nature as the sample for analysis, as the foreign matters which ether dissolves from wine, beer, or cider alter the delicacy of the color test.

#### SULPHITES.

The use of sulphurous acid as a preservative agent in beer and wine, either in the form of soluble sulphites, liquid sulphite of lime, or sulphur fumes, is not at all recent. It is one of the oldest preservatives known. Together with other chemical preservatives its use is forbidden in France, and the German authorities include it with borax as an agent whose physiological effect is still too little known to allow of its indiscriminate use. It is also sometimes introduced into beers by the hops, which are very generally preserved by means of sulphur fumes. The Bavarian authorities allow its use in sulphuring barrels and hops, as will be seen when their method of analysis is described later. Of course the quantities brought into the beer in this way are very small.

The qualitative test, which is given by many of the books on the subject,<sup>2</sup> viz., the reduction of the sulphur to hydric sulphide gas by means of nascent hydrogen, is entirely erroneous, as I have proved by experi-

<sup>1</sup> Compt. Rend. 95, 786.

<sup>2</sup> König, for instance, p. 410; Dietzsch, p. 123.

ments made upon the various albuminous constituents of beer with the same test. Hops (known to be free from  $\text{SO}_2$ ), malt, and even ground barley, treated with hydrochloric acid and zinc gave a very distinct blackening of lead acetate paper in the course of fifteen minutes, and the test applied to the beers examined gave a distinct reaction *in every case*. I concluded from the above-described experiments that the  $\text{H}_2\text{S}$  came from the sulphur contained in the albuminous bodies of the grain, which was reduced by the nascent hydrogen. Blank experiments with the reagents used gave no test for sulphur. Since I made these experiments similar conclusions were reached by M. von Klobulow,<sup>1</sup> who found that sulphur was reduced from any of its compounds by nascent hydrogen, and so complete is this action that he has made it the basis for a new method of estimating sulphur. It was probably by this test that sulphurous acid was found in very old wines, as has been reported.

The method of detecting the presence of  $\text{SO}_2$  by its oxidation to  $\text{H}_2\text{SO}_4$  is probably the best, and can be employed very successfully for the quantitative estimation also. There are various methods in use for affecting the oxidation, as well as for the subsequent determination of the sulphuric acid formed. In the method employed by the Paris Municipal Laboratory the beer is acidulated with sulphuric acid, and a current of pure carbonic acid gas is drawn through the liquid and then into a solution of chloride of barium mixed with iodine water. If  $\text{SO}_2$  is present, a precipitate of sulphate of barium forms in the latter mixture. Other oxidizing agents may be used instead of the iodine. Wartha<sup>2</sup> used nitrate of silver solution for a qualitative test; bichromate of potash furnishes a very convenient agent, and the solution of it may be made standard and titrated afterwards to determine the extent of oxidation. I have used permanganate of potash with very good results for a qualitative test. But probably the best method for both qualitative and quantitative determination is that used by the union of Bavarian chemists, which I have employed in testing the samples examined. It is as follows: 100cc. of the liquid to be examined are acidulated with phosphoric acid, and distilled in a stream of carbonic acid gas, and the distillate received in a flask containing 5cc. of normal iodine solution. After the first third is distilled off, the distillate, which should still contain excess of free iodine, is acidified with hydrochloric acid, heated, and barium chloride solution added. If a precipitate of more than 10 milligrams is obtained in the barium solution, the wine or beer contains sulphurous acid in excess of the legalized limit. (The allowance of 10 milligrams of barium sulphate is made to admit of the hops being sulphured.) In using this method I have found it necessary not only to have the delivery tube from the condenser dip into the iodine solution, but also to attach a mercury valve to the flask in which it is received.

<sup>1</sup> Zeit. Anal. Chem. 25, 155; Chem. News, 1886, 325.

<sup>2</sup> Berichte d. Deutsch. Chem. Gesell. 13, 657.

A few of the samples examined by this test gave a slight turbidity with barium chloride, viz: Serial Nos. 4804-6-10-13 and 14, while only one, No. 4815, gave sufficient precipitate to justify the assertion that a sulphite had been added to it. I have not been able to find any recorded instance of sulphurous acid being found in American beers.

#### BORAX.

This agent, although used very extensively in preserving meats, vegetables, and canned goods, does not seem to have been applied to malt liquors to any great extent, although it has been found in wines. Its use is prohibited in France and Germany. The test for boracic acid is best applied to the ash. If this is rubbed up with water acidulated with a little hydrochloric acid and a piece of turmeric paper dipped into the solution and then dried, it will show a peculiar reddish tint if borax be present. For a very delicate test a large quantity of the liquid to be tested may be evaporated to a sirup, with a slight addition of sulphuric acid, the residue extracted with alcohol, and the latter ignited. The edges of the flame will be colored green if borax is present. None of the samples examined gave any test for borax.

In conclusion of the work on preservatives, it may be noted that it was done during the cold weather of January, February, and March. It is quite probable that during warm weather the use of preservative agents is still more general than shown by the analyses.

#### MINERAL ADDITIONS.

The presence of lead, copper, or zinc, sometimes observed in malt liquors, is due usually to the use of brass faucets or lead pipes by the retailer in drawing off the liquor or in filling bottles. The amount of these metals taken up by acid liquors in this way is quite small usually, but may be considerable if they are long left in contact with the metallic surface. Thus the first glass drawn from a faucet in the morning is apt to contain considerable copper and zinc in solution. In Paris the apparatus used for drawing beer is subject to supervision, and a frequent cleansing and proper kind of material is insisted on. The Brooklyn Department of Health issued an order in 1886 prohibiting the use of unprotected brass faucets in drawing beer, but its enforcement has not been insisted on.<sup>1</sup> Analyses made for the board by Otto Grothe of ales drawn through pumps showed small quantities of copper, zinc, and lead in every case.<sup>2</sup>

Alum is sometimes used as a clarifying agent in the brewing of beer. The method of detecting the presence of the metals in liquors need not be dwelt on here. It is best performed in the ash from a large quantity of the suspected sample.

<sup>1</sup>Annual Report Dept. Health, City of Brooklyn, 1886, p. 87; and 1887, p. 63.

<sup>2</sup>Ibid.

## BICARBONATE OF SODA.

This salt is added to beer for the purpose either of correcting an undue acidity of the beer, resulting from improper brewing, or of imparting to it an increased "head," or content of carbonic-acid gas, or for both purposes. The salt is decomposed by the free acid of the beer and the gas liberated, lactate and acetate of soda being left dissolved in the beer. This seems to be purely an American practice; at least I have failed to find any mention of it in European authorities. Some of them mention the use of marble dust or magnesia for the correction of acidity, but very little consideration is given to the subject. In this country, however, it seems to be very widespread. The following extracts are taken from a paper read by Otto Grothe, Ph. D., before the American Society of Public Analysts:<sup>1</sup>

The Health Department of the city of Brooklyn has for some time carried on investigations with reference to the brewing of lager beer as practiced in that city. The peculiar cathartic effect of some of the Brooklyn beers seemed to indicate the presence of some substitutes, principally for hops. The analysis of such suspicious beer failed, however, to reveal anything of importance, either on account of the absence of such substitutes or because the quantities of beer used were too small. Before going to the expense of purchasing a keg or two from each brewery for the chemical laboratory, Dr. Bartley thought it to be the best to have the breweries, in the first place, inspected. These inspections resulted principally in the discovery of a variety of substances used by beer brewers as clearing and improving agents, the latter being considered the most objectionable. They were sodium bicarbonate, tartaric acid, cream of tartar, isinglass, or gelatine, glucose, grape sugar, juniper berries, and salicylic acid.

Sodium bicarbonate is a substance more regularly used by brewers. \* \* \* The opinion of the brewers about the necessity of this addition is very much divided; while some believe it to be utterly necessary, others say they would rather do without it, as it causes them a heavy expense. They all say, however, they cannot avoid it, because the public wants a perfectly neutral beverage. There is beer in the market which has no addition of bicarbonate. \* \* \* The quantity of soda added varies very much, and we may say in proportion to the quantity of acid contained in the beer. This quantity of acid in the beer depends upon the knowledge and the attention of the brewer. Thus we find that breweries which have clean, well-ventilated, and flushed cellars, in which refrigerating machines are in use, and which are conducted in a scientific way by an expert foreman, can afford to sell their product with less than one-third of the quantity of sodic bicarbonate used by smaller concerns which are not so well conducted and which have not the facilities of their larger competitors.

The largest quantity of bicarbonate used is about  $2\frac{1}{2}$  ounces to the keg, or quarter of a barrel. The size of a barrel varies from  $31\frac{1}{2}$  to 33 gallons, according to the age, the older kegs becoming smaller by the contraction of the wood. A keg, therefore, contains 8 gallons of beer, or 64 pints, which is considered equal to about a hundred glasses as sold in the beer saloons over the counter. A glass of beer, therefore, contains in some cases three-fourths of a gram of bicarbonate of soda; and as a moderate beer drinker will, under certain circumstances, for instance in hot weather, drink about twenty glasses of beer a day, he takes about 15 grams, or 252 grains of bicarbonate of soda with it. A heavy beer drinker—say, a laborer who works outdoors and who buys the beer by the pint—may consume as many as forty glasses a day, and he takes an ounce of bicarbonate of soda with it. The smallest quantity of bicarbonate of

<sup>1</sup> Ann. Rep. Dept. of Health, City of Brooklyn, 1885, p. 92.

soda used in our breweries is 1 ounce to a half barrel, and the difference in the effect of that addition is a very remarkable one, the beer tasting slightly acid.

There cannot be any doubt that large quantities of bicarbonate of soda regularly introduced into the stomach are detrimental to the health. Inasmuch as the lager beer is used as a food by many people, it would be greatly appreciated by intelligent beer-brewers and beer-drinkers if the use of bicarbonate of soda could be regulated by the authorities, or, if possible, entirely abolished. By such regulations the unclean brewer would be compelled to either keep his brewery clean, or go out of the business altogether. Such regulations should also be extended to the quality of the metals of the apparatus used in the different brewing processes, so that to the American lager beer the same name can be given as to the German beer, which Justus von Liebig called "liquid bread."

There are several rather misleading statements in the above. Dr. Grothe says in the first place that "the public wants a perfectly neutral beverage," which is open to considerable doubt; and again, "the smallest quantity of bicarbonate of soda used is one ounce to a half of a barrel, and the difference in the effects of that addition is a very remarkable one, the beer tasting *slightly acid*." If this latter statement is taken in a strictly chemical sense, it is rather paradoxical, for a bicarbonate added to a liquid of course tends to make it alkaline. What is meant by its tasting slightly acid doubtless is that it acquired a pungency to the taste on account of the liberation of carbonic acid gas from the bicarbonate by the free acid existing in the beer. One of the beers I examined (No. 4816) was actually alkaline in reaction from excess of added bicarbonate, and the taste was far from being agreeable.

I would hardly take so decided a stand as Dr. Grothe in regard to the injury done to the health of the beer-drinker by bicarbonate of soda *per se*. It may be necessary to explain to a non-scientific reader that the bicarbonate does not remain in the beer as *bicarbonate*, unless there is an amount added in excess of the quantity of free acid present in the beer. This free acid (mostly acetic in soured beers, but due chiefly to acid phosphates in normal beers) combines with the bicarbonate, setting free carbonic acid, and forming acetate of soda and basic phosphate, which remain in solution. The reaction is very similar to that which takes place in using baking powders for cooking purposes, except that in the latter case tartrate of soda and potash (Rochelle salts) is left instead of acetate and phosphate of soda. Where bitartrate of potash is added to the beer along with the soda (as sometimes occurs according to the Brooklyn report) the reaction is precisely the same. In these days of the almost universal consumption of baking powders there is doubtless enough alkaline salts thrown into a man's stomach with his food without pumping them in with his drinks as well. At all events there can be but little question of the propriety of prohibiting the use of bicarbonate of soda in beer. It is entirely unnecessary and foreign to the production or preservation of pure beer. Moreover, its use serves to cover up and hide the effects of poor brewing and improper storing or refrigerating, and should be prohibited from this cause alone if there were no other.

## DETECTION.

The detection of the addition of very small quantities of bicarbonate of soda to beer is by no means an easy matter when the constant presence of soda salts in beer ash is considered, and the very variable content of alkali in the waters used for brewing purposes. The ash of beer is of very variable composition, being obtained in part from each of the principal constituents which enter into the preparation of the drink, viz, the malt, the hops, and the water used in the brewing. The content of soda ( $\text{Na}_2\text{O}$ ) in the ash varies in different published analyses, from less than 4 per cent. up to 35 per cent. of the ash,<sup>1</sup> and this without a sufficient proportion of chlorine to account for the large per cent. of soda as salt. The presence of any considerable quantities of carbonate in beer ash, however, is abnormal, and indicates the addition of bicarbonate of soda to the beer, the acetates, lactates, &c., formed from it being converted into carbonates by the process of incineration. So far as I have been able to ascertain, no carbonic acid has ever been found in the ash of normal beer, its alkalinity being due to the presence of alkaline phosphates. I have found no statement as to whether the ash of normal beer reacts acid or alkaline in any of the books on the subject except Elsner,<sup>2</sup> who says it reacts acid. This must certainly be a mistake, for the ash of every sample I examined gave a strong alkaline reaction, requiring from 5 to 3.5cc. of decinormal acid for the neutralization of the ash from 100cc. of beer. Hassall<sup>3</sup> says, on the other hand, "the alkalinity of the ash must be estimated" in determining whether some alkaline earth or alkali has been added.

In order to investigate this question I procured a sample of beer made in Lafayette, Ind., which was guaranteed to contain no bicarbonate of soda, and which, from my knowledge of the parties through whom it was obtained, I have every reason to believe to be a sample of perfectly pure beer.<sup>4</sup>

The complete analysis of this sample was as follows:

Specific gravity .....	1.0201
Per cent. alcohol by weight .....	3.72
Do. alcohol by volume .....	4.64
Do. extract .....	6.34
Do. reducing sugar as maltose .....	1.42
Do. ash .....	.286
Do. fixed acid as lactic .....	.201
Do. volatile acid as acetic .....	.049
Do. phosphoric acid .....	.059
Do. carbonic acid .....	.238
Normal polarization .....	+69.7

Several portions of 100cc. each were taken, various quantities of bicarbonate of soda added, and after solution each portion evaporated

<sup>1</sup> See Wolff, *Aschen-Analysen*, p. 23.

<sup>2</sup> Page 89.

<sup>3</sup> Page 706.

<sup>4</sup> A small quantity of rice grit was admitted to have been used in its manufacture.

to dryness and burned to ash. The alkalinity of the ash was then ascertained, with the following results:

	No. 1.	No. 2.	No. 3.	No. 4.
Amount, in grams, of bicarbonate added....	None ....	.1	.5	1.0
Number of cubic centimeters decinormal acid required for neutralization.....	1.5	5.	17.5	30.

From these results it will be seen that the alkalinity of the ash shows very plainly the addition of considerable quantities of bicarbonate, but small additions would not be detected in this way. Again, the books state that the addition of bicarbonate of soda can be recognized by the strong effervescence of the ash with an acid. This statement is based entirely upon theoretical grounds, which have been shown by later investigations to be fallacious. When the acidity of normal beer was supposed to be due chiefly to lactic acid, the formation of lactate of soda, which would be converted into a carbonate on ignition, would, theoretically, make an ash which would effervesce strongly with acid. But now that it is known that the acidity is due to acid phosphates, it is easily seen that the addition of bicarbonate would only tend to the production of neutral or alkaline phosphates, which would give no effervescence with acid. This is shown by experiments I made with the sample of normal beer. Several portions of 100cc. each were taken, and different quantities of bicarbonate of soda added, and when solution had been effected the beer evaporated, and the residue carefully incinerated. The test for the presence of carbonate was made very carefully, by pouring a few drops of water on the ash, and turning the whole into a test-tube containing dilute acid.

No.	Amount bicarbonate of soda added.	Remarks.
1.....	None .....	No effervescence.
2.....	.1	Do.
3.....	.2	Do.
4.....	.5	Slight effervescence.
5.....	1.0	Strong effervescence.

It will be seen from the above that there was no carbonate in the ash until sufficient bicarbonate was added to the beer to neutralize all its acidity, leaving an excess of bicarbonate in the beer. In Nos. 4 and 5 the beer reacted alkaline before evaporation.

The same beer was allowed to stand until it had become quite sour and spoiled, when the above set of experiments was repeated, with the following results:

No.	Amount bicarbonate added.	Remarks.
1.....	None .....	No effervescence.
2.....	.1	Slight effervescence.
3.....	.2	Strong effervescence.
4.....	.5	Do.
5.....	1.0	Do.

From the above results it would seem that the bicarbonate united by preference with the acetic and lactic acids formed rather than with the acid phosphates of the beer, though it would require more experiments on the subject to thoroughly establish this point. If such is the case, however, it will be possible to detect the addition of bicarbonate to a beer that has soured, though not to a normal beer. The smallest quantity used in practice, according to the Brooklyn report, is one ounce to a half a barrel, which would be about 1 to 2,000, while the addition of .1 gram to 100cc. would be 1 to 1,000. Girard<sup>1</sup> gives as a test for the addition of bicarbonate of soda to ciders the following procedure: Decolorize with bone-black, evaporate to dryness, treat the residue with alcohol, which dissolves the acetates, which can be detected in the alcoholic solution. I have tried this test with very indifferent results, as I find the alcohol dissolves so much other matter from the dried residue that it is difficult to detect the presence of acetic acid in small quantities. The distillation of the beer in a current of steam<sup>2</sup> after the addition of phosphoric acid offers a better method for the separation of acetic acid, which may be searched for in the distillate, and if found to be present in any quantity, while the acidity of the beer itself is normal or below normal, the addition of bicarbonate of soda will be pretty well established. In this way I established the fact of the addition of bicarbonate in Nos. 4814 and 4816, which were the only samples of which I could be positive; and although I strongly suspected several of the other samples, owing to the difficulties of the test I would not pronounce positively upon them.

#### SALT.

A variable quantity of chloride of sodium is a normal constituent of all beers, being derived principally from the water used in the brewing. Even a slight further addition of salt might be deemed admissible to properly "season" the beer to the taste, just as breadstuffs are treated. Many brewers, however, are in the habit of adding a large quantity, either for the purpose of covering up some objectionable taste, or of increasing the thirst of the consumer. The English Government places

<sup>1</sup> Report Paris Municipal Laboratory.

<sup>2</sup> See under "Free volatile acids in wines," page 342.

the limit of chloride of soda which might come from the normal constituents at 50 grains to the gallon, or about .086 per cent., and treats any excess of that amount as evidence of an improper addition. This standard is undoubtedly a very generous one. Dr. Englehart found quite a large number of the samples examined by him to overstep the limit of 50 grains to the gallon, one sample containing as high as .338 per cent. Of the samples examined here none were beyond it.

#### ESTIMATION.

The estimation is very readily carried out on the ash, either gravimetrically or by a standard solution of silver nitrate with potassic chromate as indicator. For careful work the ash should be simply charred, so as to avoid loss of the chloride by volatilization, and the charred mass extracted by repeated additions of small quantities of hot water.

#### CLOUDY BEER.

Cloudiness in beer is sometimes due to the separating out of albuminous matter from changes in temperature, but usually to the presence of yeast, the fermentation not having been complete. This condition of things is best detected by means of the microscope, which shows the presence of quantities of yeast cells, and, in case other fermentations have set in, of their characteristic bacteria. "Yeast-cloudy" (*hefe trübes*) beer is considered unhealthy in Germany, and it is considered one of the qualifications of a good beer that it shall be absolutely bright and clear. An extensive investigation of the unhealthfulness of yeast-cloudy beer lately made by Dr. N. P. Simonowsky<sup>1</sup> in Pettenkofer's laboratory, who found that such beer had a disturbing effect in both natural and artificial digestion, producing in persons using it obstinate catarrh of the stomach, which persisted for some time. Both Simonowsky and Pettenkofer conclude that the sale of yeast-cloudy beer should be prohibited.

The Bavarian chemists at their last meeting at Würzburg, in August, 1886, adopted the following resolution in relation to yeast-cloudy beer:

Beers which are incompletely fermented for use must be entirely free from yeast; that is, must not contain yeast in a cloudy suspension.

<sup>1</sup>Zeit. für das gesammte Brauwesen ?, 9 Jahrg. 1886, No. 7, 8, 9; abstract Bied. Cent., 1887, p. 70



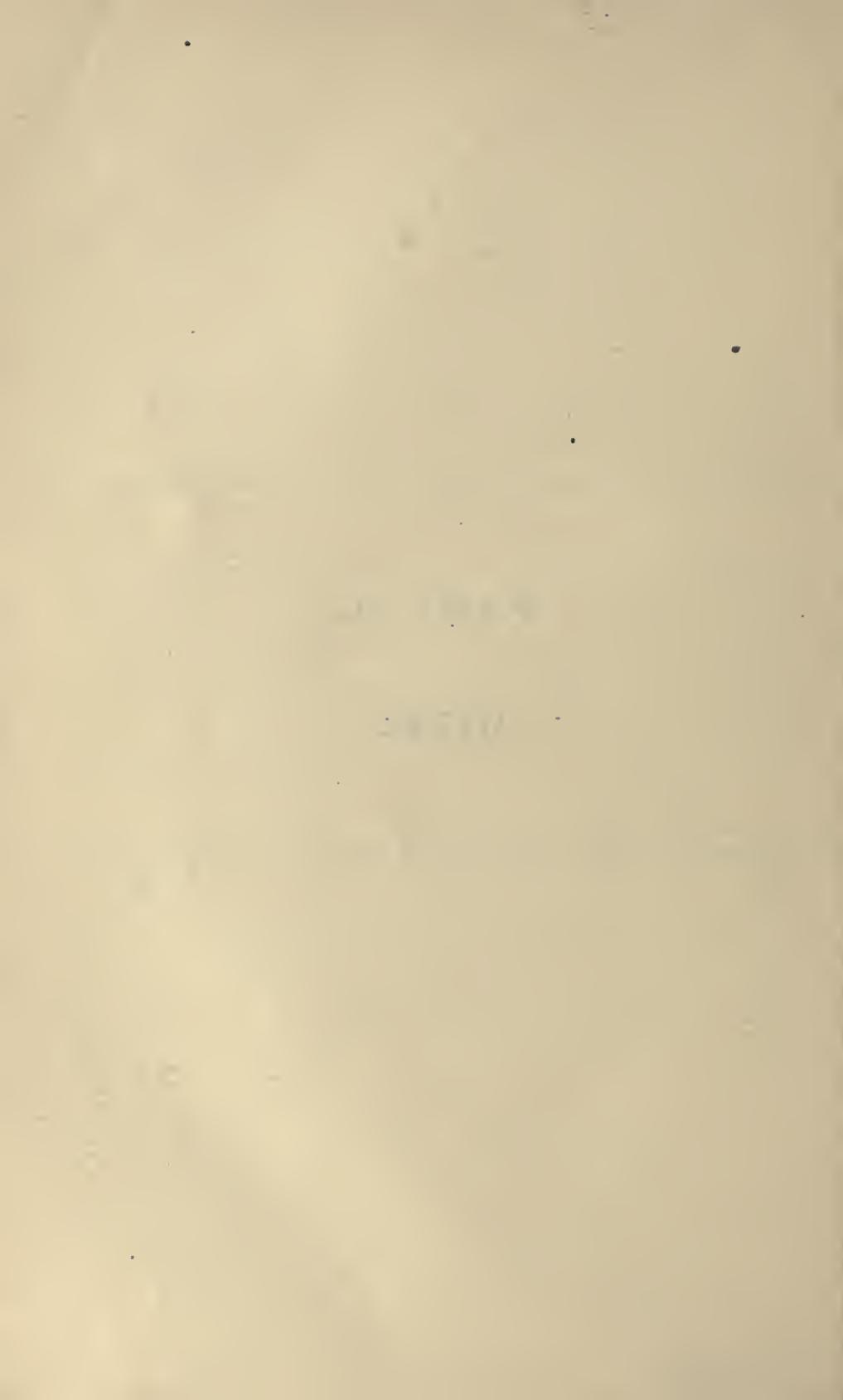
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**PART II.**

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**WINES.**

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## WINES.

The statistics in regard to the consumption and production of wines can be observed by referring to the table given under malt liquors (page 267), where it will be seen that in the year 1886, 22,067,220 gallons were consumed, of which 17,366,393 gallons were produced in this country. The consumption per capita has not increased very greatly during the forty-six years since 1840, but the total amount consumed has increased very greatly, it being less than 5,000,000 gallons in 1840. It will be noticed also that the amount produced in this country in proportion to the amount imported has increased to a remarkable degree. In 1840 there was about thirty-eight times as much wine imported as was produced in this country; in 1886 the amount of domestic wine consumed was nearly four times as great as the amount of wine imported. This does not fully represent the production, however, for it does not include the exports, which have increased very greatly of late years, as I am reliably informed, although I have no accurate data upon this point. The largely increased domestic production is principally due to the development of the industry in California.

The following table shows the relative rank of this country among the wine-producing countries of the world; it is taken from the same source as the preceding statistics:

### WINE PRODUCTION OF THE WORLD.

*Average production of wine in the principal wine-growing countries of the world.*

[Estimate by M. Tisserand in 1884, taken from "Journal of the Statistical Society," London, 1885.]

Countries.	Production.	Countries.	Production.
	<i>Imperial gallons.</i>		<i>Imperial gallons.</i>
France .....	765,175,972	Greece .....	28,600,000
Algeria .....	722,000,000	United States .....	18,000,000
Italy .....	605,000,000	Turkey .....	22,000,000
Spain .....	484,000,000	Cape of Good Hope .....	15,400,000
Austria-Hungary .....	187,000,000	Roumania .....	15,400,000
Portugal .....	88,000,000	Servia .....	11,000,000
Germany .....	81,290,000	Australia .....	1,933,800
Russia .....	77,000,000	Total .....	2,485,599,772
Cyprus .....	35,200,000		
Switzerland .....	28,600,000		

## PREPARATION OF WINE.

The growing of grapes for wine and the proper treatment of the juice for its conversion into wine have formed the subject of numerous treatises, that branch of technology having received a great deal of attention and study in countries where it is carried on. Only a short sketch of the leading features of the process can be given here, necessary to a proper understanding of the product itself.

Wine is properly the pure fermented juice of grapes; its composition is very variable, and the differences in the varieties of grapes used admit of almost endless modifications of the product obtained from them. Moreover, many other conditions affect more or less the composition of wine, as the nature of the soil, the climate, the method of cultivation pursued, the weather during the particular season when the grapes were ripened, &c. Thus the same variety of grapes when grown under different conditions of soil, climate, &c., produces different wines, and even in the same country the same variety of grape produces wines varying considerably in different seasons.

The most important constituent in the grape is its sugar, from which the alcohol is formed, so as a general rule the grapes are allowed to become fully ripened before they are removed from the vine. The first step is the production of the *must*. To this end the grapes are first bruised and crushed, either by the aid of machinery or by the more primitive but very effective method of trampling them by the feet of men. In some cases, and for very fine wine, the woody stems are removed from the crushed grapes (*dérâpage*). In other cases, especially in white wines, they are left, their contents of tannin making them a desirable addition to the grapes. To obtain the juice the grapes are subjected to pressure. The amount obtained varies with the means employed, the kind of grape, &c., but may be stated at about 60 to 70 per cent. of the weight of the grapes. For red wines the juice is allowed to stand in contact with the skins a variable length of time until it has acquired from them the desired depth of color, and in this case the fermentation commences before the juice is expressed. All musts contain pretty much the same proximate principles, their differences being due solely to the relative proportions of the different constituents. Briefly stated, these constituents are as follows:

1. Saccharine matter (chiefly dextrose), which may constitute as high as 25 to 30 per cent. of the must.
2. Albuminoid matter.
3. Gummy matter, peetin, &c.
4. Extractive matter, ill-defined substances, comprising the coloring matters, if any, the flavoring matters, &c.
5. Organic acids and their salts, comprising malic acid (especially in bad seasons), a slight trace of tannic acid derived either from the stems or skins, and tartrates of potassium and calcium.
6. Mineral matters: Phosphoric, sulphuric, hydrochloric, and silicic acids combined with potassium, sodium, iron, and magnesium.

### 7. Water, 70 to 90 per cent.

The must is fermented in suitable vats of wood or stone, according to the usage of the country; the fermentation is produced spontaneously, that is, by germs accidentally introduced into it from the air or on the surface of the grapes themselves. If the fermentation does not take place promptly it is started up by introducing into it a supply of yeast-cells from some must which is already in a state of fermentation. Sometimes a small quantity of must is fermented in anticipation of the vintage season as a "sponge," its fermentation being first induced by a small quantity of well washed beer yeast. The use of albuminous yeasts, such as bread yeast, &c., is generally avoided as much as possible, however, as tending to produce lactic and acetic or other objectionable fermentations entirely incompatible with the production of a wine with a delicate flavor.

The temperature at which the fermentation is carried on has a very decided influence upon the character of its product, and the practice differs in different countries in this respect. In California, Spain, South of France, Austria, and Hungary fermentation is conducted at a comparatively high temperature,  $15^{\circ}$  to  $20^{\circ}$  C., while in Germany a low temperature,  $5^{\circ}$  to  $15^{\circ}$  C., is employed. As with beer, the yeast of either variety of fermentation, high or low, reproduces the same kind of fermentation in musts to which it is added, but the subject of the different ferments, as applied to wine, has not been so carefully studied as with beer. The high fermentation is said to give a wine rich in alcohol, but lacking in bouquet, while the reverse is the case with the low fermentation.

The duration of the fermentation varies with the temperature, the amount of sugar to be transformed, &c.; the completion of the process may be known by the cessation of the disengagement of carbonic acid gas and by the diminution of the specific gravity of the liquid, so that the areometer marks zero or less.

After fermentation is complete, the wine is drawn off from any sediment it may contain into casks or barrels, where a second slow fermentation takes place, continuing sometimes several months. When it is over, the wine is "racked off" into fresh casks, which are closely bunged up. The operation of racking off may have to be repeated several times, and it is sometimes necessary to add isinglass, or other gelatinous material, which serves to clarify the liquid, acting on the tannin which it contains. This operation is called "fining."

### CHANGES PRODUCED BY FERMENTATION.

The principal change in the chemical constitution of the must produced by fermentation is the conversion of the sugar into alcohol and carbonic acid. One hundred parts of sugar produce 50 parts of alcohol, in round numbers. All the sugar, however, is not converted into alcohol and carbonic acid; a small part is converted into glycerine and succinic acid.

The bitartrate of potash, being insoluble in alcohol, is gradually deposited as the content of alcohol in the wine increases, and forms the substance known as "argol" or crude tartar. This distinctive constituent, tartaric acid, constitutes the superiority of grapes, over other fruits for wine-making purposes, the comparative insolubility of its acid salts furnishing a means of removing the excess without the addition of other chemical agents.

Other changes take place, especially during the slow second fermentation, not so well defined or so well understood as those mentioned, but of great importance in their relation to the quality of the final product. These changes, which continue after the fermentation has ended, constitute what is called the "ageing" of the wine and produce its "bouquet" or flavor, generally attributed to the etherification produced by a slow action of the acids upon the alcohols. Wine improves with age, but there is a limit after which it degenerates again and loses its flavor.

#### METHODS FOR "IMPROVING" WINES.

In France and Germany several methods are in use for increasing the yield of wine or improving its quality. These are especially resorted to in unfavorable seasons, when the want of sufficient sun prevents the formation of enough sugar in the grape and the proportion of acid is high.

*Chaptalization* consists in neutralizing the excess of acidity in the must by the addition of marble dust, and increasing the saccharine content by the addition of a certain quantity of cane sugar, which the vintners sometimes replace by starch sugar. In this process the quantity of the wine is not increased, but it becomes richer in alcohol, poorer in acid, and the bouquet is not injured. It is much used in Burgundy.

*Gallization*, which was invented by a German, Dr. Ludwig Gall, has for its object the production of a standard must, which shall contain a definite proportion of acid and sugar. This is brought about by the analysis of the must and the addition to it of water and sugar, the quantity to be added being ascertained by reference to tables.

*Petiotization*.—This process, which takes its name from Petiot, a proprietor in Burgundy, is carried out as follows: The *marc* from which the juice has been separated as usual by pressure is mixed with a solution of sugar and water, and the mixture again fermented—the second steeping containing, like the first, notable quantities of bitartrate of potash, tannic acid, &c., which are far from being exhausted by one extraction. The process may be repeated several times, the different infusions being mixed. This process is very largely used in France, and is said to produce wines rich in alcohol, of as good bouquet as the original wine, and of good keeping qualities. It is not allowed to be sold there, however, as *natural* wine.

To what extent these methods obtain in this country I am unable to state. It is probable, however, that they are but little used, as the principal fault found with American wines is their deficiency in bouquet,

not in their content of sugar. The detection of wines made in any of the above-mentioned ways is rather a difficult matter chemically, and requires a knowledge of the composition of the pure product only obtained from large numbers of analyses, extending over many years; which data, although existing in abundance in European countries, are, as yet, lacking here, owing to the comparatively recent development of the industry and the small amount of work done on the subject.

#### PRESERVATION OF WINE.

The method *par excellence* for the preservation of wines is Pasteurization, already alluded to in this report on malt liquors. The temperature employed is from 50° to 65° C., and serves to completely destroy all vegetable life in the wine. When a process so unobjectionable in every way answers its purpose so admirably, it furnishes an additional argument in favor of the legal suppression of all chemical means of arresting fermentation by the use of antiseptics, &c.

In regard to the use of antiseptics for the preservation of wines, I cannot do better than to give the opinion of Prof. E. W. Hilgard, of the University of California, who has probably done more than any other one man towards placing the wine industry of California upon a scientific basis, and whose work, published in the Bulletins of the State Agricultural Experiment Station, I shall have frequent occasion to refer to in the course of this investigation.<sup>1</sup>

\* \* \* \* \*

*Addition of antiseptics.*—As before stated, any of the fermentations above referred to may be stopped by the action of the substances known as disinfectants, antiseptics, or poisons. It should be unnecessary to argue regarding the admissibility of additions coming properly under the latter designation; yet it is true that in Europe such additions have not unfrequently been discovered in wines that, if left to themselves, would soon have become unsalable. It is not easy to draw the exact line between poisons proper and those substances of which the use to a certain degree, and in a certain way, may be considered admissible for the purpose of stopping undesirable fermentations in wines. There is, however, one point of view which covers the whole ground in connection with the use of wines for hygienic purposes, namely, that whatever impedes fermentations also impedes digestion, which is itself in a great degree a process of fermentation. The habitual use of wines containing antiseptics will, therefore, inevitably result in functional derangements; and this is so well understood that in Europe the extreme amounts of those allowed at all is strictly limited by law. Thus in the case of *sulphuric acid*, one of the germicides most commonly employed, partly in the form of the acid itself, but more commonly in that of plaster (sulphate of lime) added to the grapes, or to the wine itself. The tartaric acid of the wine is thus partially or wholly replaced by the sulphuric, tartrate of lime being thrown down; and thus badly made wines may be prevented from passing onward into the improper fermentations, and becoming undrinkable. *Salicylic acid* is effectual in much smaller quantities, and at one time it was thought that it would be admissible to employ it freely. But while its effects upon the human system are not apparent at first in most cases, yet the decided and unpleasant results often produced in the case of persons of weak digestion have but served to emphasize the general axiom, that we cannot, with impunity, continue to introduce into the human body substances foreign to the vegetable and animal products that have from time immemorial constituted the nutriment of mankind. If some persons are able to bear for a

<sup>1</sup> Report of Viticultural Work, 1883-'84, and 1884-'85, page 32.

time doses of salicylic acid that will completely stop digestion for some hours in the case of others, it is altogether unlikely that even the strongest person could continue its use indefinitely without injury. After some years of toleration, the legal prohibition of its use in articles of food or drink seems, in Europe, to be only a question of time; the more as in the case of wines, the process of "Pasteurizing" removes all legitimate reason for the longer continuation of a doubtful practice, liable to gross abuse.

In view of this fact, it is curious that its use for the conservation of must in the unfermented condition has not only been extensively introduced in this country, but the resulting beverage is especially recommended, as a healthful and harmless substitute for wine, by those who consider alcohol as necessarily harmful in any form and quantity. A few years' experience will doubtless show how unfortunate has been the choice of a substitute in this case.

And again as follows:<sup>1</sup>

Finally, when wines are not entirely sound—and with the methods of fermentation now in vogue this is a very prevalent condition—the remedy to be applied should not lie in the use of antiseptics, sulphuring, salicylic or boracic acids, and the like, but in the simple and rational heating process devised by Pasteur, and named for him. The "Pasteurizer" should be an indispensable appliance in every wine-house; and its use, if properly understood and practiced, will at once do away with nine-tenths of all doctoring for unsoundness. The universal adoption of this simple and inexpensive expedient will save all losses now sustained in the shipment of our young wines, and will soon do away with the reproach that "California wines will not keep."

If in the face of all these facts and legitimate substitutes for medication there are those who desire to adhere to the old doctoring system, it is at least the right of those who do without them and furnish the consumers the pure product of the grape to have a legalized mode of expressing the fact on the packages.

#### VARIETIES.

The different kinds of wines sold can be numbered by the hundreds. They refer usually either to the country where it is produced, or of whose product it is an imitation, as Port, Sherry, Hochheimer, Madeira, &c., or to the variety of grape from which it is made, as catawba, riesling, zinfandel, &c.

No generally recognized classification is made except into *white* or *red* wines according to their color; and into *dry* or *sweet* wines according to their content of sugar. The general name of *champagne* is given to effervescent wines.

#### COMPOSITION OF WINE.

In countries where the production of wine is one of the leading industries, like France and parts of Germany, the composition of the wines made is very well established. Scarcely any article of consumption has been the subject of so much chemical investigation as wine. Thousands of analyses have been published, so that one is at a loss to choose among them for representative figures.

In a general way the normal constituents of a natural wine may be divided into two classes, volatile and fixed.

The volatile matters are as follows: Water, constituting from 80 to 90 per cent. of the weight; alcohol, 5 to 15 per cent.; glycerine, 2 to 8

<sup>1</sup> Bulletin No. 65, Univ. of Cal. Agl. Ex. Station.

per cent.; volatile acids, acetic, oenanthic, &c., constituting one-fourth to one-third of the total acidity; aldehyde, compound ethers, together with the other fragrant, indefinite constituents, which give the wine its flavor and bouquet; carbonic acid gas in small quantities in young wines.

The fixed matters are: Glucose or grape sugar in small quantities in most wines; bitartrate of potash, tartaric, malic, and phosphoric acid, partly free and partly combined with potash, lime, soda, aluminum, magnesium, iron, and manganese, of which salts phosphate of lime is the most abundant, constituting from 20 to 60 per cent. of the weight of the ash, the remainder being chiefly carbonate of potash resulting from the calcination of the bitartrate, with a little sulphate and traces of chlorides. Coloring matters: Peetin and analogous gummy matters; tannin, 1 to 2 per cent. in red wines, mere traces only existing in white.

The following table of the composition of French wines is taken from Wurtz's *Dictionnaire de chimie*:

*Average composition of French wines.*

	Alcohol by vol- ume.	In grams per liter.					Remarks.
		Extract at 100° C.	Glycer- ine.	Bitar- trate of potash.	Ash.	Total acidity as $H_2SO_4$ .	
<i>Red wines.</i>							
Mean of French wines.	Per cent. 10.0	18.9	5.6-7.6	1.2-5.0	1.2-3.8	2.5	
Burgundy.....		16.9	5.8	1.8	.....	2.5	
Meursault.....	10.3	.....	.....	.....	.....	.....	
Pomard.....	11.5	.....	.....	.....	.....	.....	
Volnay.....	10.5	.....	.....	.....	.....	.....	
Richebourg, ordi- nary.	11.2	23.6	.....	.....	.....	.....	
Petits Burgundy ..	7.8	15.6	.....	.....	.....	.....	
Beaujolais.....	10.4	20.7	.....	.....	2.17	.....	
Bordeaux, ordinary.	9.4	.....	7.1	2.3	1.6-3.0	.....	
Bordeaux, superior.	9.1	16.4	.....	.....	.....	2.15	
Vins de l'Hérault, (not plastered).	10.1	19.0	6.5-7.6	2.2	1.7-3.5	2.5-5.6	
Vins de l'Hérault, (plastered).	10.3	20.0-25.0	.....	.....	3.2-4.0	4.9-5.1	
Aramon(plastered).	10.0	24.0	.....	.....	.....	2.05	
Beaux Narbonnes (1876).	12.4	.....	.....	.....	.....	4.2	
Narbonnes (not plastered).	10.8	18.8	.....	.....	.....	2.0-3.0	
Petits Narbonnes (1876).	7.8	.....	.....	.....	.....	.....	
Rousillons de table (1875).	14.8	.....	.....	.....	2.4	2.66	
Vins de la Marne ..	11.4	24.1	.....	.....	.....	1.8-5.3	
Vins de Cahors ..	10.7	.....	.....	.....	.....	.....	
Vins de la Haute- Garonne.	9.8	23.8	.....	1.4	1.4-2.0	.....	
<i>White wines.</i>							
Chablis (6 months) ..	9.7	14.0	.....	.....	.....	.....	
Bordeaux.....		.....	.....	.....	.....	2.51	
Picpoul .....	11.5	18.2	.....	3.5	.....	4.5	

The following table of the average composition of wines of various  
U. S. W.:

*Average composition of the wines of all*

Country.	No. of wines analyzed.	Specific gravity.			Alcohol by volume.			Acids, as tartaric.			
		No. of estimations.			No. of estimations.			No. of estimations.			
		Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	
America (Virginia)...	12	12. 1. 0.117	0.9875	0.9956	12	12.69 <sup>1</sup>	8.50 <sup>1</sup>	10.62 <sup>1</sup>	12. 1. 0.2	0.52	0.671
Australia...	5	.....	.....	.....	5	18.0	14.1	15.5	5. 0. 5.10	0.450	0.491
Africa...	4	1	.....	0.9938	2	20.3	18.0	19.1	3. 0. 3.70	0.224	0.275
Minor Asia...	8	7. 1. 0.0892	1. 0.051	1. 0.0235	8	18.0	13.0	14.3			
Crimea...	31	31. 1. 0.011	0.9875	0.9942	31	16.93	9.08	12.80	31. 0. 854	0.356	0.592
Greco...	9	7. 1. 0.0251	0.9909	1. 0.0109	9	18.0	12.4	15.4			
Spain...	9	7. 1. 0.0700	1. 0.0370	1. 0.0593	8	16.1	12.5	14.6	1. 0. 1	0.339	
Italy...	40	407. 1. 0.0879	0.9934	1. 0.0119	407	21.95	8.40	13.86	407. 1. 0.425	0.276	0.674
Sicily...	86	86. 1. 0.076	0.9895	1. 0.0225	86	27.15	13.70	18.08	86. 0. 8352	0.380	0.591
France...	60	27. 1. 0.0119	0.9910	1. 0.0932	47	14.0	0.5	9.9	40. 0. 970	0.191	0.506
Switzerland...	68	14. 0. 0.9930	0.9980	1. 0.0904	68	13.1	6.0	9.0	68. 0. 750	0.370	0.528
Austria...	523	488. 1. 0.0791	0.9806	1. 0.0941	503	18.8	7.5	11.0	409. 0. 995	0.111	0.588
1. Lower Austria...	122	113. 1. 0.084	0.9898	0.9857	122	15.7	8.6	13.0	113. 0. 910	0.434	0.632
2. Styria...	95	80. 1. 0.0797	0.9908	0.9967	86	15.3	8.1	12.0	92. 0. 995	0.116	0.661
3. Tyrol...	4	4. 0. 0.9960	0.9952	0.9959	4	8.3	7.5	7.9	3. 0. 0.012	0.461	0.523
4. Mähren...	4	4. 0. 0.9942	0.9932	0.9937	4	11.7	9.8	10.6	4. 0. 727	0.496	0.577
5. Bohemia...	19	19. 0. 0.994	0.9921	0.9950	19	14.1	9.5	11.6	19. 0. 800	0.426	0.613
6. Carniola...	11	11. 0. 0.9963	0.9939	0.9952	11	10.5	8.5	9.2	11. 0. 811	0.465	0.601
7. Hungary...	154	145. 1. 0.0201	0.9910	0.9963	148	18.8	8.4	12.2	144. 0. 955	0.349	0.635
8. Transylvania...	4	4. 0. 0.9928	0.9870	0.9914	4	12.8	10.5	11.7	4. 0. 498	0.433	0.469
9. Slavonia...	38	38. 1. 0.0112	0.9921	0.9941	38	15.1	9.3	11.3	38. 0. 928	0.573	0.697
10. Croatia...	72	70. 1. 0.086	0.9910	0.9943	66	13.6	7.5	10.5	70. 0. 944	0.449	0.687
11. Dalmatia...	1	1. 0. 0. 0.9950	1	.....					1. 0. 1	0.480	
Germany...	616	224. 1. 0.0833	0.9900	0.9954	615	16.0	5.7	10.1	544. 1. 354	0.050	0.599
1. Saxony...	2	1. 0. 0. 0.9975	2	.....					9. 0. 0		
2. Silesia...	1	1. 0. 0. 0.9976	1	.....					6. 5		
3. Mosel-Saar...	11	4. 0. 0.9977	0.9930	0.9946	11	14.2	6.7	10.6	7. 0. 0.660	0.560	0.612
4. Ahrgegend...	11	11. 0. 0.9960	0.9915	0.9944	11	11.2	7.9	9.8	11. 0. 559	0.390	0.485
5. Ittheingau...	57	42. 1. 0.0323	0.9960	0.9956	56	16.0	8.2	11.3	48. 0. 750	0.332	0.543
6. Rheingau-Berg- str...	30	26. 0. 0.9996	0.9910	0.9950	30	11.7	7.9	10.0	25. 0. 810	0.350	0.592
7. Palatinat...	62	41. 1. 0.0334	0.9868	0.9955	62	13.3	6.8	10.7	24. 0. 779	0.350	0.541
8. Franconia...	78	69. 1. 0.0833	0.9762	0.9940	78	13.3	6.5	10.6	73. 1. 334	0.453	0.850
9. Württemburg...	27	13. 0. 0.9982	0.9937	0.9956	27	15.5	7.0	11.4	23. 0. 830	0.350	0.618
10. Baden...	336	16. 0. 0.9986	0.9935	0.9945	336	15.6	5.7	11.2	333. 1. 087	0.050	0.546
11. Alsace...	12	37. 0. 0.9950	0.9860	0.9907	37	12.06	6.20	10.28	37. 0. 705	0.332	0.543
<i>German kinds.</i>											
Riesling...	85	17. 1. 0.0225	0.9911	0.9948	85	14.7	8.8	11.3	78. 0. 960	0.080	0.659
Traminer...	40	6. 0. 0.9907	0.9762	0.9921	40	14.3	8.7	11.8	30. 0. 907	0.330	0.588
Ruländer...	20	.....	.....	.....	20	14.6	9.4	11.0	19. 0. 780	0.420	0.562
Gutedel...	33	5. 0. 0.9939	0.9927	0.9934	33	12.1	8.9	10.4	32. 0. 705	0.274	0.531
Weißherbst...	15	2. 0. 0. 0.9960	1	.....	15	13.5	8.3	11.2	15. 0. 610	0.286	0.526
Burgunder...	26	2. 0. 0. 0.9950	1	.....	26	13.4	9.0	11.2	26. 0. 756	0.429	0.571
Clevnerblan...	30	6. 0. 0.9982	0.9933	0.9955	30	15.2	8.2	11.5	30. 0. 907	0.416	0.505

<sup>1</sup> Per cent. by weight.

origin consumed in Germany is taken from König's *Nahrungsmittel, countries (Wagenmann and König)*.

No. of esti- mations.	Sugar.			Extract.			Tannin and coloring matter.			Ash.					
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.			
12	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.	P. et.			
12	3.703	0.031	0.724	12	0.41	1.41	2.581	12	0.019 <sup>2</sup>	0.02 <sup>2</sup>	0.009 <sup>2</sup>	12	0.30	0.12	0.175
5	3.500	0.840	1.486	5	4.800	2.600	3.240	-----	-----	-----	-----	1	-----	0.428	
-----	-----	-----	2	5.510	4.190	4.580	-----	-----	-----	-----	-----	-----	-----	-----	
5	3.403	0.040	1.563	33	4.515	1.353	2.516	9	0.503	0.143	0.207	33	0.382	0.153	0.227
7	14.700	9.900	11.900	8	18.780	14.400	16.520	-----	-----	-----	-----	-----	-----	-----	
82	20.076	0.179	3.630	407	21.886	0.746	3.250	82	0.330	0.003	0.073	407	0.743	0.057	0.214
-----	-----	-----	86	27.940	0.746	8.442	-----	-----	-----	-----	-----	86	0.857	0.185	0.363
3	0.180	0.109	0.159	40	12.600	1.080	3.036	14	0.220	0.186	0.207	17	0.252	0.174	0.216
14	0.120	0.025	0.063	14	2.689	1.293	1.878	-----	-----	-----	-----	14	0.502	0.134	0.240
9	6.100	1.200	2.647	495	23.140	0.870	2.422	15	0.194	0.109	0.130	186	0.323	0.077	0.188
-----	-----	-----	122	6.320	1.790	2.650	10	0.194	0.109	0.130	17	0.311	0.162	0.241	
2	3.360	77.23.140	1.090	3.730	-----	-----	-----	-----	-----	-----	47	0.305	0.087	0.165	
-----	-----	-----	4	1.850	1.070	1.750	-----	-----	-----	-----	4	0.228	0.154	0.184	
-----	-----	-----	4	2.280	1.880	2.010	-----	-----	-----	-----	-----	-----	-----	-----	
-----	-----	-----	19	3.910	1.850	2.620	-----	-----	-----	-----	7	0.297	0.128	0.197	
1	1.620	11.2.510	1.480	2.000	-----	-----	-----	-----	-----	-----	11	0.269	0.120	0.182	
6	6.100	1.200	2.960	149	10.700	0.870	3.050	5	0.175	0.111	0.141	36	0.288	0.077	0.192
-----	-----	-----	4	2.170	1.450	1.750	-----	-----	-----	-----	14	0.286	0.145	0.191	
-----	-----	-----	38	3.890	1.510	2.360	-----	-----	-----	-----	49	0.323	0.111	0.167	
-----	-----	-----	66	5.000	1.210	2.140	-----	-----	-----	-----	1	-----	0.170	-----	
409	8.628	0.010	0.470	386	10.555	0.520	2.502	41	0.272	0.076	0.145	94	0.314	0.108	0.194
1	-----	1.000	2	-----	-----	3.000	-----	-----	-----	-----	-----	-----	-----	-----	
-----	-----	-----	1	-----	-----	2.100	-----	-----	-----	-----	-----	-----	-----	-----	
7	0.520	0.120	0.241	9	2.500	1.500	1.892	-----	-----	-----	-----	1	-----	0.203	
11	0.674	0.056	0.159	11	2.885	2.137	2.671	10	0.272	0.090	0.214	11	0.261	0.099	0.213
30	8.628	0.080	0.964	56	10.555	1.640	3.087	16	0.261	0.091	0.141	29	0.314	0.120	0.215
15	1.500	0.100	0.410	31	4.100	1.040	2.588	13	0.235	0.091	0.148	17	0.275	0.125	0.213
43	3.500	0.010	0.560	49	7.300	1.710	2.236	2	-----	0.076	-----	22	0.205	0.108	0.137
6	0.154	0.081	0.138	76	9.445	1.112	3.165	-----	-----	-----	-----	-----	-----	-----	
18	1.400	0.080	0.305	17	2.920	2.750	2.255	-----	-----	-----	-----	-----	-----	-----	
279	4.540	0.046	0.442	134	3.545	0.520	1.730	-----	-----	-----	-----	14	0.255	0.163	0.183
29	0.188	0.013	0.087	36	2.389	1.228	1.784	-----	-----	-----	-----	25	0.387	0.105	0.225
75	4.540	0.091	0.458	36	9.940	0.790	2.462	-----	-----	-----	-----	5	0.190	0.146	0.170
33	4.160	0.046	0.480	16	3.612	0.520	2.200	-----	-----	-----	-----	2	-----	0.183	
20	1.500	0.059	0.239	14	2.700	1.320	1.848	-----	-----	-----	-----	-----	-----	-----	
26	0.610	0.077	0.124	24	2.735	1.420	1.781	-----	-----	-----	-----	5	0.183	0.160	0.172
12	2.100	0.070	0.586	4	2.290	1.770	2.012	-----	-----	-----	-----	2	-----	0.190	
23	0.275	0.087	0.119	14	2.706	1.360	2.170	1	-----	0.272	-----	2	-----	0.229	
28	3.330	0.076	0.871	12	4.167	1.390	2.364	3	0.272	0.190	0.230	3	0.253	0.181	0.212

<sup>2</sup> Given as tannic acid.

## COMPOSITION OF AMERICAN WINES.

The earliest analyses of American wines on record were made by Merrick,<sup>1</sup> in 1875, comprising six varieties of California wines.

In October of the same year Mallet and Cooper<sup>2</sup> published analyses of twelve samples of Virginia wines. The mean of these analyses is given in the table from König.

The work of Professor Hilgard on California wines began in 1880 and has continued down to the present day, the results being published in the Bulletins of the Station. These publications include extensive series of analyses, which afford a most valuable index of the composition of California wines, especially as many of the analyses were made on wines manufactured in the laboratory and hence known to be absolutely pure. A standard of composition could very properly be established from them, and a limit for the amount of each constituent present in pure wines, by which the addition of alcohol, water, sugar, &c., in sophisticated wines could be detected. The number of different determinations made on each sample is not very large, unfortunately, including only the more important constituents. I give below tables of the entire work done on wines known to be pure:

*Composition of wines made at the Viticultural Laboratory, 1884.*

Variety.	Body.	Alcohol.		Tannin.	Acid as tartaric.	Ash.
		By weight.	By volume.			
<i>Bordeaux type.</i>						
Malbeck .....	2.68	8.34	10.42	.100	.450	.393
Cabernet Franc .....	2.84	9.63	12.00	.035	.480	.432
Do .....	2.13	8.48	10.58	.070	.607	.293
Cabernet Sauvignon .....	3.19	9.92	12.36	.079	.540	.447
Merlot .....	2.44	9.20	11.42	.065	.467	.394
Verdot .....	2.77	9.78	11.82	.071	.438	.409
Tannat .....	2.69	7.46	8.92	.171	.633	.353
Beclan .....	2.64	8.84	11.00	.053	.381	.260
Carignano .....	2.18	7.92	9.90	.063	.527	.285
Grossblaue .....	2.10	9.20	11.42	.065	.572	.252
<i>Burgundy type.</i>						
Black Burgundy .....	2.77	9.34	11.64	.188	.705	.277
Black Pinot .....	2.11	6.42	8.00	.133	.750	.310
Meunier .....	2.55	10.07	12.55	.025	.525	.419
Do .....	2.44	6.95	8.64	.055	.622	.285
Zinfandel .....						
Do .....	2.36	7.85	9.75	.036	.450	.425
Do .....	1.93	7.43	9.25	.080	.633	.200
Do .....						
Do .....						
<i>Southern French and Italian types.</i>						
Trousseau .....	2.66	11.23	13.73	.075	.485	.379
Do .....	2.28	9.92	11.64	.050	.474	.349
Petite Sirah .....	2.65	10.07	12.54	.092	.401	.406
Do .....	2.79	10.81	13.27	.108	.293	.339
Serine .....	2.49	8.48	10.58	.063	.510	.400

## Composition of wines made at the Viticultural Laboratory, 1884.

Variety.	Body.	Alcohol.		Tannin.	Acid as tartaric.	Ash.
		By weight.	By volume			
<i>Southern French and Italian types—Continued.</i>						
Mondeuse.....		2.64	9.56	11.89	.173	.498
Do.....		2.84	9.92	12.27	.141	.405
Cinsaut.....		2.60	10.44	12.90	.070	.490
Aramon.....		2.32	9.05	10.73	.065	.496
Mourastel.....		2.60	7.92	9.88	.054	.553
Grenache.....		1.67	8.48	10.58	.105	.480
Do.....		1.93	7.43	9.27	.065	.532
Petit Bouschet.....		2.44	8.84	11.00	.089	.583
Do.....		2.87	9.27	11.55	.117	.493
Clairetto Rouge.....		2.72	9.92	12.86	.063	.510
Barbera.....						
Lenoir.....		3.00	8.13	10.16	.060	.630
Do.....		3.71	8.98	11.17	.075	.582
Blan-Ebling.....		1.93	8.27	10.25	.040	.585
Mission.....						
Black Prince.....						
<i>Dry white wine varieties.</i>						
Semillon.....						
Do.....		1.93	9.92	12.36		.450
Sauvignon Blane.....						
Do.....		2.10	9.92	12.36		.453
Do.....		2.18	10.26	12.75		.553
Muscadello du Bordelais (loose bunches).....		2.13	11.46	14.00		.432
Muscadello du Bordelais (compact bunches).....		2.44	10.81	13.27		.498
Folle Blanche ("Tannat").....		1.66	9.92	12.36		.593
Folle Blanche.....		1.80	7.43	8.75		.570
Burger.....		2.05	8.41	10.50		.600
Do.....		1.65	8.55	10.63		.528
Roussanne.....		1.36	12.54	13.00		.468
Marsanne.....		1.88	9.20	11.46		.563
Clairette Blanche.....		1.85	10.54	13.00		.428
"Pecoui Touar" (?).....		1.66	9.27	11.58		.573
<i>Sherry and Madeira varieties.</i>						
Pedro Jimenes.....		1.18	9.63	12.00		.555
Palomino.....		1.85	11.08	13.50		.528
Pernino.....		1.95	9.92	12.50		.519
Mantuo de Pilas.....		1.32	7.85	9.82		.525
Monrisco Branco.....						
Beba.....		2.10	11.69	14.27		.503
Verdelho.....		2.82	12.39	15.20		.417
Boal Madeira.....		2.00	9.27	11.58		.660
Ugni Blanc.....		2.00	8.91	11.10		.510
Malmsey.....		1.60	7.92	9.91		.543
Malaga.....		1.69	8.13	9.82		.217
<i>Port wine varieties.</i>						
Tinta Cao.....						
Tinta Madeira.....						
Mourisco Preto.....						
Tinta Amarela (not fully ripe).....		2.36	9.05	11.27	.058	.501
Tinta Amarela (fully ripe).....		2.39	9.56	11.91	Slight	.525
Moretto.....						
Bastardo.....		3.12	10.35	12.83	.062	.507
						.564

*Analyses of wines made at the Viticultural Laboratory, 1885.*

Variety.	Body.	Alcohol.		Tannin.	Acid.		Ash.
		By weight.	By volume.		Tartaric.	Volatile.	
<i>Bordeaux type.</i>							
Malbeck <sup>1</sup>				Per cent.	Per cent.	Per cent.	P. ct.
Pfeffer's Cabernet	2.99	8.84	11.00	.123	.846	-----	.273
Do	2.69	10.54	13.00	.101	.516	-----	.290
Do	2.69	8.13	10.17	{ (1).179	{ .486	-----	.366
Merlet	2.69	9.20	11.45	{ (2).153	{ .420	-----	.310
<i>Burgundy type.</i>							
Burgundy	3.07	9.63	12.00	.071	.555	-----	.226
Bl. Pinot or Tree Burgundy	2.84	8.91	11.09	.110	.600	-----	.315
Black Pinot	2.20	7.37	9.18	.040	.525	-----	.214
Franc Pinot <sup>1</sup>							
Meunier <sup>1</sup>							
Zinfandel <sup>1</sup>							
Do	2.69	8.84	11.00	.086	.576	-----	.290
Do	2.69	11.46	14.00	.133	.417	-----	.280
Charbono <sup>1</sup>							
Do	2.69	7.09	8.85	.112	.495	-----	.270
Do	2.26	7.78	9.73	.160	.420	-----	.290
Do	2.18	7.23	9.00	.209	.547	-----	.270
Do	1.80	6.42	8.08	.130	.502	-----	.330
<i>Southern French and Italian type.</i>							
Trousseau <sup>1</sup>							
Mataro	1.98	7.23	9.00	.080	.487	-----	.290
Teinturier	3.07	8.06	10.08	.184	.555	-----	.340
Kadarka	1.80	6.82	8.54	.070	.450	-----	.236
Blend: 90 per cent. Zinfandel, 10 per cent. Charbono	2.84	8.84	11.00	.108	.576	-----	.213
<i>Dry white varieties.</i>							
Chanché Gris	1.80	10.54	13.00	-----	.351	-----	.250
Verdal	1.80	6.42	8.07	-----	.517	-----	.188
One-third Golden Chasselas, two-thirds Burger	1.80	9.63	12.00	-----	.615	-----	.217
Burger	2.69	9.63	12.00	-----	.675	-----	.450
Do	1.93	8.13	10.16	-----	.645	-----	.192
Do	1.68	8.84	11.00	-----	.540	-----	.183
Do	1.52	4.76	6.00	-----	.480	-----	.225
Franken Riesling <sup>2</sup>	3.82	8.48	10.58	-----	.600	.366	.270
Riesling	1.80	9.05	11.27	-----	.352	-----	.160
Colombar	2.26	8.84	11.00	-----	.591	-----	.280
Zinfandel, first crop	2.30	11.23	13.73	-----	.481	-----	.244
Zinfandel, second crop	2.15	10.54	13.00	-----	.525	-----	.140
Zinfandel, second crop, "filtered"	2.60	10.54	13.00	-----	.576	-----	.187
Zinfandel, 97½ per cent.; Verdal, 2½ per cent.	2.15	10.54	13.00	-----	.503	-----	.191
Burger, 50 per cent.; Zinfandel 50 per cent.	2.10	11.08	13.54	-----	.713	-----	.184

<sup>1</sup> Not enough for wine-making.<sup>2</sup> Sugar = 65 per cent.

*Composition of wines made at Viticultural Laboratory, 1886.*

Variety.	Alcohol.			
	By weight.	By volume.	Tannin.	Acid as tartaric, at pressing.
<i>Bordeaux type.</i>				
Malbeck .....	8.27	10.36	.132	.62
Cabernet Franc.....	9.78	12.18	.264	.45
Cabernet Sauvignon.....	8.48	10.58	.226	.52
Pfeiffer's Cabernet.....	9.05	11.27	.190	.42
Do .....	8.34	10.42	.208	.73
Gros Verdot.....	7.92	9.90	.166	.79
Tannat .....	9.70	12.09	.316	.77
Beelan .....	8.34	10.42	.090	.60
Charbono .....	7.23	9.00	.251	.53
Do .....	7.64	9.54	.186	.60
Carignane .....	9.27	11.55	.172	.69
Do .....	6.95	8.70	.169	.81
Grossblane .....	7.09	8.85	.193	.72
Black Hamburg.....	7.23	9.00	.108	.52
West's St. Peter's (?) .....	8.84	11.00	.276	.57
<i>Burgundy type.</i>				
Burgundy .....	9.99	12.46	.226	.43
Crabb's Black Burgundy .....	7.64	9.54	.220	.47
Do .....	9.92	12.36	.074	.41
"Burgundy" (Chauché Noir ?) .....	8.48	10.58	.106	.38
Chauché Noir .....	9.92	12.36	.076	.53
Petit Pinot (?) .....	10.81	13.27	.153	.35
Pinot (?) .....	9.02	12.36	.112	.53
Pinot St. George .....	9.49	11.82	.133	.55
Meunier .....	9.92	12.36	.193	.40
Zinfandel .....	9.92	12.36	.104	.54
Do .....	11.62	14.20	.140	.54
Do .....	0.20	11.45	.091	.59
Do .....	8.48	10.58	.160	.94
Do .....	7.23	9.00	.148	.55
Zinfandel, second crop .....	7.78	9.73	.103	.71
Zinfandel (?) (Row 31) .....	7.09	8.85	.151	.64
<i>Southern French and Italian types.</i>				
Trousscan .....	10.63	13.00	.090	.45
Ploussard .....	9.56	11.91	.051	.51
Sirah .....	8.84	11.00	.107	.65
Mondense .....	7.64	9.54	.127	.78
Do .....	7.99	10.10	.317	.64
Cinsant .....	7.78	9.73	.153	.90
Do .....	7.90	10.00	.110	.52
Barbera .....	9.20	11.45	.153	.85
Teinturier .....	9.85	12.27	.278	.73
Do .....	8.34	10.42	.114	.64
Gamay Teinturier .....	8.48	10.58	.169	.90
Nebbiolo Bourgu.....	8.13	10.17	.172	.82
Nebbiolo fino .....	8.91	11.69	.215	.86
Fresa .....	6.49	8.15	.160	1.12
Blend: Barbera; Herbemont, one-third; Fresa .....	7.37	9.18	.256	.63
<i>American type.</i>				
Herbemont .....	6.89	8.61	.095	.74
Do .....	7.43	9.27	.146	.50
Californica .....	7.09	8.85	.179	.61
Isabella regia .....				
<i>Dry white wine varieties.</i>				
Semillon .....	7.23	9.00		.55
Sauvignon Vert .....	7.23	9.00		.53
Follo Blanche .....	7.78	9.73		.59
Burger .....	8.48	10.56		.47

*Composition of wines made at Viticultural Laboratory, 1886—Continued.*

Variety.	Alcohol.		Tannin.	Acid as tartaric, at pressing.
	By weight.	By volume.		
<i>Dry white wine varieties—Continued.</i>				
Burger.....	Per cent.	Per cent.	P. ct.	P. ct.
Do.....	4.14	5.23		.81
Kleinberger.....	7.99	10.00		.33
Marsanno.....	6.95	8.70		.43
Clairette Blanche.....	8.48	10.58		.56
Do.....	7.09	8.85		.47
Do.....	7.57	9.45		.35
Do.....	8.48	10.58		.47
"Pecoui Tonar".....	7.09	8.85		.45
Franken Riesling.....				
Do.....	7.23	9.00		.50
Riesling.....	7.37	9.18		.36
Riesling (?).....	9.20	11.45		.55
Johannisberger Riesling.....	8.13	10.17		.60
Chasselas de Fontainebleau.....	6.89	8.61		.41
Chauch�� Gris.....	8.27	10.30		.45
"Gray d Ischia," Chauch�� Gris.....	8.98	11.18		.38
Seedless Sultana.....	8.27	10.36		.51
White Muscateller.....	8.70	10.83		.44
Blau-Elbling.....	8.13	10.17		.54
White Zinfandel.....	9.05	11.27		.41
Cinsaut.....	9.27	11.55		.48
Verdal.....	8.48	10.58		.25
Do.....				
<i>Sherry and Madeira varieties.</i>				
Pedro Jimenes.....	7.92	9.90		.42
Palomino.....				
Palomino ("Golden Chasselas").....	6.35	8.00		.54
Mautuo de Pilas.....	7.78	9.73		.45
Do.....	8.48	10.58		.35
Mourisco Branco.....	10.07	12.54		.33
Verdelho.....				
Muscat of Alexandria.....	11.62	14.20		.42
Do.....	9.56	11.91		.36
Do.....				
West's White Prolific, with some smaller grapes.....	7.78	9.73		.72
West's White Prolific.....	7.78	9.73		.71
"White St. Peter's".....	8.48	10.58		.45

As this work seems to be very important as establishing the average composition of pure wines made in California, I have prepared from Professor Hilgard's reports a table showing the maximum, minimum, and mean composition of the pure wines analysed, as well as of the wines which were made outside and sent in to the laboratory for analysis:

*Maximum, minimum, and mean composition of California wines, as shown by the analyses made at the California State Viticultural Laboratory.*

	No. of samples analyzed.	Alcohol by weight.			Body or extract.			Total acids as tar-taric.			Ash.		
		Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
<i>Pure wines made at laboratory in 1884.</i>													
Bordeaux type .....	10	9.92	7.46	8.81	3.19	2.10	2.57	.633	.381	.510	.447	.252	.352
Burgundy type .....	6	10.07	6.42	9.01	2.77	1.93	2.36	.765	.450	.624	.425	.200	.319
Southern French and Italian type .....	18	11.23	7.43	9.26	3.71	1.67	2.56	.585	.393	.506	.511	.234	.343
Dry white wine varieties .....	13	11.46	7.43	9.71	2.44	1.36	1.90	.600	.428	.516	.342	.157	.219
Sherry and Madeira varieties .....	10	12.39	7.85	9.68	2.82	1.18	1.83	.660	.217	.498	.390	.160	.288
Port wine varieties .....	3	10.35	9.05	9.65	3.12	2.36	2.62	.525	.501	.511	.564	.446	.493
<i>Pure wines made at laboratory in 1885.</i>													
Bordeaux type .....	4	10.54	8.13	9.18	2.99	2.69	2.76	.846	.420	.567	.366	.273	.310
Burgundy type .....	9	11.46	6.42	8.30	3.07	1.80	2.49	.600	.417	.515	.330	.214	.276
Southern French and Italian type .....	4	8.84	6.82	7.74	3.07	1.80	2.42	.576	.450	.517	.340	.213	.270
Dry white wine varieties .....	15	11.23	4.76	9.22	3.82	1.52	2.10	.713	.351	.544	.450	.140	.224
<i>Pure wines made at laboratory in 1886.</i>													
Bordeaux type .....	15	9.78	6.95	8.28	.....	.....	.....	.810	.420	.620	.....	.....	.....
Burgundy type .....	16	11.62	7.09	9.21	.....	.....	.....	.940	.350	.540	.....	.....	.....
Southern French and Italian type .....	15	10.63	6.49	8.48	.....	.....	.....	1.120	.450	.730	.....	.....	.....
Dry white wine varieties .....	25	9.27	4.14	7.86	.....	.....	.....	.810	.250	.480	.....	.....	.....
Sherry and Madeira varieties .....	10	11.62	6.35	8.58	.....	.....	.....	.720	.330	.480	.....	.....	.....
American type .....	3	7.43	6.89	7.14	.....	.....	.....	.740	.500	.620	.....	.....	.....
<i>Wines sent to laboratory for analysis, 1884-'85.</i>													
Reds .....	20	10.69	7.64	9.63	3.62	2.05	2.87	.750	.395	.563	.534	.219	.340
Whites .....	5	10.81	8.98	9.80	3.87	2.05	2.67	.527	.397	.473	.367	.255	.303
<i>Wines sent to laboratory for analysis, 1885-'86.</i>													
Reds .....	55	16.42	7.99	10.48	13.77	2.10	4.30	.879	.225	.554	.470	.230	.324
Whites .....	16	12.39	8.84	10.82	4.20	1.80	2.66	.750	.210	.473	.300	.170	.261
Zinfandel wines analyzed, 1879-'85. ....	45	12.39	7.43	10.55	8.64	1.46	2.89	.873	.337	.573	.546	.154	.301

In the year 1880 a large number of samples of wine were purchased in the market of Washington and analyzed by the Department of Agriculture. The work was under the charge of the late Henry B. Parsons, one of the most competent analysts ever in the service of the Department. The results are published in the Annual Report for 1880, forming part of the Chemists' report for that year. I have thought it proper to reproduce them here, as the results include a large number of determinations on each sample, and give very important information in regard to the composition of American wines as they are supplied to the consumer.

## I.—DRY RED WINES.

No.	Name.	Specific gravity.	Alcohol, per cent.	Total residue.	Total ash.	Tartaric acid, as ascorbic acid.	Pyridine acetate.	Glucose.	Total acid.	Pyridine acetate, tartaric acid.	Volatile acid.	Ascorbic acid.	Maker.
3	Virginia Claret, Concord, 1879.	.9941	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Monticello Wine Company.
16	Virginia Claret, Concord, 1879.	.9933	9.61	12.05	0.193	0.13	0.725	0.363	0.206	0.363	0.426	0.206	Do.
17	Virginia Claret, 1879.	.9950	9.82	12.31	2.56	1.74	0.709	.784	.513	.784	.817	.513	Do.
18	Cynthiana, 1880.	.9969	10.24	12.87	2.95	2.38	None	647	376	647	621	376	Do.
19	Alrey, 1880.	.9931	9.77	12.22	2.13	1.74	Trace...	.09	.408	Trace...	.146	.408	Do.
20	Norton's Virginia, 1879.	.9937	10.21	12.17	2.88	2.98	do...	.772	.316	do...	.316	.772	Do.
22	Ives' Seedling, 1879.	.9944	8.68	10.82	2.18	2.47	do...	.723	.512	do...	.512	.723	Do.
32	Norton's Virginia, 1875.	.9906	6.36	8.01	2.62	2.75	do...	.825	.355	do...	.355	.825	White Elk Vineyards.
35	do.	.9982	8.26	10.38	2.95	2.95	do...	.762	.481	do...	.481	.762	Do.
31	Concord, 1873.	1.0011	5.75	7.25	2.80	2.73	do...	.704	.385	do...	.385	.704	Do.
33	do.	.9970	7.65	9.62	2.53	3.59	None	.722	.316	do...	.325	.722	Do.
33	Clinton, 1872.	.9982	5.71	7.17	0.97	1.92	Trace...	.902	.292	do...	.488	.902	Do.
37	do.	.9961	7.98	9.95	2.19	1.96	None	.798	.391	do...	.326	.798	Do.
34	St. Julien.	.9939	7.14	8.96	1.99	2.10	do...	.587	.247	do...	.247	.587	Do.
35	La Rose.	.9987	7.91	9.95	2.61	2.68	do...	.766	.400	do...	.400	.766	Do.
59	Claret, 1874.	.9985	9.02	11.35	2.09	2.36	do...	.826	.411	do...	.411	.826	Do.
60	Concord, 1880.	.9823	8.72	10.91	2.38	2.38	do...	.45	.619	do...	.619	.332	C. Xander.
61	Clinton, 1880.	.9920	10.90	13.62	2.49	1.65	do...	.30	.020	do...	.020	.324	Do.
62	Norton's Virginia, 1880.	.9925	8.65	10.82	1.17	1.52	do...	.20	.680	do...	.254	.680	Do.
63	Ives and Clinton, 1880.	.9941	8.99	11.26	2.38	2.22	do...	.12	.662	do...	.283	.662	Do.
64	Ives and Clinton, 1879.	.9920	9.62	12.05	2.17	1.83	Trace...	.635	.372	do...	.372	.635	Do.
65	Ives and Clinton, 1879.	.9928	9.28	11.61	2.49	2.08	do...	.709	.560	do...	.560	.709	Do.
84	Concord and Clinton, 1879.	.9943	9.76	12.22	2.38	2.02	do...	.754	.395	do...	.395	.754	Do.
87	Franklin, 1868.	.9935	8.77	11.00	2.32	1.66	do...	.659	.362	do...	.362	.659	Do.
74	Lothlink, 1868.	.9935	9.72	10.91	1.06	1.61	do...	.830	.233	do...	.233	.830	Julius Ilmeke.
74	Ruby Claret, 1875.	.9910	9.73	12.13	2.22	1.56	do...	.726	.475	do...	.475	.726	J. H. Barnhhr.
76	Ruby Claret, 1876.	.9917	10.05	12.87	2.06	1.86	do...	.696	.463	do...	.463	.696	Do.
76	Ruby Claret, 1877.	.9927	9.56	11.96	1.94	1.65	do...	.695	.438	do...	.438	.695	Do.
78	Ruby Claret, 1878.	.9902	11.82	14.74	1.87	1.40	do...	.667	.373	do...	.373	.667	Do.
78	Ruby Claret, 1879.	.9918	10.15	12.68	1.84	1.54	do...	.650	.399	do...	.399	.650	Do.
79	Ruby Claret, 1880.	.9922	10.75	13.43	1.82	1.83	do...	.544	.372	do...	.372	.544	Do.
80	Clevenor, 1876.	.9984	7.94	9.95	2.15	2.50	do...	.511	.264	do...	.264	.511	Do.
81	Cynthiana, 1876.	.9939	7.94	9.95	2.28	2.08	do...	.770	.526	do...	.526	.770	Do.
82	Franklin, 1876.	.9945	8.64	10.82	2.01	1.80	do...	.724	.347	do...	.347	.724	Do.
83	Norton's Virginia, 1877.	.9914	10.38	12.96	1.87	1.66	do...	.655	.378	do...	.378	.655	Do.
83	Ives' Seedling, 1880.	.9925	9.55	11.73	1.98	1.76	do...	.653	.378	do...	.378	.653	Poeschel, Scherer & Co.

157	Cynthiana, 1880.	9.26	11.61	2.66	343	...do...	.561	.218	Poeschel, Scherer & Co.
158	Virginia, Scoddling, 1880	.9952	12.21	15.21	285	...do...	.540	.185	Do.
160	Virginia, Scoddling, 1880	.9954	15.40	9.89	220	...do...	.494	.302	Do.
162	Concord, 1880	.9950	12.40	3.00	365	...do...	.494	.154	Do.
163	Sonoma Red Mission, 1879	.9913	10.38	12.96	233	Trace...	.496	.302	Gretsch & Mayer.
147	Sonoma Red Mission, 1879	.9968	7.99	10.03	428	None...	.729	.301	Do.
154	Virginia, Zinfandel, 1879	.9962	7.80	9.78	243	255 Trace...	.693	.391	Gretsch & Mayer.
159	Virginia, Concord, 1879	.9965	7.60	9.54	211	231 ...do...	.753	.421	Do.
89	Virginia, Concord	.9944	8.43	10.56	185	130 ...do...	.753	.421	Do.
90	Virginia, Seedling	.9938	9.62	12.05	206	218 ...do...	.648	.272	Isaac Cook.
91	Fine Claret	.9937	9.21	9.52	197	189 ...do...	.648	.272	Do.
92	Burgundy	.9940	9.21	11.52	203	210 ...do...	.664	.287	Do.
120	"Land N," 1880	.9949	8.22	10.30	209	318 None...	.754	.242	Wehrle, Werk & Co.
121	Norton, 1880	.9988	12.14	15.12	232	176 Trace...	.754	.362	Do.
122	Ives, 1880	.9901	6.59	8.27	172	198 None...	.516	.258	Do.
124	Concord, 1880	.9947	8.14	10.21	217	209 ...do...	.602	.346	Do.
15	Black Rose	.9920	9.86	12.31	194	170 Trace...	.618	.347	Do.
56	"Old Dominion,"	.9970	6.16	7.74	165	160 ...do...	.756	.287	Charles Saalmann.
126	"Old Dominion,"	.9947	7.01	8.20	152	152 Trace...	.707	.277	C. A. Hefner.
1127	"Prince William,"	.9945	10.20	12.77	316	297 ...do...	.782	.391	Do.
114	Mission Claret,	.9975	7.39	9.29	240	532 ...do...	.699	.317	Do.
115	Red Zinfandel	.9966	9.04	11.35	267	280 ...do...	.917	.271	B. Dreyfus & Co.
117	Norton's Seedling, 1878	.9945	9.34	11.70	236	261 ...do...	.768	.277	Do.
112	Concord and Norton, 1879	.9986	7.58	9.54	239	223 None...	.685	.356	H. T. Dewey & Son.
66	Zinfandel, 1878	.9957	8.21	10.30	245	213 Trace...	.997	.281	Dresel & Co.
67	Zinfandel, 1879	.9963	8.83	11.08	268	188 ...do...	.825	.437	Do.
38	California Claret	.9947	9.83	12.31	256	376 ...do...	.798	.376	Do.
4	California Zinfandel	.9964	8.41	10.56	243	326 ...do...	.814	.323	George Hamlin & Co.
11	Concord Bouquet	.9930	10.58	13.24	221	237 ...do...	.903	.331	Do.
92	Concord Bouquet	.9928	9.84	12.31	218	141 ...do...	.726	.266	Do.
97	Red Wine	1.0069	9.07	11.44	390	185 ...do...	.790	.402	Do.
130	Dry Concord, 1880	.9913	9.44	11.79	166	998 Trace...	.681	.272	W. J. Green Tokay Vineyards.
180	Ives, 1874	1.0006	5.69	7.17	68	193 ...do...	.764	.420	White Elk Vineyards.
181	Red Concord	.9953	7.34	9.21	197	None ...	.838	.289	Fritz Bader.
182	Clinton	.9944	9.27	11.61	250	217 ...do...	.778	.483	Do.
183	Norton's	.9933	10.66	12.70	234	358 ...do...	.634	.323	Do.

## II.—DRY WHITE WINES.

1	California, White Hock.....	.9892	13.94	17.37	2.62	0.243	0.69	0.855	0.231	0.311
5	Missouri.....	.9908	10.68	13.34	1.45	.204	.05	.347	.227	.256
6	Kiesling.....	.9902	10.55	13.05	1.62	.181	.04	.620	.227	.314
8	California, Hock.....	.9913	16.29	12.87	1.44	.147	.09	.767	.375	.311
9	California, Muscatel.....	.9913	10.67	13.34	1.41	.190	.12	.767	.272	.396
7	Cattawa, Ohio.....	.9945	9.91	12.40	2.22	.154	.19	.632	.302	.264
13	California, Sonoma Hock.....	.9845	9.66	12.05	1.18	.190	.13	.422	.213	.167
21	Virginia, Hock, 1879.....	.9905	9.58	11.96	1.39	.166	Trace	.636	.378	.206
29	White Concord, 1875.....	.9954	8.42	10.56	2.64	.160	None	.789	.332	.365
45	Cattawa.....	.9903	10.99	13.71	2.10	.135	Trace	.833	.480	.282

## Analyses of American wines by United States Department of Agriculture in 1880—Continued.

## II.—DRY WHITE WINES.—Continued.

No.	Name.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Total residue.	Glucose.	Total ash.	Tartaric acid, as tartarate.	Fixed acid, as tartarate.	Volatile acid, as acetate.	Per cent.					
130	Catawba.	.9238	10.52	13.15	2.19	121	.....	.....	.....	.....	.470	.542	.542	.542	.470	.470
48	Brockton Catawba.	.9880	12.28	15.30	2.09	121	.....	.....	.....	.....	.385	.385	.385	.385	.323	.323
50	California Riesling Hock.	.9932	9.90	11.20	1.67	223	Trace	.....	.....	.....	.211	.846	.846	.846	.508	.508
68	California Hock.	.9935	9.07	11.35	1.92	208	.....	.....	.....	.....	.211	.223	.223	.223	.534	.534
72	Catawba.	.9938	7.75	9.70	1.34	117	.....	.....	.....	.....	.272	.528	.528	.528	.205	.205
85	Jerica, 1868.	.9919	9.64	12.05	1.91	138	Trace	.....	.....	.....	.287	.726	.726	.726	.511	.511
86	Catawba, 1868.	.9944	7.16	8.96	1.44	167	.....	.....	.....	.....	.280	.800	.800	.800	.350	.350
93	Catawba, Missouri.	.9911	8.88	11.08	1.67	129	.....	.....	.....	.....	.367	.772	.772	.772	.308	.308
106	Dry Muscat.	.9928	9.14	11.44	1.82	150	.....	.....	.....	.....	.348	.619	.619	.619	.289	.289
107	White Zinfandel.	.9911	9.02	11.26	1.47	139	.....	.....	.....	.....	.227	.590	.590	.590	.260	.260
108	Riesling.	.9918	9.64	12.95	1.72	221	.....	.....	.....	.....	.210	.696	.696	.696	.389	.389
109	Gutedel.	.9920	9.36	11.70	1.58	196	.....	.....	.....	.....	.212	.726	.726	.726	.411	.411
110	Hock.	.9939	7.73	9.70	1.73	209	.....	.....	.....	.....	.211	.723	.723	.723	.410	.410
118	"A. A." Catawba, second quality.	.9929	7.69	9.62	1.29	192	None	.....	.....	.....	.257	.710	.710	.710	.362	.362
119	"A. A. A." Catawba, first quality.	.9912	9.69	11.35	1.28	164	Trace	.....	.....	.....	.371	.772	.772	.772	.371	.371
123	Delaware.	.9940	7.08	8.80	1.51	255	None	.....	.....	.....	.226	.664	.664	.664	.250	.250
125	Mount Vernon.	.9902	7.73	9.70	2.07	202	.....	.....	.....	.....	.286	.485	.485	.485	.291	.291
128	Native wine, N. Mex.	.9894	10.55	13.15	1.80	275	Trace	.....	.....	.....	.121	.856	.856	.856	.300	.300
138	Sonoma Mission, 1878.	.9932	8.44	10.56	1.63	184	.....	.....	.....	.....	.311	.619	.619	.619	.246	.246
140	Sonoma Riesling, 1877 (1).	.9926	10.53	12.15	2.28	229	do	.....	.....	.....	.290	.695	.695	.695	.292	.292
141	Sonoma Riesling, 1879.	.9906	10.54	13.15	1.70	194	do	.....	.....	.....	.257	.575	.575	.575	.254	.254
142	Sonoma Mission, 1879.	.9935	8.30	10.38	1.67	193	do	.....	.....	.....	.317	.619	.619	.619	.242	.242
143	Sonoma Gutedel, 1879.	.9921	9.50	11.87	1.71	197	do	.....	.....	.....	.242	.589	.589	.589	.242	.242
144	Dry Muscat, 1874 (1).	.9921	9.92	12.40	1.66	256	do	.....	.....	.....	.411	.816	.816	.816	.411	.411
145	White Zinfandel, 1878.	.9928	9.56	11.96	1.96	211	do	.....	.....	.....	.294	.761	.761	.761	.393	.393
146	White Zinfandel, 1879.	.9927	8.80	11.00	1.87	182	do	.....	.....	.....	.326	.740	.740	.740	.332	.332
148	Ohio Catawba, 1879.	.9892	10.25	12.77	1.63	113	do	.....	.....	.....	.424	.728	.728	.728	.424	.424
149	Ohio Catawba, 1880.	.9935	9.28	11.61	1.80	111	do	.....	.....	.....	.125	.628	.628	.628	.125	.125
150	Helena (1), 1879.	.9954	7.80	9.78	2.06	229	do	.....	.....	.....	.250	.693	.693	.693	.271	.271
156	Riesling, 1880.	.9936	8.08	10.00	1.38	335	None	.....	.....	.....	.349	.683	.683	.683	.303	.303
159	Ida.	.9914	10.46	13.05	1.90	199	Trace	.....	.....	.....	.302	.545	.545	.545	.194	.194
161	Delaware, 1880.	.9912	9.25	11.70	1.88	255	do	.....	.....	.....	.322	.562	.562	.562	.184	.184
165	Taylor, 1880.	.9921	10.37	12.96	1.99	185	do	.....	.....	.....	.332	.732	.732	.732	.322	.322
166	Goethe, 1880.	.9962	8.53	10.73	1.83	223	do	.....	.....	.....	.314	.693	.693	.693	.301	.301
173	Catawba, 1879.	.9941	7.55	9.45	1.57	114	do	.....	.....	.....	.218	.830	.830	.830	.218	.218
175	Iona and Catawba, 1871.	.9306	9.73	12.13	1.91	103	Trace	.....	.....	.....	.561	.742	.742	.742	.561	.561
176	Iona, 1870.	.9582	12.05	15.02	1.62	do	.....	.....	.....	.....	.189	.810	.810	.810	.189	.189

Pleasant Valley Wine Company.

G. E. Ryckman.

Dressed &amp; Co.

Do.

Kelly Island Wine Co.

Julius Hincke.

Isaac Cook.

Dreyfus &amp; Co.

Do.

Wehrle, Werk &amp; Co.

Do.

C. A. Heineken.

L. &amp; H. Hinling.

Greisch &amp; Mayer.

Do.

Poesdul, Scherer &amp; Co.

HILL, -SWEEET WINE.

*Analyses of American wines, &c.—Continued.*

### III.—SWEET WINES—Continued.

*Averages and extremes of American dry wines.*

Constituents, &c.	Dry red wines.			Dry white wines.		
	Average (64 analyses).	Highest.	Lowest.	Average (51 analyses).	Highest.	Lowest.
Specific gravity.....	.9933	1.0011	.9804	.9926	1.0105	.9845
Alcohol, by weight.....	8.92	12.21	5.71	9.35	13.94	7.03
Alcohol, by volume.....	11.04	15.21	7.17	11.70	17.37	8.80
Total residue.....	2.28	3.16	1.65	1.75	2.64	1.18
Total ash.....	0.231	0.532	0.130	0.181	0.335	0.090
Glucose.....	do.	Traces.	0.450	None.	Traces.	0.300
Total acid, as tartaric.....	0.723	0.987	0.511	0.680	0.855	0.422
Fixed acid, as tartaric.....	0.360	0.646	0.226	0.313	0.561	0.121
Volatile acid, as acetic.....	0.290	0.517	0.138	0.294	0.508	0.068

A very excellent monograph on California wines has been very recently published by Dr. George Baumert,<sup>1</sup> of the University at Halle. Although the analyses were made on only eight samples, the examination was very complete, including a search for adulterations; then follows a very exhaustive discussion of the composition, together with a comparison of the samples analyzed, and similar wines made in Germany, so that the paper is a very interesting and instructive contribution to the literature of American wines. I shall have occasion to allude to it again in discussing the subject of adulterations. Other analyses of American wines published in German periodicals and alluded to by Baumert are: "Investigation of a California Wine" by A. Kayser; <sup>2</sup> "Two Analyses of California Wines, Riesling and Zinfandel," by A. Stutzer; <sup>3</sup> "Analysis of Two California Wines, Zinfandel and Gutedel Cabinet," by J. L. de Fremery.<sup>4</sup>

## METHODS OF ANALYSIS.

The methods of analysis used in the present investigation of wines can be conveniently arranged under two heads: First, those designed for the establishment of the composition of the sample examined; and, second, those employed in the search for adulterants.

The determinations embraced under the first head are: Specific gravity, alcoholic content, total solids, acidity, content of sugars, of glycerine, of tannic acid, and of ash. These are the principal and most important determinations in a wine analysis, though there are several others which are sometimes made, such as the nitrogenous content, phosphoric acid, chlorine, malic, succinic, and citric acids, &c.

In my investigation of methods for wine analysis I have drawn especially upon three sources: First, the methods in use in the Municipal Laboratory in Paris, and set forth in the report already quoted under malt liquors; second, the methods officially authorized in the health

<sup>1</sup> Landw. Versuchs-Stationen, 1886, 33, 39.

<sup>2</sup> Mitth. d. Bayr. Gewerbe-Museum zu Nürnberg, 1879, Nr. 19.

<sup>3</sup> Rep. der anal. Chem. 1882, Nr. 14.

<sup>4</sup> Ber. der Deutschen Chem. Gesell., 1885, 426.

offices in Germany, decided upon by the "Berlin Commission;" third, the methods adopted by the Bavarian chemists.

It would seem that in France and Germany, where wine analysis has been carried on for so many years, and to so large an extent, the methods in use would be as well defined and exact as is possible in such work; nevertheless, none of the methods have been accepted without a thorough trial of their accuracy, unless otherwise indicated.

The estimation of several of the constituents of wine is so similar to the same determination in beer, that it will be necessary simply to refer to the part on beer analysis, thus avoiding unnecessary repetition.

#### SPECIFIC GRAVITY.

This is taken with the piconometer, in the same manner as with beer.

#### ALCOHOL.

The estimation of the alcohol can be made in the same manner as in beer, with a few precautions. Thus, in the distillation method it is better to neutralize the sample taken with carbonate of soda, or standard alkali, and a slight addition of tannin will generally be found necessary to prevent frothing. If the indirect method is used, more accurate results will be obtained by neutralization of the sample taken by shaking up with magnesia before the specific gravity is taken. In the present work I have made use of the distillation method exclusively, weighing out the sample taken, about 50 grams, making up to about 100cc., and distilling off 50cc., which is also weighed. Tables for the calculation of alcohol content from the specific gravity have already been given (page 285).

#### EXTRACT OR TOTAL SOLIDS.

The determination of the extract in wine presents the same difficulties as with beer on account of the content of glycerine, which is greater in the former than the latter. For this reason the indirect method would seem especially applicable, but the difficulty here is that there is a larger amount of volatile acid present, which is only partially driven off with the alcohol, and the solution density of the solids of wine have not been so well established as that of malt extract, so that different tables and factors vary widely.<sup>1</sup>

For the direct estimation the French method is to evaporate 25cc. in a flat-bottomed dish, with vertical sides, and dry the residue to a constant weight in a water bath. The Berlin method is as follows:

#### EXTRACT.

Fifty cubic centimeters of wine are measured (at 15° C.) into a platinum dish (85mm. in diameter, 20mm. in height, and of 75cc. capacity, weight about 20 grams), evaporated on the water bath, and the residue heated 2½ hours at 100° C. Of wines rich in sugar, *i. e.*, containing over 5 grams sugar in 100cc., a smaller amount should be taken, so that the weight of extract shall not exceed 1, or at most 1.5 grams.

<sup>1</sup> Baumert found in his work on California wines that the tables of Schulze (Zeit Anal. Chem., 1880, 104) intended for beer extracts gave results in the indirect estimation which approximated much more closely to the results by direct estimation than figures obtained from Hager's tables (Chem. Cent., 1878, 415), which were intended especially for wine extract.

The Bavarian Chemists' Union depart from this method by drying the residue to a constant weight.

It is of great importance that chemists should adopt a uniform method of analysis, as results differ considerably by different methods. This is shown by the following results obtained from the samples analyzed by me, most of which were subjected to analysis by both methods, that is, drying to a constant weight and drying a certain length of time. In the very sweet wines, of course, such as the Angelica, Muscat, &c., containing so large a percentage of sugar, it can only be satisfactorily determined by using a small quantity, allowing it to flow well over the bottom of the dish, and drying to a constant weight as nearly as possible.

*Comparison of methods for the estimation of extract in wine.*

Serial Number.	Drying for 2½ hours at 100° C.	Drying to a constant weight.	Serial Number.	Drying for 2½ hours at 100° C.	Drying to a constant weight.
4995	2.69	2.00	5089	2.01	1.22
4996	2.82	2.26	5094	2.30	1.52
4997	2.08	1.44	5095	2.96	2.18
4998	1.57	1.16	5096	2.92	1.71
4999	2.18	1.75	5097	1.82	1.18
5000	2.24	1.74	5098	1.90	1.34
5001	4.13	3.82	5099	2.20	1.43
5002	9.78	9.30	5100	2.47	1.96
5004	9.62	9.53	5101	2.40	1.82
5005	2.51	2.09	5103	2.19	1.71
5081	1.77	1.16	5104	2.42	1.82
5083	1.80	1.16			
5084	2.12	1.39	average	2.98	2.38
5088	3.64	2.83			

I have adopted the results obtained from the estimation by drying to a constant weight, and would recommend the following procedure for the determination of extract in wine. Weigh out 10 to 50 grams of the wine (according to its content of sugar) into a flat-bottomed platinum dish, evaporate on the water bath and dry the residue at 100° C. in an air bath, until a constant weight is obtained. This may be known to be the case when by weighing the dish and contents at frequent intervals (15 minutes) the losses in weight are found to be slight and equal for the same length of time of drying.

#### ACIDITY.

The total acidity of wines may be determined by titration with  $\frac{N}{5}$  or  $\frac{N}{10}$  alkali. The end reaction may be observed by means of a drop brought into contact with delicate litmus paper, which, while not entirely satisfactory, still is probably the best method we have at hand, any means for decolorizing the wine so as to admit of the use of other indicators being likely to change the content of acid. I have found that by the use of turmeric paper in connection with the litmus, the establishment of the point of neutralization is greatly facilitated.

Very white wines, if entirely free from carbonic acid, may be titrated with phenol-phthaline as an indicator. From 10 to 25cc. of wine may conveniently be taken for titration. It is very desirable to estimate both the fixed and volatile free acids in wine, and to this end the latter should be determined directly; all methods for this estimation by difference by evaporating the wine to dryness, and titrating the residue, having been shown to be faulty. The German method is to distill off the acetic and other volatile acids in a current of steam; and I have found it a most satisfactory one, giving very coincident results when carefully carried out, the only objection being that it is a little tedious. The theory of the method is that acetic acid, which forms the greater part of the pure volatile acids, while it does not distill off at a temperature lower than 120° C., can still be distilled off at a lower temperature than its boiling point by passing through the liquid in which it is contained a current of steam, providing that the quantity of water passed through in the shape of steam is about four times as great as the quantity of the liquid in which the acid is contained. The manner of applying it is as follows:

Fifty cc. of the wine are measured into a flask of about 300cc. capacity, which is connected with a condenser by one tube which passes just below the rubber stopper, which is also perforated by another tube which reaches to the bottom of the flask and terminates there in a finely drawn out point; this tube is bent at right angles where it passes out of the flask, and connects it with a larger flask placed alongside which contains about 300cc. of water, being also provided with a safety tube. When the operation is begun, lamps are placed under the two flasks and the contents of both brought to a boil, when the flame under the flask containing the wine is lowered somewhat and the distillation so conducted that after 200cc. have distilled off the wine shall not be decreased to less than one-third or one-fourth of its original volume. The 200cc. of distillate is received in a properly graduated flask, and titrated with standard alkali, using phenol-phthaline as an indicator.

The number of cubic centimeters of normal alkali required for the titration of the volatile acids, subtracted from the number of cubic centimeters required for the neutralization of 50cc. of the original wine, gives the amount of alkali neutralized by the free *fixed* acids; the total free acids and free fixed acids are generally calculated as tartaric, the volatile as acetic acid.<sup>1</sup>

#### BITARTRATE OF POTASH.

The German Commission gives two methods, as follows:

In two stoppered flasks two samples of 20cc. of wine each are treated with 200cc. ether-aleohol (equal volumes), after adding to one flask 2-3 drops of a 20 per cent. solution of acetate of potash. The mixtures are well shaken and allowed to stand 16 to 18 hours at a low temperature (0-10° C.), the precipitate filtered off, washed with

<sup>1</sup> One cubic centimeter of normal alkali neutralizes .075 gram of tartaric, .06 gram of acetic acid.

ether-alcohol, and titrated. (The solution of acetate of potash must be neutral or acid. The addition of too much acetate of potash may cause the retention of some bitartrate in solution.) It is best on the score of safety to add to the filtrate from the estimation of the total tartaric acid a further portion of 2 drops of acetate of potash to see if a further precipitation takes place.

In special cases the following method is recommended for a control over the other:

Fifty cc. of wine are evaporated to the consistency of a thin sirup (best with the addition of sand), the residue brought into a flask by means of small washings of 96 per cent. alcohol, and with continual shaking more alcohol is gradually added, until the entire quantity of alcohol is about 100cc. The flask and contents are corked and allowed to stand 4 hours in a cool place, then filtered, and the precipitate washed with 96 per cent. alcohol; the filter paper, together with the partly flocculent, partly crystalline precipitate, is returned to the flask, treated with 30cc. warm water, titrated after cooling, and the acidity reckoned as bitartrate. The result is sometimes too high, if pectinous bodies separate out in small lumps, inclosing a small portion of free acids (this error may, however, be avoided by the addition of sand and thorough shaking). In the alcoholic filtrate the alcohol is evaporated, 0.5cc. of a 20 per cent. potassic acetate solution added, which has been acidified by a slight excess of acetic acid, and thus the formation of bitartrate from the free tartaric acid in the wine facilitated. The whole is now, like the first residue of evaporation, treated with (sand and) 96 per cent. alcohol, and carefully brought into a flask, the volume of alcohol increased to 100cc., well shaken, corked, allowed to stand in a cold place 4 hours, filtered, the precipitate washed, dissolved in warm water, and titrated, and for 1 equivalent of alkali 2 equivalents of tartaric acid are reckoned. This method for the estimation of the free tartaric acid has the advantage over the former of being free from all errors of estimation by difference.

I have preferred making the qualitative test for free tartaric acid separately from the bitartrate estimation, and for the latter I have used essentially the first method given, omitting, of course, the parallel precipitation with the addition of acetate of potash, modifying it by using only 80cc. of the alcohol-ether solution for the precipitation and allowing it to stand over night. The titration is nicely performed with phenolphthaleine and decinormal soda with white wines; 1cc. decinormal soda corresponding to .0188 grams of potassic bitartrate. In the case of dark colored wines I passed them first through a small amount of bone-black, afterwards washing the bone-black thoroughly, so as to avoid the presence of coloring matter in the tartar precipitate.

#### SACCHARINE MATTER.

For the estimation of the saccharine matter use was made of the Department method of employing Fehling's solution, already referred to under malt liquors. The Germans usually employ the gravimetric estimation, with Soxhlet's modifications, but I believe the other to be fully as accurate, and much more convenient. The wine should be evaporated about one-third to remove the alcohol, and carbonate of soda added to neutralize the acid. In the case of dark colored wines it is necessary to decolorize and clarify them by the use of subacetate of lead or bone-black. If much excess of lead is used it should be removed with sulphate of soda, and if bone-black is used the first portions filtered should be rejected. The amount of reduction is calculated as dextrose.

## ASH.

The ash may be estimated in the residue from 50 to 100cc. of the wine. If 50cc. have been used for the estimation of the extract, the same sample will serve conveniently for incineration. This operation is best performed in a muffle, and must be very carefully carried out, at as low a heat as possible. If the ash does not readily burn white, it should be treated with a little water to dissolve the alkali salts, the dish placed in such a position as to bring the water away from the undissolved ash, the water evaporated, and the incineration completed. Little difficulty is generally experienced in getting in this way a very satisfactory white ash.

## GLYCERINE.

The estimation of glycerine in wines, if it could be made with exactness, would be a very important one, as the glycerine is produced by the fermentation of the sugar, and the quantity formed is presumably fairly constant for the same amount of sugar fermented. This being the case, the quantity of glycerine in a wine should be a good index of the quantity of sugar which had undergone fermentation, and would thus show whether alcohol had been added to the wine. Unfortunately, the amount of glycerine present is so small, and its exact estimation so difficult on account of its volatile nature, that it is rather an unsafe reliance. The Germans attach considerable weight to the determination in establishing the character of a wine, using the following method :

One hundred cc. of wine (sweet wines excepted) are evaporated in a roomy, not too shallow, porcelain dish, to about 10cc., a little sand added, and milk of lime to a strong alkaline reaction, and the whole brought nearly to dryness. The residue is extracted with 50cc. of 96 per cent. alcohol on the water bath with continual stirring. The solution is poured off through a filter and the residue exhausted by treatment with small portions of alcohol. For this 50 to 150cc. are generally sufficient, so that the entire filtrate measures 100 to 200cc. The alcoholic solution is evaporated on the water bath to a sirupy consistence. (The principal part of the alcohol may be distilled off, if desired.) The residue is taken up by 10cc. of absolute alcohol, mixed in a stoppered flask with 15cc. of ether and allowed to stand until clear, when the clear liquid is poured off into a glass-stoppered weighing glass, filtering the last portions of the solution. The solution is then evaporated in the weighing glass until the residue no longer flows readily, after which it is dried one hour longer in a water jacket. After cooling, it is weighed.

In the case of sweet wines (containing over 5 grams of sugar in 100cc.), 50cc. are taken in a good-sized flask, some sand added and a sufficient quantity of powdered slacklime, and heated with frequent shaking in the water bath. After cooling, 100cc. of 96 per cent. alcohol are added, the precipitate which forms allowed to separate, the solution filtered, and the residue washed with alcohol of the same strength. The alcoholic solution is evaporated and the residue treated as above.

In regard to the performance of the official method, as given above, Dr. Barth<sup>1</sup> adds the following commentaries and cautions :

In case the residue from the first evaporation with lime becomes entirely dry it should be moistened with a little alcohol, the residue removed from the sides of the

<sup>1</sup> Die Weinanalyse, p. 17.

dish with a spatula, and the entire mass rubbed up with a pestle to a uniform pasty mass, the pestle and spatula being rinsed with a little alcohol; in heating up the alcoholic paste with lime, bumping and spouting may be avoided by careful stirring; the heating and subsequent washing out with hot alcohol is necessary, however, to dissolve out the glycerine properly. In evaporating with both the alcoholic and the ether-alcohol solution, all violent boiling of the liquid must be avoided, or mechanical losses will occur. The best way is to place the vessels containing the solutions inside of beakers filled with water on the bath. The clearing up of the ether-alcohol solution can be hastened by energetic shaking in the stoppered flask containing it. The vessel in which the ether-purified glycerine is finally weighed should have vertical walls at least 40mm. in height. The losses which are caused by the volatility of glycerine cannot be entirely avoided, but may be reduced to a minimum by a careful observance of all the directions, even those which are apparently unimportant. That the loss of glycerine is smaller by heating in a drying oven than on the open water bath has been noticed in the estimation of the extract; the choice of weighing tubes also with proportionally high, vertical walls has for its object the lessening of the possibility of losses in weight.

For the estimation of the glycerine in sweet wines the following precautions should be observed: Sufficient powdered lime must be added to the wine to convert the whole of the sugar into its lime compound. The formation of the latter takes place gradually during the heating on the water bath. The mass becomes at first dark brown (special care is necessary to prevent its foaming over the neck of the flask), but when the saturation with lime is complete it becomes somewhat clearer, and, together with the characteristic smell of the sugar-lime, a caustic odor becomes manifest.

If the residue obtained from the concentration of the alcoholic solution remains somewhat thin even after cooling, it is not necessary to repeat the treatment with lime. The purification with ether-alcohol in the way described will be all that is necessary.

The above described method for glycerine estimation is intended to obtain the glycerine in a state of purity by its separation from all the other constituents of wine, either by their volatility, by their insolubility in alcohol, or their lime combinations, or finally by their insolubility in a mixture of one volume of alcohol with  $1\frac{1}{2}$  volumes of ether. If pointed crystals appear on cooling, the presence of mannite is indicated. Since the separation of glycerine in an insoluble condition in a form or union peculiar to itself has not yet been accomplished, the extraction method must serve for its estimation, but the latter is only useful for the conclusions which are drawn from its results, when it is carried out with a strict observance of the preceding conditions.

Several methods have lately been proposed for the estimation of glycerine, and it was with the hope of some of them proving more exact and less tedious than the above that a somewhat hasty examination of these methods has been made.

Benedikt and Zsigmondy<sup>1</sup> published in 1885 a method for the estimation of glycerine by its oxidation to oxalic acid by permanganate of potash, precipitating the oxalic acid with calcium acetate, and determining it volumetrically by titration with acid. This method is also claimed by Fox and Wanklyn.<sup>2</sup> At the time of the publication of this method I made several trials of it on pure glycerine with very satisfactory results, and Allen<sup>3</sup> has confirmed the accuracy attributed to it by

<sup>1</sup> Chem. Ztg. 9, 975; Analyst 10, 206.

<sup>2</sup> Chem. News 53, 15.

<sup>3</sup> Analyst 11, 52.

Benedikt and Zsigmondy, and used it on saponified fats. It has never been applied to wine or beer, so far as I know.

Legler<sup>1</sup> has formulated a method, intended, as the author says in his paper, to supply the place of the method of the Berlin committee, and atone for its deficiencies. It depends on the oxidation of glycerine to carbonic acid by means of sulphuric acid and potassic bichromate.<sup>2</sup> The estimation of organic bodies by the oxidation of their contained carbon has been proposed and carried out by Cross and Bevan<sup>3</sup> who operate in a dry, and by Burghardt,<sup>4</sup> who operates in a wet, way. The operation was performed by Legler in a Will's carbonic acid apparatus, as follows:

The air flask contains the glycerine mixed with a saturated solution of potassic dichromate; the other contains, as usual, strong sulphuric acid. After the apparatus has been weighed, a little air is drawn out which causes some of the acid to mix with the chromate. A regular evolution of carbonic acid soon sets in, but must be assisted towards the last by gently boiling. The flask containing the sulphuric acid must be kept cool. When no more gas bubbles are formed, the apparatus is cooled by partial immersion in cold water, and the remaining carbonic acid is expelled by a current of dry air. The apparatus is now reweighed and the loss represents carbonic acid. The following equation shows the action taking place:



One part of glycerine therefore requires about 7.5 parts at  $\text{K}_2\text{Cr}_2\text{O}_7$  and 10 parts of  $\text{H}_2\text{SO}_4$ , but an excess of each is of course used.

The mode of procedure in operating on wine is as follows: The crude glycerine obtained from 100cc. of wine, after evaporation with 3cc. milk of lime and 2 grams of quartz, and extracting the mass with alcohol of 96 per cent., is, after weighing, diluted up to a definite bulk, and aliquot parts are taken for the ash and the oxidation process. A white wine, containing 8.54 per cent. alcohol and 2.07 per cent. solid matter, gave in 100cc. 1.4 grams crude glycerine, with .1278 grams ash, 25cc. of the glycerine diluted up to 50cc. yielding .725 gm.  $\text{CO}_2$  = 1.10 per cent. glycerine. A duplicate experiment gave 1.47 crude glycerine with .136 ash, .710  $\text{CO}_2$  = .99 per cent. glycerine, the average thus being 1 per cent.<sup>5</sup> and the relation between alcohol and glycerine as 100 : 11.7.

Estimation of glycerine in wine after it has been purposely added.

Three lots of 100cc. each of the same wine were mixed respectively with .125, .250, and .500 grams of glycerine, and analyzed as before. The results were as follows:

Crude glycerine.	Ash.	$\text{CO}_2$ (25cc. from 50cc.)	Glycerine in 100cc.
1.55	.1496	.80	1.115
1.75	.1400	.90	1.254
2.07	.1172	1.07	1.492

Allowing for the 1. per cent. of natural glycerine in the sample we obtain .115, .254, and .492 per cent. of glycerine.

<sup>1</sup> Rep. Anal. Chem. 6, 631; Analyst 12, 14.

<sup>2</sup> Chem. News 53, 297; also 55, 2.

<sup>3</sup> Chem. News 55, 34; see also 55, 46.

<sup>4</sup> *Ibid.*

<sup>5</sup> The average is really 1.05 per cent., which would make some difference in the figures which follow.

The same wine was also submitted to an analysis by the old process, somewhat higher results being obtained, from which Legler concludes that his method gives lower but more correct figures. He also obtained promising results from beer and sweet wines. This method was tried on some of the samples analyzed in the Chemical Division, the analytical work being done by Mr. Felix Lengfeld.

The apparatus for the estimation of the carbonic acid was a specially designed one used for most carbonic acid estimates in the laboratory, and provided for the absorption of the dried gas by soda lime. A trial was first made with pure glycerine, which gave very satisfactory results, as follows: Weight of pure glycerine taken, .3645; weight glycerine obtained, .3605; difference, .004.

When it was applied to the wines, however, it was found that the results obtained varied widely from those obtained from the same samples by the old method, and instead of the variation being constant, it was found that sometimes the results were higher, sometimes lower, than by the old method, the manner of manipulation making a very marked difference in the quantities obtained. Thus when just about sufficient lime was taken to combine with the sugar present and the mass not evaporated very closely, higher results were obtained by Legler's process than by the old method, while if a large excess of lime was added and the whole evaporated very nearly to dryness the results were lower.

Thus Nos. 4998 and 4999, treated with considerable lime and evaporated nearly to dryness, gave the following results:

Numbers.	By Legler's method.	By old method.
4998.....	.326	.436
4999.....	.542	.797

While Nos. 4995, 5000, and 5002, treated with a smaller amount of lime, and not evaporated so closely, gave:

Numbers.	By Legler's method.	By old method.
4995.....	.972	.730
5000.....	1.123	.835
5002.....	1.549	.657

From these and from several other determinations, where the differences were still more marked, it was concluded that Legler's method as it stands now cannot be relied on to give constant results with wines, for though undoubtedly an accurate method of estimating pure glycerine, it is open to the same objection that applies to the method by oxidation with permanganate, viz, that other organic bodies are also oxidized, when present, and give too high results. In the extraction of the glycerine

from wine by alcohol other organic bodies are undoubtedly taken up by it (the most of which are removed in the old method, by the purification with ether-alcohol) which make the results too high. The only way that results could be obtained as low as the old method gave was as indicated above, by long treatment in the bath with a large excess of lime, and in these cases there was undoubtedly a loss of glycerine by evaporation, as the following experiments show:

1. .3645 grams pure glycerine were mixed with a small quantity of lime, alcohol and water added, the whole evaporated nearly to dryness on the water bath, extracted with alcohol, and the glycerine determined by Legler's method in the residue from the alcoholic solution; result, glycerine .303, or a loss of .0615.

2. .3645 grams glycerine were taken and treated as above, except that a large amount of lime was added; result, .275; loss, .0895.

3. .3645 grams were taken and treated as before, except that the evaporation was carried to dryness, the dish being allowed to stand on the bath about half an hour longer than in the other experiments; result, .251; loss, .1135.

Hehner<sup>1</sup> has applied Legler's method to the estimation of glycerine in fats, with some modifications based upon the very important fact he claims to have established in his work, that "dilute solutions of glycerine (.6 glycerine to 1,000 of fluid) do not, as is commonly supposed, volatilize in concentrating the fluid, be it on the water-bath or over the naked flame."

Other methods that have been published by Benseman,<sup>2</sup> Amthor,<sup>3</sup> and Medicus,<sup>4</sup> are essentially slight modifications of the Berlin method, and can only be referred to here. Sulman and Berry,<sup>5</sup> in their article on "The Examination of Commercial Glycerine," give a very thorough résumé of various methods in use up to the date of the article for the estimation of glycerine.

The most recent method published is by Diez,<sup>6</sup> which I have not had time to investigate closely. The method is quite a novel one, and differs from the previously described methods in that it separates out the glycerine as an insoluble compound, viz., as a tribenzoate of glycerine. This is accomplished by shaking the (.5 to 1.0 per cent.) solution of glycerine with benzoyl chloride after an addition of alkali. As applied to dry wines it is described by the author as follows:<sup>7</sup> "20cc. are evaporated to a moderately dry condition after the addition of lime. The residue is exhausted with 20cc. of hot, 96 per cent., alcohol. After cooling 30cc. of water-free ether are added, and filtered after standing, the filter being washed with water-free alcohol ether (2:3). After the evaporation of the solvent the glycerine is dissolved in 10-20cc. of water,

<sup>1</sup> Analyst 12, 44.

<sup>5</sup> Analyst 11, 12 and 34.

<sup>2</sup> Chem. Zeit. 10, 554.

<sup>6</sup> Zeit. f. Physio. Chem. 11, 472.

<sup>3</sup> Rep. der. Anal. Chem., 1886, 12.

<sup>7</sup> *Ibid.*, 480.

<sup>4</sup> *Ibid.*, 1886, 1.

according to the quantity, and shaken up with 5cc. benzoyl chloride and 35cc. of 10 per cent. soda solution for 10-15 minutes without cessation and with frequent cooling. The *estergemenge* or precipitate obtained, is collected upon a filter, washed and dried for 2 to 3 hours at 100° C.; 0.385 gram of the weighed precipitate corresponds to 0.1 gram glycerine." The objection to this process seems to be that it is not applicable directly to the wine or beer, but the glycerine must be separated out in a state of comparative purity before it can be converted into the benzoate, and there is still the liability to loss during the process of purification.

The results given in the table are by the old method.

#### TANNIN.

The estimation of tannin in wines, where considerable accuracy is required, may be made by the permanganate and indigo method, which has been so much discussed and modified in chemical literature. The following approximate method is given by the Berlin Commission:

In 10cc. of wine the free acids are, if necessary, reduced by the addition of standard alkali solution to .5 grams in 100cc. Then 1cc. of a 40 per cent. solution of acetate of soda is added, and finally, drop by drop, 10 per cent. solution of chloride of iron, avoiding an excess. One drop of the chloride of iron solution suffices for the precipitation of about .05 per cent. of tannin.

For the estimation of the bulk of the precipitate test-tubes are used, which are much narrowed at the bottom, with the constricted portion graduated into tenths of a centimeter. The following table gives the approximate content of tannin from the bulk of the precipitate after standing twenty-four hours:

Precipitate after 24 hours.		Tannin in the wine.		Precipitate after 24 hours.		Tannin in the wine.	
cm.	Per cent.	cm.	Per cent.	cm.	Per cent.	cm.	Per cent.
0.1	0.003	1.0	0.033				
0.2	0.007	2.0	0.066				
0.3	0.010	3.0	0.10				
0.4	0.013	4.0	0.13				
0.5	0.017	5.0	0.17				
0.6	0.020	6.0	0.20				
0.7	0.023	9.0	0.30				
0.8	0.027	12.0	0.40				
0.9	0.030						

I can see no practical conclusions to be drawn from so indefinite a determination and have omitted it in my work, simply quoting it for the benefit of any one who might desire to make the estimation.

## ANALYSES MADE BY THE DEPARTMENT OF AGRICULTURE.

In the work on wines during the present investigation, 70 samples, purchased in the market of Washington, were examined. Inasmuch as the analyses made in 1880 included so many samples, and represented very fairly the composition of the wine sold here, it was thought inadvisable to make a complete analysis of all the samples, especially as many of them were identical in origin with those examined by Mr. Parsons. Accordingly only about one-half the samples (36) were submitted to a very careful and complete analysis, the rest being examined for adulteration only, especially preservatives. Only those samples were chosen for complete analysis which did not correspond to any of the samples analyzed in 1880. The samples are all wines of American origin, of which by far the greater bulk of the wines consumed here consists. Most of the samples are Californian, a few coming from Virginia and other States. Several of the samples had foreign labels, in imitation of some imported wine of the same general class, but in each case the dealer admitted that the wines were American.

The time and scope allowed to the work did not admit of the extension of the investigation to imported wines.

*Analyses of wines made by United States Department of Agriculture in 1887.*

*Analyses of wines made by the United States Department of Agriculture in 1867—Continued.*

Designation.	Made in—	Sweet wines.	Number of analyses.	Specific gravity.	Alcohol by weight.	Total acids as tartaric.	Fixed acids as tartaric.	Volatile acids as acetic.	Dihydroxy acids as dextrose.	Glycerine.	AsB.	Polarization in degrees, cath.
Sherry.....	California.....	1883 5001 5069	1 2	Per ct. 9929 9939	16.16 15.99	Per ct. 82 82	Per ct. 638 510	Per ct. 445 338	Per ct. 157 114	Per ct. 0.89 0.80	Per ct. 312 278	-12. -6.6
Do.....	do.....	1883 5001 5069	3 4	Per ct. 9929 10164	15.39 15.53	Per ct. 19.08 19.08	Per ct. 683 615	Per ct. 431 490	Per ct. 292 150	Per ct. 0.76 0.65	Per ct. 602 542	-29.2 -30.6
Port.....	do.....	1884 5002 5067	5 6	Per ct. 10164 10145	14.50 14.50	Per ct. 17.87 17.87	Per ct. 8.39 8.39	Per ct. 410 410	Per ct. 0.67 0.66	Per ct. 0.57 0.56	Per ct. 612 557	-29.2 -30.6
Sweet Burgundy.....	do.....	1884 5002 5067	6 7	Per ct. 10167 10167	10.98 10.98	Per ct. 13.60 13.60	Per ct. 46.5 46.5	Per ct. 417 417	Per ct. 0.18 0.18	Per ct. 113 113	Per ct. -11.8 -11.8	-8.3 -8.3
Sweet Catawba.....	do.....	1886 5004 5102	7 6	Per ct. 10167 10167	14.58 14.58	Per ct. 17.92 17.92	Per ct. 9.53 9.53	Per ct. 364 364	Per ct. 0.18 0.18	Per ct. 11.18 11.18	Per ct. 0.93 0.93	-8.3 -8.3
Do.....	California.....	1884 5003 5068	8 9	Per ct. 10161 10161	12.99 12.99	Per ct. 16.05 16.05	Per ct. 13.64 13.64	Per ct. 375 375	Per ct. 325 325	Per ct. 0.48 0.48	Per ct. 192 192	-20.2 -20.2
Sweet Muscatel.....	do.....	1884 5002 5069	10 11	Per ct. 10160 10162	15.45 15.45	Per ct. 18.00 18.00	Per ct. 16.27 16.27	Per ct. 593 593	Per ct. 283 283	Per ct. 0.57 0.57	Per ct. 11.11 11.11	-30.6 -30.6
Sweet Muscatel.....	do.....	1884 48914 50933	10 11	Per ct. 10162 10163	12.54 15.37	Per ct. 15.49 15.37	Per ct. 13.27 13.24	Per ct. 575 360	Per ct. 285 285	Per ct. 0.63 0.60	Per ct. 13.92 11.873	-30.0 -29.9
Angelica.....	do.....	1884 48914 50933	11	Per ct. 10163	15.37	Per ct. 18.90	Per ct. 13.24	Per ct. 360	Per ct. 0.60	Per ct. 0.63 0.62	Per ct. 14.200 11.873	-30.0 -29.9
Do.....	Average.....	.....	.....	1.0261	14.50	17.85	11.21	.511	.378	.104	.067	8.48 -351

*Maximum, minimum, and mean composition of the samples examined.*

Constituents.	Sixteen samples red wines.			Nine samples white wines.			Eleven samples sweet wines.		
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
Specific gravity .....	.9983	.9903	.9946	.9919	.9882	.9912	1.0511	.9929	1.0261
Alcohol by weight..... per cent.	11.98	7.78	9.66	13.35	9.01	10.44	16.16	10.98	14.50
Alcohol by volume..... do.	14.74	9.68	11.95	16.52	11.17	12.94	19.87	13.60	17.85
Extract..... do.	2.83	1.39	1.94	1.75	1.16	1.35	17.20	3.38	11.21
Total acids as tartaric..... do.	.870	.383	.611	.750	.488	.665	.683	.360	.511
Fixed acids as tartaric..... do.	.724	.113	.397	.595	.385	.498	.490	.285	.378
Volatile acids as acetic..... do.	.281	.084	.169	.187	.082	.131	.202	.025	.104
Bitartrate of potash..... do.	.133	.029	.068	.255	.039	.152	.132	.039	.067
Reducing sugars as dextrose..... do.	.508	none.	.164	.980	none.	.250	15.05	1.85	8.48
Glycerine..... do.	.893	.303	.490	.835	.365	.528	.657	.052	.260
Ash..... do.	.453	.176	.290	.260	.181	.220	.686	.118	.351

A comparison of the composition of American wines with those of other countries, as shown in these analyses, and a discussion of the points of difference or agreement would be a most interesting and instructive task. It would require considerable space, however, for a proper presentation of the subject, and must be omitted in the present investigation, as not being of so direct importance to the question of the adulteration of wines.

#### THE ADULTERATION OF WINES.

The adulteration of wine has been practiced from a very early date in those countries where the consumption is large. It has increased in amount and in the skillfulness of its practitioners until at the present day it requires for its detection all the knowledge and resources which chemical science can bring to bear upon it, and even then a large part doubtless escapes detection. It must be remembered, however, that in Europe the definition of adulteration has rather a wide scope, including the addition of substances which are simply diluents. The Paris Laboratory considers as a fraud "the addition of any substance for the purpose of gain which changes the composition of the natural wine." In Germany, on account of the northern situation of the country, it is permitted to the wine-growers in bad years, when the grapes contain a relatively high percentage of acid and a low percentage of sugar, to make use of pure sugar as an addition to the must, which addition is not considered as an offense against the adulteration laws, so long as the product is sold as "wine" simply. The amount of water added with the sugar must not be greater than twice the weight of the former, and the product must not be offered for sale as "natural wine."

By far the greater part of the adulteration carried on in the European countries consists of this addition of water (*mouillage*) and sugar (*sucrage*). Such wines result from the methods of manufacture already

described—petiotization, gallization, and chaptalization. For the detection of such wines it is necessary to establish maximum and minimum limits for the principal constituents of wines, and the relation in which these constituents stand to one another. To establish these limits is rather difficult, and requires a large series of analyses extending over many years. The constituents most relied on for the establishment of the character of a wine in judging whether it has been diluted or not are: the extract, content of free acid, and the relation between the extract and mineral matters.

In Germany the lowest limit of the extract in a natural wine is placed at 1.5 grams in 100cc., and after subtracting the amount of free fixed acids calculated as tartaric from the extract, the amount of the latter left must be not less than 1.1 grams; or after the subtraction of the total free acids as tartaric, not less than 1. gram in 100cc. In the Paris Laboratory no exact limits are set, the decision being left to the judgment of the analyst, after a careful comparison of the sample with analyses of previous similar wines done in the laboratory in past years. The sugar added is often glucose, which introduces into the wine substances more or less injurious, depending upon its character. Ordinary glucose contains usually 10 or 15 per cent. of non-fermentable substances, which serves to increase the weight of the extract, thus masking the addition of water. Its fermentation gives rise to the formation of a small quantity of amylic alcohol, which increases the intoxicating effect of such wines, and causes headache and nausea in those partaking of them.

In the detection of this substance advantage is taken of the non-fermentable character of the dextrin it contains, and of the presence of amylin, a non-fermentable, highly dextrorotatory body found in commercial starch sugar. 50cc. of wine, after driving off the alcohol by evaporation, are subjected to fermentation by the addition of a little well-washed yeast. After the fermentation is complete the liquid is clarified by means of lead or bone black, and polarized. If starch sugar has been used a strong rotation to the right will be observed, while if the wine was natural, or if the sugar used was cane sugar, there would be no rotation. The following procedure is also given by the German Commission:

Two hundred and ten cubic centimeters of the wine are evaporated to a thin syrup on the water-bath after the addition of a few drops of a 20 per cent. solution of acetate of potash. To the residue is gradually added with continual stirring 200cc. of 90 per cent. alcohol. The alcoholic solution when perfectly clear is poured off or filtered into a flask and the alcohol driven off until only about 5cc. remain. The residue is treated with about 15cc. water and a little bone-black, filtered into a graduated cylinder and washed with water until the volume measures 30cc. If, now, this liquid shows a rotation of more than +0.5, Wild, the wine contains the unfermentable matters of commercial potato sugar (amylin).

I have made no trial of these methods on American wines, and give them simply as a reference. It is well known that American starch

sugar, made from corn, is quite different in composition from the European article, which is usually made from potato starch, and I do not know that the presence of amylin has been demonstrated in the American article. Whether the latter contains much dextrin or not depends upon the character of the "glucose" used; if it is the liquid glucose, I can testify from experience that it contains a considerable percentage of dextrin; but if it is the highly converted "grape sugar" or solid glucose that is used, probably not much dextrin is introduced into the wine from it.

Fraudulent wines are frequently made from raisins or dried grapes in France, and, according to French authorities, can easily be recognized by their high percentage of reducing sugar, and left-handed polarization after fermentation.

The *plastering* of wines, which is also very extensively carried on in France, consists in adding to the wine or must a large excess of gypsum, or sulphate of lime.

The sulphuric acid of the lime salt replaces the tartaric acid which is combined with potash, and forms an acid sulphate of potash, while the tartaric acid separates out as tartrate of lime. The operation is said to give the wine a brighter color and to enhance its keeping qualities, probably by a mechanical carrying down of some of the albuminous matters. Some authorities seem to regard the addition as a pardonable one on this account, but most condemn it. It certainly introduces into the wine a salt entirely foreign to the grape and of a more objectionable nature than that which it supplants, viz, the bitartrate of potash. Both Germany and France are in accord as to the limit of sulphuric acid which can be used in a wine, requiring a wine with a content of  $\text{SO}_3$ , corresponding to over 2 grams of potassic sulphate ( $\text{K}_2\text{SO}_4$ ) per liter, to be designated as a plastered wine. This figure affords a pretty wide margin, for the average content of wines, according to most observers, is not over one-fourth of the standard, or .5 grams  $\text{K}_2\text{SO}_4$  to the liter.

The determination of the sulphuric acid can be made directly by precipitation of the wine with barium chloride, but is much more conveniently and rapidly carried out as follows:

Take 14 grams of pure, dry, crystallized barium chloride, together with 50cc. hydrochloric acid, and make up to a liter. If 10cc. of wine are used, every 1cc. of this solution required indicates a content of 1 gram  $\text{K}_2\text{SO}_4$  to the liter of wine; accordingly to several portions of wine of 10cc. each are added, respectively, 0.7, 1, 1.5, 2cc. and more if necessary, the solution heated, and allowed to stand. When cool they are filtered, and a little more barium chloride added to each test. The appearance or non-appearance of a further precipitate in the different tests will show between what limits the content of  $\text{SO}_3$  lies.

The use of starch sugar is also likely to introduce sulphates into the wine.

*Fortification* of wine consists in the addition of alcohol derived from some other source. The alcohol may be added either to the must or the

wine. It allows of better incorporation with the wine if it is added to the must before fermentation. In either case, however, it precipitates a part of the constituents originally dissolved, lowers the quantity of extract, deprives the wine of its original bouquet and flavor, and renders it more heady and intoxicating. The least objectionable addition is alcohol distilled from grapes; but the high price of the latter renders it much less likely to be used than corn spirit, which contains considerably more fusel oil. The practice of fortification prevails especially in the more southern wine-growing countries, as Portugal, Spain, and the South of France. Growers in those countries declare it to be a necessary addition in their warm climates for the preservation of the wines, as these latter contain a considerable quantity of unfermented sugar, which would soon produce the souring of the wine if the alcoholic content were not greater than can be obtained by fermentation. In France, for ordinary red wines, the addition of alcohol is decided by the relation of the alcohol to the extract (sugar deducted) exceeding sensibly the relation of 4 to 4.5. In Germany the relation of alcohol to glycerine is relied upon, the maximum proportion allowed being 100 parts by weight of alcohol to 14 of glycerine and the minimum 100 to 7. Wines going above the maximum are condemned as having suffered an addition of glycerine, those going below the minimum as being fortified with alcohol. With "sweet wines" these figures do not apply, as they are based on natural wines made in Germany. Moreover, no definite stand is taken upon the question of the fortification of the sweet wines from other countries sold in Germany. The Bavarian chemists require the content of alcohol and sugar in sweet wines used for medicinal purposes to be shown on the label, a very excellent provision, for no two samples of sweet wine can be depended upon to be of similar composition, and the physician is altogether in doubt as to what sort of a compound he is administering to his patient under the name of "port" or "sherry." Any wine with a higher percentage of alcohol than 15 per cent. by volume (12 per cent. by weight) can be safely declared to be fortified, for it has been shown that fermentation is arrested when the alcoholic content reaches about that point.

The *preservative agents* added to wine are entirely similar to those used in malt liquors. The subject of the use of salicylic acid has been so thoroughly investigated in the portion of the bulletin devoted to beer that a further treatment of the subject is unnecessary. The methods for detection and estimation already given for beer are applied equally as well to wine. The same may be said of sulphites and borax.

*Mineral additions* to wine are generally introduced accidentally, the strong acidity of the liquid making it very liable to contamination from metallic vessels, pipes, &c. Lead oxide was sometimes added to wine to counteract excessive acidity in former days, and Hassall gives cases of deaths traced to the use of such wines. Such additions belong probably to the adulterations of the past, although the possibility of

such a contamination should never be forgotten, especially if any of the symptoms of lead poisoning have been produced by a suspected sample. The search for mineral constituents in wine presents no difficulties and need not be further dwelt upon.

*Gummy substances* are sometimes added to watered wines to make up for their deficiency in extract. Gum arabic, or commercial dextrin, have been used for this purpose. The addition may be detected by the following method, taken from the German Commission: "10cc. of 95 per cent. alcohol are added to 4cc. of wine and the whole well shaken; if gums are present the liquid becomes milky and does not clear up completely even after standing several hours. The precipitate formed adheres in part to the sides of the glass, and forms lumps. In genuine wines flakes form after a short time, which subside and remain rather loose. For a more exact test the wine should be evaporated to a syrup, extracted with alcohol, and the insoluble part dissolved in water. This solution is treated with 1cc. hydrochloric acid, heated under pressure for two hours and the reducing power ascertained with Fehling's solution. With genuine wines no considerable reduction is obtained in this way." I have made no trial of this method.

The adulteration of wines by substituting for it, wholly or in part, the fermented juices of other fruits, such as cider, is a matter difficult of detection. The presence in such wines of malic acid and the absence of tartaric was formerly considered a sufficient proof of the addition, but it is found that in bad years malic acid often predominates in grape juice, and on the other hand various causes may greatly reduce the content of bitartrate of potash, or even cause it to entirely disappear. The proof of such addition by chemical means rests chiefly upon conclusions drawn from the general composition of the sample analyzed. Often the taste or odor of the residue of evaporation of the sample itself or of the distillate will give some clew to such addition. Often the recognition of free tartaric acid in such wines will condemn them as artificial, for natural wines contain a very small amount, if any, of the free acid; according to the German Commission never more than one-sixth of the total free fixed acids. Tartaric acid is often added also to wines which have been deprived of part of their normal acidity by the addition of water or sugar solutions. The qualitative determination is as follows:

To 20 or 30cc. of wine is added precipitated and finely powdered bitartrate of potash, the whole well shaken and filtered after standing an hour. To the clear solution is added 2 or 3 drops of a 20 per cent. solution of acetate of potash and the whole allowed to stand twelve hours. The shaking and standing of the solution must take place at as nearly as possible the same temperature. If at the end of this time any considerable precipitate has separated out, the quantitative estimation should be undertaken.

*Foreign coloring matters* are frequently added to red wines, either to brighten and improve the color obtained from the grapes, or, more frequently, to cover up the effects of previous dilution. These colors may

be of vegetable origin, obtained from the various vegetable dyes, or by mixing the juice of other highly colored berries or fruits with the wine; or they may be some of the numerous varieties of aniline dyes obtained from coal-tar. A few examples of the vegetable dyes said to be used may be mentioned as follows: Logwood, cochineal, elderberries, whortleberries, red cabbage, beet-root, mallow, indigo, &c.

Very elaborate and extensive schemes for the detection of these coloring matters have been devised, and chemical literature is full of articles written upon the subject, yet the positive identification of any of the vegetable coloring matters used is only very exceptionally carried out. Most of these schemes are based upon the difference in the color of the precipitates given with various reagents, and the coloring matters of the grape resemble so very closely in their behavior others of vegetable origin, and the variations in the amount of tannin present has so great an influence upon the character of the precipitate, that definite conclusions are well-nigh impossible.

The Berlin commission rejects all methods for the detection of vegetable coloring matters as not being capable of yielding positive proof, and gives only methods for the detection of coal-tar colors. The Paris Laboratory, on the other hand, gives a very elaborate scheme for the detection of both vegetable and aniline colors, designed to cover all substances likely to be used for such purposes. This scheme is based chiefly upon Gautier's and the French authorities claim that with it a chemist who is expert by long experience can detect the coloration of a wine by either vegetable or mineral foreign matters, though he may not perhaps be able in all cases to identify the particular coloring matter used. These schemes can only be referred to here, as I consider that their value is not sufficient to justify their reproduction.

The detection of aniline coloring matters can be made with tolerable certainty. The following method is essentially that given by the German commission, and originally devised, I believe, by König. Two samples of 100cc. each of wine are taken, and shaken up with about 30cc. of ether, after one has been rendered alkaline by the addition of 5cc. of ammonia. After separation has taken place, about 20cc. of the clear ethereal solution from each test are poured off (not filtered) and evaporated spontaneously in porcelain dishes in which are placed threads of pure white wool, about 5 cm. in length. With wines which are free from aniline colors, the wool, with the residue of the ammoniacal solution, remains of a perfectly white color, and the thread in the solution which was not treated with ammonia will be of a brownish color. The presence of fuchsine is readily detected, however; for out of the perfectly colorless ammoniacal ether solution a bright red color will appear as it evaporates, and becomes fixed upon the woolen thread. Those varieties of aniline dyes, which are more readily taken up by ether from acid solutions than from alkaline will be detected by the red coloring of the wool in the ether from the sample which received no addition of ammonia.

The coloring matter may also be extracted by means of amyl alcohol, which color will be discharged from the solvent by ammonia if the aniline dye used be of an acid nature, in which case the amyl alcohol will dissolve little coloring matter from the wine in presence of ammonia.

The *diseases* of wine may be considered in the light of an adulteration, as it is a fraud to offer wines for sale as pure wines which have undergone a change which alters their composition and renders them unfit for use. The researches of Pasteur on fermentation have shown that nearly all of the diseases of wine are due to the development in them of microscopical vegetable growths, whose germs are carried in the air. Each disease has its own special organism peculiar to itself, which may be detected by the microscope. These different organisms produce the souring, molding, bittering, cloudiness, blackening, &c., of wine. The best wines are said to be the most subject to these alterations; every year large quantities of the finest wines of Burgundy are spoiled by the disease called bittering (*l'amer*).

In wines that have become entirely unfit for use through the development of one of these diseases the fact is rendered sufficiently evident by the senses, especially to an expert taster. To detect the first beginning of such alteration, however, is more readily done by means of the microscope in the hands of an expert.

#### EXAMINATION FOR ADULTERATION OF THE WINES ANALYZED BY THE DEPARTMENT.

In the absence of any well-defined national standard as to what shall constitute a pure wine in the United States, or definitions and limitations as to the nature of the liquids which can lawfully be sold as such, I have had recourse to the well-defined and carefully worded laws of Germany and France which deal with the adulteration of wines, some of which, together with the accepted methods for the detection of adulteration, as agreed upon by chemists of prominence in those countries, I have collected together and inserted at the close of the Bulletin, under the heading of Appendix B.

The only State law I have been able to find which deals specifically with wine is a recent enactment in New York, which is also given in full in Appendix C.

The nature and extent of the different kinds of adulteration as shown by the samples examined may conveniently be taken up in the same order as was pursued in treating of the methods for detecting them, and of these the first is the dilution or watering of wine.

#### THE DILUTION OR WATERING OF WINE.

It would seem natural that in American wines, which can be produced so cheaply and in such great abundance, this adulteration, which is such a favorite one with the manufacturers of the costly wines of Bordeaux, Burgundy, &c., would be very rare. The fraud is so simple, however, so easy of execution, and so difficult of detection, that it will

probably always be a favorite one with unscrupulous dealers. It must be remembered, also, that with many American producers, whatever article they produce, more attention is paid to its quantity than its quality. Wine-growers are not the only persons who practice this method, as it can be done also by merchants and retailers, although in the latter case it is much more easy of detection. That which might be called scientific dilution, by means of the processes already described (petiotization, &c.), is much more difficult of detection than the simple attenuation of the wine by the retailer. So little official supervision has been exercised over the wines sold in this country that certainly the fear of detection has not operated very largely as a preventive of this, or in fact any other adulteration.

In Dr. Baumert's work, which has already been alluded to,<sup>1</sup> and to which I shall have occasion to refer frequently as constituting, small as it is, the only published investigation of American wines for adulteration, none of the samples fell below the German standard in percentage of extract (1.5 grams per 100cc.). On the other hand, nearly all the white wines which I submitted to a complete analysis fell below this standard, and two of the red wines. A large number of the samples analyzed by Mr. Parsons also fell below it. That this limit is not placed at too high a figure, for California wines at least, seems evident from a study of the table I have prepared of Professor Hilgard's analyses of pure wines, from which it appears that only one series of analyses gave a minimum below it, while the averages are far above it. It might possibly be too low for Virginia wines, but the majority of those that fell below it were of California origin. The New York law specifies (§ 2) that "such pure wines shall contain at least 75 per centum of pure grape or other undried fruit juice." Just how a chemist, in the absence of legal definitions of what shall constitute a "pure grape or other undried fruit juice," is to decide upon the question of such adulteration by the above law is difficult to indicate.<sup>2</sup>

The samples which would be considered as watered according to the German standard are as follows: Serial Nos. 5084, 5099, 4997, 4998, 5081, 5083, 5089, 5097, and 5098.

#### PLASTERING.

American wines would seem to be quite free from this form of adulteration. Baumert found no undue excess of sulphates in the samples he examined, but refers to a sample analyzed by Stutzer, which contained in 100cc. .141 gram  $\text{SO}_3$ . In my seventy samples I found none which exceeded the generally adopted standard of .092 gram  $\text{SO}_3$  to 100cc., or 2 grams  $\text{K}_2\text{SO}_4$  to the liter, and only three, Nos. 5100, 5107, and 5115, which contained  $\text{SO}_3$ , corresponding to over 1 gram  $\text{K}_2\text{SO}_4$  per liter.

<sup>1</sup> Page 339.

<sup>2</sup> It will be seen by the above that cider would be considered as "wine" under the construction of the law, as it is the fermented juice of "other undried fruit."

## FORTIFICATION.

It is evident that the German standard of 100 parts of alcohol by weight to 7. of glycerine, which is relied upon as a means of detecting the addition of alcohol, cannot be applied to American wines. Only three of the samples would pass muster by it, and it seems hardly possible that the practice of adding alcohol could be so widespread as would be thus indicated.

Below is given the number of grams of glycerine for 100 grams of alcohol obtained in the samples (exclusive of the sweet wines):

4995.....	7.1	5104.....	3.5
4996.....	5.6	4997.....	3.7
5005.....	9.0	4998.....	4.7
5084.....	6.7	4999.....	7.3
5088.....	4.5	5000.....	6.3
5094.....	5.7	5081.....	3.7
5095.....	3.4	5083.....	4.3
5096.....	4.3	5089.....	6.5
5099.....	5.0	5097.....	5.6
5100.....	3.1	5098.....	3.4
5101.....	6.3		
5103.....	3.7	Average.....	5.1

Baumert obtained very similar results; out of thirteen analyses (including sweet wines) made or collected by him only four contained a larger proportion of glycerine to alcohol than 7 to 100. Unfortunately no determinations of the glycerine were made in the pure wines analyzed by Hilgard; so no light is thrown on this point by them. The only possible way of deciding it, together with other questions relating to the composition of American wines, would be by the analysis of a large number of wines known to be pure. In the absence of such evidence, it would be useless to attempt to pass judgment on the above samples as to whether they had been fortified with alcohol or not.

The New York law allows of an addition to wine of "pure distilled spirits to preserve it" not to exceed 8 per cent. of its volume, which, supposing the wine to contain originally 10 per cent. by volume, would give a wine containing at least 17 per cent. by volume, or about the highest amount of alcohol which could be formed in a fermented liquor.

The sweet wines are, of course, well known to be fortified; they will be treated of more fully further on.

## PRESERVATIVES.

Especial attention has been given in the present investigation to the use of improper preserving agents in fermented drinks. It was thought that such agents were much used; so a considerable number of samples were purchased, and the examination for preservatives, as well as for other adulterations whose detection did not require a complete analysis of the wine, was extended to all. The results show the practice to be even more extensive than was supposed.

The following table shows in what samples salicylic acid and sulphites were detected. In the case of the sulphites, where a "trace" is

indicated, there was not sufficient to justify the assertion that a sulphite or sulphurous acid had been added directly to the wine; in such cases it probably came from insufficient cleansing of the casks. Where it is indicated as "present," however, there was sufficient indication of its having been added to the wine.

*Examination of wines for preservatives.*

Designation.	Made in—	Serial number.	Salicylic acid.	Sulphites.
Champagne.....	New York.....	4960	None.....	None.
Do.....	do.....	4961	do.....	Do.
Do.....	Ohio.....	4962	do.....	Do.
Do.....		4963	do.....	Present.
Burgundy.....		4961	do.....	None.
Virginia seedling.....		4963	do.....	Do.
Catawba.....	New York.....	4960	Present.....	Do.
Sweet Scuppernong.....	North Carolina.....	4967	None.....	Do.
Charbono.....	California.....	4968	do.....	Do.
Lenoir.....	do.....	4969	do.....	Do.
St. Macaire.....	do.....	4970	do.....	Do.
Angelica.....	do.....	4991	do.....	Do.
Burgundy.....	do.....	4993	do.....	Do.
Claret.....	do.....	4996	do.....	Trace.
Moselle.....	do.....	4997	do.....	None.
Riesling, Gray.....	do.....	4998	Present.....	Present.
Riesling, Johannisberg.....	do.....	4999	do.....	None.
Sauterne.....	do.....	5000	None.....	Present.
Sherry.....	do.....	5001	Present.....	None.
Sweet Burgundy.....	do.....	5002	None.....	Do.
Sweet Muscatel.....	do.....	5003	do.....	Do.
Tokay.....	do.....	5004	do.....	Do.
Zinfandel.....	do.....	5005	do.....	Do.
Catawba.....		5081	do.....	Trace.
California Hock.....		5082	do.....	Present.
California Riesling.....		5083	do.....	Trace.
Burgundy.....		5084	do.....	None.
Zinfandel.....		5085	do.....	Do.
St.-Julien Claret.....		5086	do.....	Trace.
Sweet Catawba.....		5087	do.....	None.
St.-Julien Claret.....		5088	Present.....	Trace.
Riesling.....		5089	None.....	Present.
Sherry.....	California.....	5091	do.....	None.
Port.....	do.....	5091	do.....	Do.
Muscatel.....	do.....	5092	do.....	Do.
Angelica.....	do.....	5093	Present.....	Do.
Claret.....	Virginia.....	5094	None.....	Do.
Zinfandel.....	California.....	5095	do.....	Do.
Claret.....	Virginia.....	5096	do.....	Trace.
Riesling.....	California.....	5097	do.....	Present.
California Berger.....	do.....	5098	do.....	None.
Claret.....	Virginia.....	5099	do.....	Do.
Do.....	New Jersey.....	5100	Present.....	Do.
Do.....	Virginia.....	5101	None.....	Do.
Catawba.....		5102	Present.....	Do.
Claret.....	California.....	5103	None.....	Do.
Do.....	do.....	5104	do.....	Do.
Do.....	Virginia.....	5105	do.....	Do.
Sauterne.....		5106	do.....	Present.
Hock.....		5107	Present.....	Do.
California Beaune.....		5108	do.....	None.
Sweet Catawba.....		5109	do.....	Trace.
California Gutedel.....		5110	do.....	Present.
Claret.....	Virginia.....	5111	None.....	Do.
California Zinfandel.....		5112	do.....	Trace.
California Port.....		5113	do.....	None.
Sonoma Port.....		5114	Present.....	Do.
California Angelica.....		5115	do.....	Do.
Frontignan.....		5116	None.....	Do.
Old Palo Sherry.....	California.....	5117	do.....	Do.
California Zinfandel.....	do.....	5118	Present.....	Trace.
Gutedel Hock.....	California.....	5119	None.....	Do.
Berger Hock.....	do.....	5120	do.....	Present.
California Burgundy.....	do.....	5121	Present.....	Trace.
California Madelira.....	do.....	5123	do.....	None.
California Port.....	do.....	5124	None.....	Do.
California Tokay.....	do.....	5125	Present.....	Do.
California Frontignan.....	do.....	5126	None.....	Do.
California Angelica.....	do.....	5127	do.....	Do.
California Berger Hock.....	do.....	5128	do.....	Present.

From an examination of this table it will be seen that of the seventy samples examined, *eighteen*, or over one-fourth, had received an addition of salicylic acid, and *thirteen* had been preserved by the use of sulphurous acid, either as such, or in the shape of a sulphite. In two cases both agents had been used. One of the samples which contained salicylic acid and also one containing sulphites were among the samples exhibited at the meeting of the National Viticultural Convention last year in Washington.

The question of the propriety of the use of preservatives has been very fully discussed in previous pages, and will not be further enlarged upon here.

Baumert found no salicylic acid in the samples examined by him, and only traces of sulphurous acid.

The examination of the samples for boracic acid gave such peculiar results that I hesitate about pronouncing positively upon them until I shall have had opportunity to investigate the matter more closely. The test with turmeric paper gave slight traces present in all but *two* of the thirty-six samples which were submitted to a complete analysis. With only a very few, however, could any test be obtained with the alcohol flame. Baumert obtained the same test in every one of the samples he examined. He seeks to account for this by the fact that plants have been known to assimilate boracic acid from the soil, and knowing that in some parts of California the soil contains considerable quantities of borax, he offers it as an explanation that it was taken up by the vine from the soil. This explanation seems hardly tenable, and in view of the fact that some of the samples I examined came from various parts of the country other than California, must be thrown aside as insufficient. It is a singular fact that both of the samples in which I failed to get the test, Nos. 5087 and 5102 were Catawba wines.

I can offer no explanation of the matter except the suspicion, which I hope to be able to investigate, that the trouble lies with the test.

#### ARTIFICIAL WINES.

No test for free tartaric acid was obtained with any of the samples, which would seem to indicate that none of the wines were artificial wines, in the make-up of which free tartaric acid is very apt to figure.

#### COLORING MATTERS.

All of the samples of *red* wines, about forty, were submitted to a search for aniline coloring matters, which resulted in the demonstration that one sample out of the forty, No. 4996, was colored with an aniline dye-stuff, probably fuchsine.

Baumert found one of his eight samples to be colored artificially with an aniline dye.

No search was made for foreign *vegetable* coloring matters.

## SWEET WINES.

It would seem advisable to call attention to the very variable character of these wines as shown by the analyses. Considering the extensive use that is made of such wines for medicinal purposes, it is greatly to be desired that some standard should be required for their composition, or that their relative content of alcohol and sugar, at least, should be stated on the label, as is required by the Bavarian authorities. Among Mr. Parsons' samples will be found a "Sweet Muscate" which contains as high as 31 per cent of sugar, and a "California port" which contains nearly 21 per cent. of alcohol by weight. The analyses of the sweet wines made by myself furnish a still poorer showing, for the low figures obtained for glycerine show that very little pure grape juice enters into their composition. Take the different samples of Angelica and Muscatel wines, for instance; these varieties are almost peculiar to California; they are made from a very sweet grape, of strong flavor. Comparatively few analyses have been made of them, but Baumert had among his samples two Muscatel wines and one Angelica. These contained the following percentages of glycerine: Muscat (H), .883; muscat (W), 1.424; Angelica, .698.

Compare these numbers with the percentage of glycerine contained in the following: 5003, Muscatel, .102; 5092, Muscatel, .104; 4994, Angelica, .140; 5093, Angelica, .052.

These results are so disproportionately low as to give strong ground to the suspicion that but very little of the pure juice of these strong flavored grapes entered into the composition of the samples I examined, but that they were chiefly composed of alcohol, sugar, and water. It would be an easy matter to imitate the strongly marked flavor of the grapes by means of artificial essences.

California wine-growers claim that they have in their very pure grape brandy an excellent and unobjectionable source of alcohol for the fortification of sweet wines, but certainly the samples above partake more of the nature of a liqueur, than of a natural wine.

The following table gives a classification of the wines analyzed in the Paris Municipal Laboratory during the years 1881 and 1882, showing the proportion which was declared adulterated, and the relative amount of the different varieties of adulteration as shown by the samples analyzed. It must be remembered that these analyses were made on suspected samples, and do not by any means represent an average of the quality of the wines sold in Paris.

	1881.	1882.
Number of samples analyzed .....	3,361	5,150
Good .....	357	898
Passable .....	1,093	1,590
Diseases of wine (acid, bitter, moldy, &c.) .....	per cent.	
Fortified .....	do.	6.51 5.24
Wines not plastered or plastered less than 1 gram .....	do.	9.55 7.32
Wines plastered between 1 and 2 grams .....	do.	24.45 25.47
Wines plastered above 2 grams .....	do.	52.53 41.49
Watered .....	do.	23.02 33.04
Sugared and petiotized .....	do.	41.12 29.15
Artificially colored .....	do.	3.30 6.62
Salicylated .....	do.	15.65 7.66
Salted .....	do.	4.73 5.00
Deplastered .....	do.	0.18 0.08
		0.11

A wine may be counted several times in this table; for instance, if it is at the same time watered, fortified, too much plastered, and artificially colored. The total of the percentages, therefore, adds up to over 100.

The samples analyzed by me may be tabulated as follows:

Total number analyzed .....	70
Plastered between 1 and 2 grams .....	per cent.. 4.38
Watered (according to European standards) .....	do... 12.85
Artificially colored .....	do... 1.43
Salicylated .....	do... 25.71
Sulphured .....	do... 18.57

The percentages are of the total number analyzed; not of the number adulterated, as in the French wines.



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PART III.

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CIDERS.

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## CIDER.

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Cider is the fermented juice of the apple. It is an article of very general use, especially in those parts of the country where fruit-growing is carried on. Statistics of the amount produced or consumed are rather difficult to obtain, and I am unable to present any definite statement on the subject. It is quite a favorite article of home production, nearly every farmer in regions where apples are grown making his barrel of cider for use through the winter; but a large amount also finds its way into the city markets, finding ready purchasers among people who still retain their taste for the drink, acquired during a childhood on the "old farm." A considerable quantity is also consumed in the shape of bottled cider, "champagne cider," "sparkling cider," and similar substitutes for, or imitations of, champagne wine, large quantities of this clarified cider being produced in some parts of the country, notably New Jersey. Most of the cheaper kinds of champagne (American champagne) are made in this way.

In England and France considerable quantities of cider find their way into the markets, though it is there, as here, largely an article of home consumption. Certain parts of those countries are famous for the quality of their ciders, notably Normandy, in France, and Herefordshire and Devonshire, in England. France produced, in 1883, 23,493,000 hectoliters (620,211,200 gallons) of cider, or over one-half of the quantity of wine produced, and three times as much as the total quantity of malt liquors.

### MANUFACTURE OF CIDER.

In the numerous sections of the United States where apples are grown in large quantities the manufacture of cider furnishes a most important means for the utilization of such fruit as is unfit for marketing, either from being too small or sour, or too thoroughly ripened, or bruised from handling. The conversion of these into cider, and perhaps of the cider into vinegar, is a very important branch of apple growing, and the cider press is an indispensable adjunct to a large orchard. Within the last ten years the manufacture of cider has been greatly aided by improvements, both in the machinery for crushing the fruit and in the presses for extracting the juice, but it is doubtful if the methods of treatment of the juice after extraction have undergone a corresponding development. The methods of fermentation and preserving—operations that are so carefully performed in the manufacture of other fermented

liquors—are exceedingly crude, as I can testify from personal experience. The juice, whether containing a relatively large percentage of sugar or not, is drawn into barrels and left to itself, probably exposed to a hot sun and to all the changes of temperature incident to the autumn season; and when the season is over or the cider is in danger of freezing, it is transferred to the cellar in the same barrels in which it was originally run, without any attempt at cleansing it of sediment, or filtering or racking, and when any attempt at improving its keeping quality is made it is by adding some antiseptic instead of freeing it from the matters which conduce to improper fermentations, or so conducting the process as to produce a liquor which can properly be called the "wine of apples." It seems remarkable that with these methods so palatable a drink is produced, a fact which only shows what might be done if a little care and scientific knowledge were applied to the treatment of the juice. There is a great difference between the practice here and in other countries in regard to the treatment of the juice. Here the greater part of the cider produced is treated as indicated above, and is sold to the consumer in the fall or winter of the same year it is produced, without any treatment whatever, except perhaps the addition of a dose of mustard seeds or sulphite of lime or salicylic acid, to arrest or retard the fermentation. This addition serves only to stop the fermentation for a while, probably through the winter, and in the spring whatever has not been consumed has to be thrown away or turned into vinegar. In England and France the juice is treated according to the sweetness of the apples from which it is made, very sweet juice requiring a low temperature for its fermentation in order that the operation shall not be too rapid. The juice is run into barrels or large vats, which are kept in a barn or cellar where the temperature is more or less constant, and the fermentation allowed to go on until a "chapeau" or head of scum forms on top, containing many of the impurities of the juice. The clear liquid is then "racked off" from between the impurities which have risen to the top and those that have fallen to the bottom. The casks into which it is received are scrupulously clean and are filled nearly full and transferred to a cooler cellar, where a second slow fermentation takes place. The racking-off process may be repeated if necessary, or the juice may be filtered from the first fermentation. Cider fermented and properly racked in this way will keep indefinitely at a low temperature, especially if bottled. For bottling, it generally undergoes the operation called "fining," by the addition of isinglass, which removes most of the albuminous constituents which are so inimical to its proper preservation. Cider made in this way will be much richer in alcohol, and contain much less acetic acid than when its first fermentation is allowed to take place at a high temperature and in a rapid, tumultuous manner. It is a true apple wine and will keep indefinitely. The cider of Devonshire has been kept twenty or thirty years.

## COMPOSITION OF CIDER.

The amount of chemical work done on cider is not nearly so great as has been done on wine. In fact the published analyses of cider are very few and are confined almost entirely to other countries. I have not been able to find a single published analysis of American cider.

The following analysis of Alsatian cider was made by Boussingault, and dates back to 1863:

	Grams per liter.
Alcohol .....	69.95
Sugar .....	15.40
Glycerino and succinic acid .....	2.58
Carbonic acid .....	0.27
Malic acid .....	7.74
Acetic acid .....	Traces.
Gummy matters .....	1.41
Potash .....	1.55
Lime, chlorine, &c .....	0.20
Nitrogenous matter .....	0.12
Water .....	980.78

Rousseau has published the mean of twenty analyses of Brittany cider, but his results are so low that it is thought by French authorities that his samples had been watered:

Alcohol, per cent. by volume .....	2.5
Extract grams per liter .....	19.3
Sugar .....	2.5
Total ash .....	1.52
Ash soluble in water .....	1.17

The following are analyses of pure ciders from different parts of France, made in the Paris Laboratory; the figures are in grams per liter:

	Pure cider, 1877, Bois-Guillaume.	Pure cider, made in 1876, Yvetot.	Old cider.	Pure cider, 1878, Yvetot.	Pure cider, "gross cidre," 1880, near Bayeux.	Cider No. 2.	Cider, 1st class.	Pure cider, "gross cidre," 1880, Bagnoux.
Alcohol, in weight per liter....	47.40	41.08	37.92	34.76	23.70	7.90	25.30	19.75
Extract dried at 100° C .....	57.60	30.90	20.90	61.30	53.20	69.70	81.20	63.80
Extract dried <i>in vacuo</i> .....	60.10	37.60	27.00	72.70	60.80	82.00	92.60	75.00
Total ash .....	3.50	2.50	2.50	3.00	2.60	2.54	2.30	2.80
<i>Analysis of the ash.</i>								
Phosphates insoluble in water .....	0.38	.....	0.25	0.30	0.45	0.62	0.17	20.55
Carbonate of potash .....	2.23	.....	1.40	2.00	1.80	1.51	.....	.....
Other alkaline salts .....	0.89	.....	0.85	0.70	0.35	0.41	.....	.....
Reducing sugar .....	20.00	7.50	4.40	3.70	16.50	36.00	39.00	25.00
Acidity expressed as $H_2SO_4$ .....	3.60	4.07	5.36	4.54	3.23	2.68	.....	2.08
Acidity of the cider dried <i>in vacuo</i> .....	2.50	2.40	2.59	2.31	2.68	1.11	.....	1.48

Of these samples the first four had undergone a good fermentation. They furnish the following average composition for the principal constituents:

Alcohol, per cent. by volume .....	5.2
Extract per liter, at 100° C .....	41.18
Sugar .....	8.90
Ash .....	2.87

The other four samples were partially unfermented, or sweet, ciders. Their average composition was as follows:

Alcohol, per cent. by volume .....	1.70
Extract per liter, at 100° C .....	66.98
Ash .....	2.56

From these means the Municipal Laboratory deduces the following as a type of composition for pure ciders:

Alcohol, per cent. by volume .....	5.66
Extract per liter, at 100° C .....	30.00
Ash .....	2.80

Recent analyses of pure ciders, from different parts of France, published by M. G. Lechartier,<sup>1</sup> have shown great variations from this type, and show the necessity for the examination of large numbers of samples from various parts of the country for the establishment of a proper standard of analysis.

#### ANALYSES OF SAMPLES BY THE DEPARTMENT OF AGRICULTURE.

Various conditions rendered it impossible to extend the investigation of ciders to a very large number of samples. It is hoped that an opportunity for a more extended study will present itself in the future.

The samples for the investigation were purchased in the city in the same way as the samples of wine and beer.

#### METHODS OF ANALYSIS.

The different determinations to be performed in an analysis of cider can be made by the same methods as are used in the analysis of beer and wine. These have been already sufficiently discussed, and a brief résumé showing the amount taken for determination, &c., in the samples analyzed will be all that is necessary.

The specific gravity was taken with the pycnometer.

The alcohol was determined in the distillate from 100cc., after previous neutralization of the free acid.

The total solids were determined by drying to a constant weight, 10 grams in the case of the sweet ciders, 25 to 50 grams in the more completely fermented samples.

The acidity was determined in 25 to 50cc. by titration with decinormal soda, and calculated as malic.

The sugar was determined with Fehling's solution, as indicated under beer.

The ash was determined by incinerating the residue from 50cc. in a muffle.

The albuminoids were determined by evaporating 25cc. in a *schälchen* and burning with soda lime.

The carbonic acid was determined in the bottled ciders by the method given under beer.

The polarization given is that of the normal cider in degrees of the cane-sugar scale. It was taken on a Laurent polariscope.

*Analyses of ciders by United States Department of Agriculture.*

Designation.	Serial No.	No. of analysis.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Total solids.	Ferments, as malic.	Sugar, as dextrose.	Ash.	Albuminoids.	Carbonic acid.	Polarization cane-sugar scale.
<i>Well fermented ciders.</i>												
Draft cider ("extra dry")	4830	1	1.0132	P. ct.	4.18	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	19.5
Bottled cider, known to be pure	4832	2	1.0003	8.09	10.05	1.88	.456	.....	.279	.063	trace	-7.0
Bottled cider	4833	3	1.0007	6.28	7.83	1.80	.376	.....	.310	.044	.....	-6.1
Bottled "extra dry russet" cider	4834	4	1.0264	4.48	5.61	5.52	.339	.....	.393	.031	.....	-35.2
"Champagne cider," bottled	4835	5	1.0223	4.08	5.10	5.02	.567	.....	.310	.050	.161	-23.4
"Champagne cider," bottled	4836	6	1.0143	5.45	6.79	3.69	.361	.....	.415	.038	.120	-20.4
"Sparkling cider," bottled	4927	7	1.0306	3.63	4.54	5.92	.113	.....	.506	.....	(?)	-33.8
Average	.....	.....	1.0154	5.17	6.45	3.88	.402	.....	.377	.044	.....	.....
<i>"Sweet" or incompletely fermented ciders.</i>												
Draft cider	4829	1	1.0537	0.65	0.81	9.34	.565	.....	.315	.069	.....	-41.6
"Sweet" cider	4831	2	1.0516	0.61	0.77	9.59	.302	.....	.270	.063	.....	-34.2
"Sweet" cider (draft)	4837	3	1.0567	0.20	0.25	9.53	.375	.....	.283	.075	.....	-48.4
Do.	4838	4	1.0303	3.46	4.33	3.84	.302	.....	.374	.044	.....	-24.2
Do.	4839	5	1.0552	0.55	0.67	9.75	.409	.....	.336	.031	.....	-48.5
Do.	4841	6	1.0355	2.96	3.71	6.98	.478	.....	.348	.069	.....	-39.1
Average	.....	.....	1.0455	1.40	1.76	8.17	.405	.....	.321	.059	.....	.....

<sup>1</sup>A circumstance arising after the samples had been thrown away seemed to throw considerable doubt upon the determinations of sugar, which were made by an assistant, and the entire set had to be thrown out.

<sup>2</sup>Determinations of the carbonic acid in three different bottles gave the following results: .728, .654, .482.

ADULTERATION OF CIDER.

Cider is very little subject to adulteration according to most of the authorities on foods. Even Haessall, who generally enumerates under each article of food a list of every conceivable adulteration that has ever been found or supposed to have been used in such food, only speaks of the addition of water, of burnt sugar as a coloring matter, and of the use of antacids for the correction of the acidity of spoiled cider. On the other hand, in France, where, as we have seen, it is

very largely consumed, its adulteration is by no means uncommon, although principally confined to its watering, together with additions for the purpose of covering up such attenuation, such as foreign coloring matters. In the Paris Municipal Laboratory out of 63 samples examined in 1881, 39 were pronounced "bad," among which were 26 artificially colored; in 1882, 59 samples were examined, of which 30 were declared "bad," of which 7 samples were artificially colored; 2 samples contained salicylic acid. The following is considered there as a minimum limit for the composition of a pure cider, and any sample which falls below it in any constituent is considered as watered:

Alcohol, per cent. by volume .....	3.
Extract in grams, per liter.....	18.
Ash.....	1.7

This is for a completely fermented cider; in sweet ciders the content of sugar should exceed the limit sufficiently to make up for the deficiency of alcohol, to which it should be calculated.

#### EXAMINATION OF THE SAMPLES FOR ADULTERATION.

The investigation of the samples was undertaken with the full expectation of finding a considerable number preserved with antiseptics. This supposition failed to be confirmed, however, for no salicylic acid was found, and in but one case was any test obtained for sulphites. None of the samples fell below the standard proposed by the French chemists, given above, and no metallic or other adulteration was discovered.

The single exception, however, No. 4927, was an embodiment in itself of nearly all the adulterations which have been enumerated as possible in cider. It was handsomely put up in neatly capped bottles, and of a clear, bright color. Its tremendous "head" of gas when uncorked gave rise at once to the suspicion that it had received some addition to produce an artificial pressure of gas, for pure cider does not contain sufficient sugar to produce very much after-fermentation, any more than pure wine. The low content of free acid, together with the large amount of ash and very variable content of carbonic acid in different bottles, established the fact that bicarbonate of soda had been added, probably a varying quantity to each bottle, while the dose of sulphites added was so large that a bottle has stood open in the laboratory all through the summer without souring.

To describe in detail the methods for the detection of the adulterations of cider would be simply a repetition of what has been previously given in connection with either beer or wine; the search for preservatives is precisely the same, and the detection of the addition of antacids has been fully treated of under beer. No search was made for artificial coloring matters, as it seems very improbable that any such should be used in this country, and no evidence of any dilution was found.

## APPENDIX A.

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Since the portion of this bulletin relating to malt liquors was written, a bill has been introduced into the British Parliament dealing with the question of the use of substitutes for hops and malt in beer brewing; the text of this bill is as follows:<sup>1</sup>

A BILL for better securing the purity of beer. (A. D. 1887.)

Whereas it is expedient, with a view to enable the public to distinguish between beer brewed from hops and malt from barley and beer composed of other ingredients, to amend the law relating to the sale of beer:

Be it therefore enacted by the Queen's Most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:

(*Short title.*) 1. This act may be cited as the Pure Beer Act, 1887.

(*Declaration of ingredients on selling beer.*) 2. Every person who sells or exposes for sale, by wholesale or retail, any beer brewed from or containing any ingredients other than hops and malt from barley shall keep conspicuously posted at the bar or other place where such beer is sold or exposed for sale, a legible notice stating what other ingredients are contained in such beer. Any person who sells or exposes for sale any such beer as aforesaid without complying with the above enactment shall be liable to a fine not exceeding in the case of the first offense *five pounds*, and in the case of the second or any subsequent offense *twenty pounds*. Any fine incurred under this section may be recovered summarily by any informer, and one-half of the fine shall in every case be paid to the informer.

(*Definition of beer.*) 3. In this act the term "beer" includes beer (other than black or spruce beer), ale, and porter.

(*Commencement of act.*) 4. This act shall come into operation on the *first day of January, one thousand eight hundred and eighty-eight*.

A BILL for better securing the purity of beer. (A. D. 1887.)

Whereas it is expedient, with a view to the better protection of the public from adulteration of beer, to amend the law relating to the sale of beer:

Be it therefore enacted by the Queen's Most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:

(*Short title.*) 1. This act may be cited as the Beer Adulteration Act, 1887.

(*Penalty on selling beer containing other ingredients than hops and malt without giving notice.*) 2. Every person who sells or exposes for sale by wholesale or retail any beer brewed from or containing any ingredients other than hops and malt from barley, shall keep conspicuously posted at the bar, or other place where such beer is sold or exposed for sale, a legible notice stating that other ingredients are contained in such beer.

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<sup>1</sup> Analyst, 1887, 99.

Any person who sells or exposes for sale any such beer as aforesaid, without complying with the above enactment, shall be liable to a fine not exceeding in the case of the first offense *forty shillings*, and in the case of the second or any subsequent offense *ten pounds*.

Any fine incurred under this section may be recovered summarily by any informer, and one-half of the fine shall in every case be paid to the informer.

(*Definition of beer.*) 3. In this act the term "beer" includes beer (other than black or spruce beer), ale, and porter.

(*Extent of act.*) 4. This act shall not extend to Ireland.

(*Commencement of act.*) 5. This act shall come into operation on the first day of January, one thousand eight hundred and eighty-eight.

This bill naturally excited considerable interest among food analysts, to whom would fall the duty of determining the question that would arise, under its enforcement, as to whether beers had been made from hop or malt substitutes, and a circular was sent out by the president of the Society of Public Analysts to the members of the society drawing their attention to the bill and asking them to report to the secretary any information they might have on the points whether the substances used as substitutes for hops could be detected and identified with certainty by chemical analysis, and what opinion, if any, they might have as to the effect on health of habitual small doses of such hop substitutes.<sup>1</sup>

This circular called forth a few responses, among them the following paper by Mr. Allen:<sup>2</sup>

#### AN IMPROVED METHOD OF DETECTING QUASSIA AND CERTAIN OTHER HOP SUBSTITUTES IN BEER.

[Read at the meeting in May, 1887.]

Hitherto the detection of hop substitutes in beer has had for English analysts little more than an academic interest. There has been no definition of beer, nor standard of strength or quality, and hence the brewer has been free to employ any hop substitute that could fairly be regarded as non-injurious. Now, however, that there are two bills before Parliament, both of which aim at rendering the employment of hop substitutes illegal, unless duly acknowledged, the question has acquired considerable practical importance. If the bills in question ever become law, it will devolve on the public analyst to certify to the presence or absence of hop substitutes, but, as was recently pointed out by the chancellor of the exchequer to a deputation which waited on him, it would be of no use to pass an act forbidding the unacknowledged use of hop substitutes unless it was possible to detect infringement of the law. Hence it has become important for public analysts to see how far the more important hop substitutes can be definitely detected in beer, or at any rate distinguished from hops.

The problem in question is by no means a simple one. Beer itself is a highly complex and variable product, and some of the normal constituents add to the difficulty of detecting hop substitutes. The problem is further complicated by the possible presence of several substitutes simultaneously, together with actual hops. Then the bitter principles to which the hop substitutes owe their employment have in some cases been very imperfectly studied, and belong to a class of bodies by no means distinguished for strong chemical affinities or for characteristic reactions. In fact, the

<sup>1</sup>Analyst 1887, p. 79.

<sup>2</sup>Ibid., p. 107.

most general and striking property of the majority of hop substitutes is the intensity of their bitter taste, a character which materially increases the difficulty of detecting them, owing to the very moderate amount employed to give the beer the desired flavor. There is one other complication of practical importance, and that is the very considerable quantity of beer usually recommended to be used for the analysis. Thus, if two liters be used for the main examination, as is recommended by Dragendorff, at least twice that quantity should be submitted to the analyst, and hence twelve liters would have to be purchased. The necessity of purchasing so large a quantity as two and a half gallons of each beer would almost certainly render the act abortive.

In looking into the question, therefore, I have aimed at reducing the amount of beer employed as low as possible.

In the last number of the *Analyst* I gave a list of references to articles in English periodicals, &c., connected with detection of hop substitutes in beer. These I have carefully studied, and they have formed the foundation of the following statements and proposals:

Dragendorff, who has published a very elaborate method for the recognition of a large number of possible hop substitutes (*Jour. Chem. Soc.*, XXVII, 818; XLII, 103), operates on two liters of beer, precipitates the concentrated liquid with basic acetate of lead, further concentrates the filtrate and adds a large volume (1,000 to 1,200cc.) of absolute alcohol. The spirit is subsequently driven off, and the various principles extracted by a systematic employment of immiscible solvents.

Wittstein (*Jour. Chem. Soc.*, XXIX, 767) operates in a similar manner, but works on one liter, and omits the treatment with acetate of lead, as indeed did Dragendorff in his older method. In the method described by me in my *Commercial Organic Analysis* (Vol. I, page 97), based on a process of Ender's, one liter of beer is employed, the concentrated liquid is treated with alcohol, the filtrate precipitated with ether, and the filtered liquid evaporated, the residue redissolved in alcohol, treated with water, and the solution precipitated with acetate of lead.

Several of the writers on the subject state that on precipitating a beer with basic acetate of lead the hop-bitter is wholly precipitated, and hence, if the concentrated filtrate still have a bitter taste, the presence of some hop substitute is certain.<sup>1</sup>

This difference seemed to me so important that I have very carefully investigated it, and find it perfectly in accordance with the fact. I prefer, however, to employ *neutral* acetate of lead instead of the basic or ammoniaeal acetate, as the latter reagents are liable to precipitate certain bitter principles not removed by the first. On the other hand, the hop-bitter is very perfectly precipitated by neutral lead acetate, and this statement is equally true of an infusion of hops purposely prepared and of a beer in which hops is the sole bitter used; but I have some reason to think that basic acetate of lead is liable to effect a less perfect separation of the hop-bitter.

In attempting to improve these processes I have endeavored to dispense with the use of alcohol, and yet separate the bitter principles from the sugar and other bodies which disguise the bitter taste and interfere with application of chemical tests, and find that a very satisfactory product for further treatment can be obtained by the following simple method:

One liter of the beer is evaporated to about 300cc. and is then precipitated, while hot, with a solution of neutral acetate of lead. The precipitate is filtered off, the filtrate allowed to become cool, and any further precipitate is also removed. The excess of lead is then removed from the filtrate by sulphuretted hydrogen, and the liquid fil-

<sup>1</sup> This distinction between the bitter principles of hops and hop substitutes is referred to in the work entitled *Chemistry Applied to the Arts and Manufactures*, edited by Chas. Vincent, and often described as the New Edition of Muspratt's Chemistry. The process is also described in Wynter Blyth's work on the *Analysis of Food*, and forms an essential part of Ender's method of detecting bitter substances, so that there is a very general consensus of opinion as to the value of the test.

tered and further concentrated to about 150cc. I prefer sulphuretted hydrogen to sulphuric acid or a sulphate, as the lead sulphide seems to carry down a notable quantity of coloring matter.

The treatment with lead acetate removes all tannin, phosphates, &c., and *the hop-resin and lupulin are also completely precipitated, while all or nearly all hop substitutes remain in solution.*

The next object is to separate the sugar, dextrin, and mineral constituents of the beer as perfectly as possible from the active principles of the various hop substitutes. Instead of precipitating the carbo-hydrates, &c., by excess of strong alcohol, I prefer to remove the bitters from the aqueous liquid itself by agitation with suitable immiscible solvents.

The object being to extract as many active principles as possible in the simplest possible way, leaving their mutual separation and recognition for further consideration, I employ chloroform as having the most general solvent action. It separates with tolerable ease from the aqueous liquid, and should be employed as long as it leaves a notably bitter residue on evaporation. In many of my experiments I relied on the traces of lactic and other acids naturally present in beer to produce the requisite degree of acidity, but I am now of opinion that the addition of a little dilute sulphuric acid is advantageous, if not actually necessary, in some cases. The extraction with chloroform being complete, ether should next be used, the treatment being repeated as long as any notably bitter principle is extracted. Finally, the aqueous liquid is rendered alkaline with ammonia, and agitated with chloroform or ether-chloroform, to extract any alkaloids.

The following arrangement shows the behavior of the more important bitter principles when the aqueous liquid is agitated in succession with chloroform, ether, and ammonia, and ether-chloroform. I have personally verified the behavior of the substances to the names of which asterisks are attached.

(1) Extracted by chloroform from acid solutions:

Absinthin (wormwood).

\* Anthemin (chamomiles).

Colchicine (*colchicum*), imperfectly.

\* Colocynthin (*cocoynth*, or bitter apple), imperfectly.

\* Calumbin, and probably some berberine (*calumba*), bright yellow and highly fluorescent.

\* Gentipierin (gentian), very imperfectly.

\* Pieric acid (artificial), yellow, imperfectly.

Picrotoxin (*coccus indicus*), with difficulty.

\* Quassia (*quassia wood*).

(2) Subsequently extracted by ether from acid solutions:

\* Chiratin (*chiretta*).

\* Colocynthin (*cocoynth*, or bitter apple).

\* Gentipierin (gentian).

Pieric acid, yellow.

Picrotoxin (*coccus indicus*).

(3) Subsequently extracted by ether-chloroform from alkaline solutions:

\* Berberine (*calumba root*).

Colchicine (*colchicum*).

By evaporating off the solvent, warming the residue with a little alcohol, and then adding water, solutions are obtained which will be bitter if any of the above substances be present. A very small quantity of the substance is required for this test; indeed, the use of too large an amount must be carefully avoided or the sense of taste will be found to be wholly paralyzed for the remainder of the day at any rate.

It will be seen that chloroform or ether extracts from acidulated aqueous liquids almost the whole of the above bitters. The subsequent treatment with ether-chloroform in alkaline solution is usually unnecessary, as the principles of calumba and

colchicum are in part extracted by acid chloroform. Seeing that the bitter principles of hops are entirely precipitated by neutral acetate of lead, *the presence of some hop substitute is absolutely certain if the chloroform or ether residue has a marked bitter taste.* This can be ascertained in the course of a few hours by the simple method above indicated, and half a pint of the beer is amply sufficient for the purpose.

The presence of a hop substitute being proved by the marked bitter taste of the chloroform or ether extract it will of course be very desirable to ascertain its nature, and in some important cases this can fortunately be effected very satisfactorily. In others we may expect future investigation to afford the necessary assistance.

I have made special endeavors to ascertain the possibility of definitely recognizing quassia, which is one of the most important of the hop substitutes actually employed.

For this purpose I prepared quassia in a moderately pure state by exhausting quassia wood with hot water and treating the decoction with acetate of lead and chloroform, in the manner recommended for beer. The quassia was obtained with some difficulty in a distinctly crystalline state and otherwise it presented a close general resemblance to the description of it given by other observers. The following characters and tests were specially verified :

Quassia is intensely and persistently bitter, sparingly soluble in cold water, more readily in hot, and easily soluble in alcohol. Its best solvent is chloroform, which extracts it readily from acidulated solutions.

An aqueous solution of quassia does not reduce Fehling's solution, or ammonio-nitrate of silver. The solid substance gives no coloration (or merely yellow) when treated with strong sulphuric acid or with nitric acid of 1.25 specific gravity, nor is any color produced on warming. These four negative reactions are important, for *picrotoxin* reduces Fehling's solution and gives an orange-red color with sulphuric acid; *gentipicrin* and *menyanthine* reduce ammonio-nitrate of silver, and the former gives a red color and the latter a yellowish brown, changing to violet red when warmed with sulphuric acid, and other bitters mostly give more or less characteristic reactions.

A solution of quassia gives a white precipitate with tannin. The reaction is used by Christensen, Oliveri, and others to-isolate quassia from its solutions, and by Enders to separate it from picrotoxin. In my hands the reaction has not proved satisfactory. The liquid is very difficult to filter and the filtrate still retains an intensely bitter taste, showing that the precipitation is very incomplete. As an analytical method the reaction is useless, but it is of some value as a qualitative test.<sup>1</sup> The test must be made in a cold solution.

Quassia gives a brown coloration with ferric chloride. The reaction is best observed by moistening a quassia residue in porcelain with a few drops of a weak alcoholic solution of ferric chloride, and applying a gentle heat. A fine mahogany-brown coloration is produced.

The most delicate and characteristic test for quassia is based on an observation of Christensen. On treating quassia with bromine a derivative is obtained, which is stated to be more bitter than the original substance. On adding caustic soda the bitter taste is said to be destroyed, but a product of a fine yellow color is obtained. I am unable to confirm the destruction of the bitter taste, at least entirely, but the coloration is marked and characteristic.

The following is the best way of applying the test: The substance to be tested for quassia is dissolved in a little chloroform, or if a liquid is agitated with chloroform, and the aqueous layer separated. The chloroformic solution is then treated with bromine water until the yellow color remains after agitation, showing that the bromine has been used in slight excess. The aqueous liquid is then removed (or if small in volume may be neglected), and the chloroform agitated with ammonia. This produces immediate destruction of the color due to the bromine, and if quassia be ab-

<sup>1</sup> Possibly more complete precipitation of quassia by tannin could be effected in an alcoholic solution.

sent both the chloroform and ammoniacal liquid will be colorless. In presence of quassia the ammonia will be colored a bright yellow.

The chloroform residues from camomiles, calumba, colocynth, coeculus, and chiretta do not give any similar reactions with bromine and ammonia. The *ether* residue from chiretta gives a straw-yellow coloration, gradually changing to a dull purplish brown, but the fact that no such reaction yielded by the chloroform solution of the drug renders confusion with quassia impossible. Pieric acid yields a solution in chloroform which is but slightly colored compared with the deep-yellow liquid produced on subsequent agitation with ammonia; but if its presence be suspected it can be readily and completely removed by agitating the chloroformic solution with soda or ammonia, and separating the alkaline liquid before employing bromine.

With a view of ascertaining how far the foregoing reactions of quassia were likely to be of service in practice I added to one liter of a mild beer, which had been previously proved to yield no bitter principle to chloroform after treatment with acetate of lead, sufficient infusion of quassia to make a perceptible difference in the flavor. The liquid was concentrated, precipitated with neutral lead acetate, the filtrate treated with sulphuretted hydrogen, and the refiltered liquid further concentrated and agitated with chloroform. On evaporating the chloroform a residue was obtained which had an intensely bitter taste, and yielded a solution which gave a white precipitate with tannin, but did not reduce ammonio-nitrate of silver. The residue gave no color on warming with concentrated sulphuric acid, but gave a well-developed mahogany-brown color with ferrie chloride. By the bromine and ammonia test it gave a strong yellow coloration.

The amount of residue obtained would have sufficed to obtain all these reactions several times, so that it may be considered established that *quassia can be detected with certainty and facility in a moderate quantity of beer containing it.*

The employment of chiretta as a hop substitute has been repeatedly recorded by previous observers, but no tests are given for it by Dragendorff or others who have worked on the subject. I found it in quantity in two hop substitutes I recently examined, and suspect its presence in a third. The active principle (chiratin,  $C_{25}H_{48}O_{15}$ ) is intensely bitter, sparingly soluble in cold water, rather more so in hot, and is readily dissolved by alcohol and ether, the latter solvent readily removing it from its aqueous solution. On the other hand, chloroform removes but little bitter principle from an aqueous infusion of chiretta. Chiratin is a neutral substance, decomposed by dilute acids into ophelio acid and chiratogenin. It does not reduce Fehling's solution, gives a copious precipitate with tannin, and is not precipitated by neutral lead acetate. The reaction of the other residue from infusion of chiretta with bromine and ammonia has already been described.

It is evident that our knowledge of the chemistry of the vegetable bitters available as hop substitutes is very incomplete, and it is only by its further study we can hope to fully solve the problem of their detection in beer. But I believe we can already distinguish with certainty and facility between "hops" and "not hops," and that ought to suffice in many cases. When we examine butter we are content to define the admixture as "foreign fat," and we make no attempt to specify the exact nature or origin of the foreign fat employed. I submit that we are fully able to take a similar position with respect to hops and hop substitutes.

The following are some of the remarks made in the discussion of this paper:<sup>2</sup>

Dr. Adams said that he worked on the question of hop substitutes some ten or twelve years ago, and he found no difficulty in distinguishing between the bitter of hop and the substitutes used for it. The method he found most useful was the precipitation by subacetate of lead, and there was no difficulty at all with the ordinary bitters such as quassia, calumba, gentian, chiretta, and wormwood, all remaining in

<sup>2</sup>*Ibid.*, p. 114.

solution, whilst the bitter of hop, and also the bitter of the camomile, which behaves like the hop, goes down and leaves the solution bitterless. He had specimens of all the hop substitutes in use in England at that time, and without a single exception the solution remained bitter after treatment with the subacetate of lead.

After having separated the filtrate and evaporated it down, there was no difficulty in detecting the bitter; but he did not think there was any possibility of distinguishing between the individual bitters, considering the minute quantity present, and one could only positively say there was another bitter present besides that of the hop.

Dr. Muter said that, with regard to the question of the detection of bitters in beer, he would say at once that he did not quite share the president's views as to there being no difficulty in detecting and identifying them. It was a subject that he had worked at in former years in connection with his book on *materia medica*. He had made numerous experiments himself, and he had repeated many of the published experiments and he could prognosticate that, as the president went along, he would meet with several published reactions which were partially incorrect and perfectly misleading. Although a few bitters would be found to be comparatively easy of detection—quassia, for instance, was a bitter which spoke very much for itself—many of them were, to a great extent, involved in difficulty and obscurity at the present time. There was no branch of chemistry that so much undesirable matter had been published about as that relating to *materia medica*, and many of the older researches were incorrect. No doubt one cause of this was that nowadays they had much better analytical appliances than the men who made these experiments. He did not for a moment mean to say that they were now cleverer than past observers, but, for instance, they could not wash lead and other similar intractable precipitates in former days as one could now do by the aid of the filter pump, and the color reactions for more than one proximate principle, which were given in books, were really not due to the principle at all, but to the traces of reagents and other matters that remained with them owing to the imperfect washing, which was almost certain to occur before the days of filter pumps. He was afraid that until the president and others who might take the subject up had had time to work it out by the aid of modern appliances it would be going too far to say they could swear positively that a sample contained no hop substitute.

Mr. Allen said he thought they could tell whether it contained hop or a substitute for hops.

Dr. Muter then said in that case how would they get on in cross-examination, seeing that they could not name the substitute? He did not remember how he first became acquainted with the lead process, but he believed that he could put his hands on it now. It must be quite thirteen or fourteen years ago since it was first published. Immediately it was brought out he had made experiments upon it, working on large quantities. Since that there had really been no other process that he knew of. He himself had used a process very similar to that mentioned, viz, precipitation with subacetate of lead, siphoning off the clear liquor after settlement, removing the excess of lead from this liquid, concentrating and tasting, and then extracting with immiscible solvents.

Now came a difficulty which shook his faith in his powers as an analyst as regards hop substitutes. He had always believed in the process—from practicing upon beer with various added bitters—until some time ago he got a beer which he was privately assured by the maker to have no bitter other than hops. This sample he put through the process and he got a bitter out of that beer with chloroform after lead. He worked on a fairly large quantity, but the process here showed bitters other than hop, although he was assured that the sample represented as pure a beer as could possibly be obtained. Another difficulty was the quantity they might have to work on. Supposing an inspector brought them one-half or one-third of a pint, where was the process they could use? He had put bitters in the beer and worked on such quantities and failed to find them. In a case he knew of some time ago there was some

difficulty about some strychnine that was put in beer; he was aware of the very small quantity that had been put in, because the chemist who had been stupid enough to lend himself to such a transaction had informed him of it. He made up some beer and divided it into two portions, tried for extraneous bitters in one portion by the regular beer way and there was not a sign of it; he then tried the other portion with a special toxicological process for strychnine and found it. He had, even then, to use eight ounces of the beer for this purpose, to get a really satisfactory ordinary reaction. In the present state of chemical knowledge it would not be, in his opinion safe to say they could detect any amount of added bitters to beer, however small, and go to the length of naming those bitters on the quantity they would have usually brought to them by an inspector. With a gallon of beer and an unlimited fee covering many days' work they might, however, be able to do something satisfactory towards it.

Mr. Allen, in reply: If he had an insufficient quantity of any sample, he certified that the quantity was insufficient for him to form an opinion.

He understood Mr. Norton to consider it of great importance that they should be able to distinguish between "hops" on the one side and "not hops" on the other; this, he believed, could be done with certainty, ease, and on a very moderate quantity of beer. As to the identification of the various hop substitutes he did not profess to be able to distinguish all, but he thought he could already positively recognize calumba, quassia, colocynth, and some other bitters, including picric acid and picrotoxin, and if the matter became important he believed in a year or two public analysts would have devised methods for the detection of the other bitters, just as they had conquered other analytical difficulties when the occasion arose.

From the above it would appear that the lead method, which I employed, is considered by the English analysts as capable of deciding whether substitutes for hops have been used.

No action seems to have been taken as yet on the question of malt substitutes.

## APPENDIX B.

As frequent references have been made in the body of the Bulletin to methods of analyses and manner of judging adapted by European chemist for beer and wine, I have thought it proper to give some of these methods complete. They represent the conclusions of chemists who have devoted their lives to the study of this branch of work, and may very properly be taken as a guide by those of us who have occasion to follow the same line of work in this country, where beer and wine analysis has as yet had little application.

The translation has been made as literal as possible.

The following are the methods adopted by the Berlin Commission:<sup>1</sup>

A commission of experts, appointed in the year 1834 by the chancellor of the Empire, to which was intrusted the establishment of uniform methods for the chemical investigation of wine, adopted the following resolutions, which were made public by the Prussian minister for commerce and trade by a decree of the 12th August, 1834, which provides that they shall be rigidly adhered to in public institutions for the examination of food-stuffs, and are recommended to the representatives of like private concerns:

### RESOLUTIONS OF THE COMMISSION FOR ESTABLISHING UNIFORM METHODS FOR THE ANALYSIS OF WINES.

Since, in consequence of improper manner of taking, keeping, and sending in of samples of wine for investigation by the authorities, a decomposition or change in the latter often occurs, the commission considers it advisable to give the following instructions:

#### INSTRUCTIONS FOR SAMPLING, PRESERVING, AND SENDING IN OF SAMPLES OF WINE FOR EXAMINATION BY THE AUTHORITIES.

(1) Of each sample, at least one bottle ( $\frac{1}{4}$  liter), as well filled as possible, must be taken.

(2) The bottles and corks used must be perfectly clean; the best are new bottles and corks. Pitchers or opaque bottles in which the presence of impurities cannot be seen are not to be used.

(3) Each bottle shall be provided with a label, gummed (not tied) on, upon which shall be given the index number of the sample corresponding to a description of it.

(4) The samples are to be sent to the chemical laboratory as soon as possible to avoid any chance of alteration which, under some circumstances, can take place in a very short time. If they are, for some special reason, retained in any other place for any length of time, the bottles are to be placed in a cellar and kept lying on their sides.

<sup>1</sup> Das Gesetz betreffend den Verkehr mit Nahrungsmittel, u. s. w., p. 184.

(5) If in samples of wine taken from any business concern adulteration is shown, a bottle of the water is to be taken which was presumably used in the adulteration.

(6) It is advisable, in many cases necessary, that, together with the wine, a copy of these resolutions be sent to the chemist.

*A.—Analytical methods.*

*Specific gravity.*—In this determination use is to be made of a piconometer, or a Westphal balance controlled by a piconometer. Temperature 15° C.

*Alcohol.*—The alcohol is estimated in 50–100cc. of the wine by the distillation method. The amount of alcohol is to be given in the following way: In 100cc. wine at 15° C. are contained  $n$  grams alcohol. For the calculation the tables of Baumhauer or Heben are used.

(The amounts of all the other constituents are also to be given in this way; in 100cc. wine at 15° C. are contained  $n$  grams.)

*Extract.*—For this estimation 50cc. of wine, measured out at 15° C., are evaporated on the water bath in a platinum dish (85mm. in diameter, 20mm. in height, and 75cc. capacity, weight about 20 grams), and the residue heated for two and one-half hours in a water jacket. Of wines rich in sugar (that is, wines containing over 0.5 grams of sugar in 100cc.) a smaller quantity, with corresponding dilution, is taken so that 1 or at the most 1.5 grams extract are weighed.

*Glycerine.*—One hundred cubic centimeters of wine (for sweet wines see below) are evaporated in a roomy, not too shallow, porcelain dish to about 10cc., a little sand added, and milk of lime to a strong alkaline reaction, and the whole brought nearly to dryness. The residue is extracted with 50cc. of 96 per cent. alcohol on the water bath, with frequent stirring. The solution is poured off through a filter, and the residue exhausted by treatment with small quantities of alcohol. For this 50 to 100cc. are generally sufficient, so that the entire filtrate measures 100–200cc. The alcoholic solution is evaporated on the water bath to a sirupy consistency. (The principal part of the alcohol may be distilled off if desired.) The residue is taken up by 10cc. of absolute alcohol, mixed in a stoppered flask with 15cc. of ether and allowed to stand until clear, when the clear liquid is poured off into a glass stoppered weighing glass, filtering the last portions of the solution. The solution is then evaporated in the weighing glass until the residue no longer flows readily, after which it is dried an hour longer in a water jacket. After cooling it is weighed.

In the case of sweet wines (over 0.5 grams sugar in 100cc.) 50cc. are taken in a good-sized flask, some sand added, and a sufficient quantity of powdered slack-lime, and heated with frequent shaking in the water bath. After cooling, 100cc. of 96 per cent. alcohol are added, the precipitate which forms allowed to separate, the solution filtered, and the residue washed with alcohol of the same strength. The alcoholic solution is evaporated and the residue treated as above.

*Free acids* (total quality of the acid reacting constituents of the wine).—These are to be estimated with a sufficiently dilute normal solution of alkali (at least one-third normal alkali) in 10 to 20cc. wine. If one-tenth normal alkali is used at least 10cc. of wine should be taken for titration; if one-third normal, 20cc. of wine. The drop method (*Tupsel methode*), with delicate reagent paper, is recommended for the establishment of the neutral point. Any considerable quantities of carbonic acid in the wine are to be previously removed by shaking. These "free acids" are to be reckoned and reported as tartaric acid ( $C_4H_6O_6$ ).

*Volatile acids.*—These are to be estimated by distillation in a current of steam, and not indirectly, and reported as acetic acid ( $C_2H_4O_2$ ). The amount of the "fixed acids" is found by subtracting from the amount of "free acids" found, the amount of tartaric acid corresponding to the "volatile acids" found.

*Bitartrate of potash and free tartaric acid.*—(a) Qualitative detection of free tartaric acid: 20–30cc. of the wine are treated with precipitated and finely powdered bitartrate of potash, shaken repeatedly, filtered off after an hour, and 2–3 drops of a 20 per cent. solution of acetate of potash added to the clear filtrate, and the solution

allowed to stand twelve hours. The shaking and standing of the solution must take place at as nearly as possible the same temperature. If any considerable precipitate forms during this time free tartaric acid is present, and the estimation of it and of the bitartrate of potash may be necessary.

(b) Quantitative estimation of the bitartrate of potash and free tartaric acid: In two stoppered flasks two samples of 20cc. of wine each are treated with 200cc. ether-alcohol (equal volumes), after adding to the one flask 2-3 drops of a 20 per cent. solution of acetate of potash. The mixtures are well shaken, and allowed to stand 16-18 hours at a low temperature (0-10° C.), the precipitate filtered off, washed with ether-alcohol, and titrated. (The solution of acetate of potash must be neutral or acid. The addition of too much acetate may cause the retention of some bitartrate in solution.) It is best on the score of safety to add to the filtrate from the estimation of the total tartaric acid a further portion of 2 drops of acetate of potash to see if a further precipitation takes place.

In special cases the following procedure of Nessler and Barth may be used as a control:

Fifty cubic centimeters of wine are evaporated to the consistency of a thin syrup (best with the addition of quartz sand), the residue brought into a flask by means of small washings of 93 per cent. alcohol, and with continual shaking more alcohol is gradually added, until the entire quantity of alcohol is about 100cc. The flask and contents are corked and allowed to stand four hours in a cool place, then filtered, and the precipitate washed with 93 per cent. alcohol; the filter paper, together with the partly flocculent, partly crystalline, precipitate, is returned to the flask, treated with 30cc. warm water, titrated after cooling, and the acidity reckoned as bitartrate. The result is sometimes too high if pectinous bodies separate out in small lumps, inclosing a small portion of free acids.

In the alcoholic filtrate the alcohol is evaporated, 0.5cc. of a 20 per cent. potassic acetate solution added, which has been acidified by a slight excess of acetic acid, and thus the formation of bitartrate from the free tartaric acid in the wine facilitated. The whole is now, like the first residue of evaporation, treated with (sand and) 96 per cent. alcohol, and carefully brought into a flask, the volume of alcohol increased to 100cc., well shaken, corked, allowed to stand in a cold place four hours, filtered, the precipitate washed, dissolved in warm water, titrated, and for one equivalent of alkali two equivalents of tartaric acid are reckoned.

This method for the estimation of the free tartaric acid has the advantage over the former of being free from all errors of estimation by difference. The presence of considerable quantities of sulphates impairs the accuracy of the method.

*Malic acid, succinic acid, citric acid.*—Methods for the separation and estimation of these acids cannot be recommenced at the present time.

*Salicylic acid.*—For the detection of this, 100cc. of wine are repeatedly shaken out with chloroform, the latter evaporated and the aqueous solution of the residue tested with very dilute solution of ferric chloride. For the approximately quantitative determination it is sufficient to weigh the chloroform residue, after it has been again recrystallized from chloroform.

*Coloring matter.*—Red wines are always to be tested for coal tar colors. Conclusions in regard to the presence of other foreign coloring matters drawn from the color of precipitates and other color reactions are only exceptionally to be regarded as safe. In the search for coal tar colors the shaking out of 100cc. of the wine with ether before and after its neutralization with ammonia is recommended. The etherial solutions are to be tested separately.

*Tannin.*—In case a quantitative determination of tannin (or tannin and coloring matter) appears necessary the permanganate method of Nenbauer is to be employed. As a rule the following estimation of the amount of tannin will suffice: The free acids are neutralized to within 0.5 grams in 100cc. with standard alkali, if necessary. Then 1cc. of 40 per cent. sodic acetate solution is added, and drop by drop a 10 per cent. solution of ferric chloride, avoiding an excess. One drop of ferric chloride is

sufficient for the precipitation of 0.05 per cent. of tannin. (New wines are deprived of the carbonic acid held in solution by repeated shaking.)

*Sugar.*—The sugar should be determined after the addition of carbonate of soda by means of Fehling's solution, using dilute solutions, and, in wines rich in sugar (*i. e.* wines containing over .5 gram in 100cc.), with observance of Soxhlet's modifications, and calculated as grape sugar. Highly colored wines are to be decolorized with animal charcoal if their content of sugar is low, and with acetate of lead and sodium carbonate if it is high.

If the polarization indicates the presence of cane sugar (compare under polarization) the estimation is to be repeated in the manner indicated after the inversion (heating with hydrochloric acid) of the solution. From the difference the cane sugar can be calculated.

*Polarization.*—(1) With white wines: 60cc. of wine are treated with 3cc. acetate of lead solution in a graduated cylinder, and the precipitate filtered off. To 30cc. of the filtrate is added 1.5cc. of a saturated solution of sodic carbonate, filtered again, and the filtrate polarized. This gives a dilution of 10:11 which must be allowed for.

(2) With red wines: 60cc. wines are treated with 6cc. acetate of lead, and to 30cc. of the filtrate 3cc. of the saturated solution of sodic carbonate added, filtered again, and polarized. In this way a dilution of 5:6 is obtained.

The above conditions are so arranged (with white and red wines) that the last filtrate suffices to fill the 220mm. tube of the Wild polaristrobometer of which the capacity is about 28cc.

In place of the acetate of lead very small quantities of animal charcoal can be used. In this case an addition of sodic carbonate is not necessary, nor is the volume of the wine altered. If a portion of the undiluted wine 220mm. long shows a higher right-handed rotation than  $0.3^\circ$  Wild, the following procedure is necessary:

Two hundred and ten cubic centimeters of the wine are evaporated on the water bath to a thin sirup, after the addition of a few drops of a 20 per cent. solution of acetate of potash. To the residue is added gradually, with continual stirring, 200cc. of 90 per cent. alcohol. The alcoholic solution, when perfectly clear, is poured off or filtered into a flask, and the alcohol distilled or evaporated off down to about 5cc. The residue is treated with about 15cc. water and a little bone black, filtered into a graduated cylinder, and washed with water until the filtrate measures 30cc. If this shows on polarization a rotation of more than  $+0.5^\circ$  Wild, the wine contains the unfermentable matter of commercial potato sugar (amylin). If in the estimation of the sugar by Fehling's solution more than 0.3 grams sugar in 100cc. was found, the original right-rotation caused by the amylin may be diminished by the left-rotating sugar; the above precipitation with alcohol is in this case to be undertaken, even when the right-rotation is less than  $0.3^\circ$  Wild. The sugar is, however, first fermented by the addition of pure yeast. With very considerable content in (Fehling's solution) reducing sugar and proportionally small left-rotation, the diminishing of the left-rotation may be brought about by cane sugar or dextrin or amylin. For the detection of the first the wine is inverted by heating with hydrochloric acid (to 50cc. wine, 5cc. dilute hydrochloric acid of specific gravity 1.10), and again polarized. If the left-rotation has increased, the presence of cane sugar is demonstrated. The presence of dextrin is shown as given in the section on "gum." In case cane sugar is present well washed yeast, as pure as possible, should be added, and the wine polarized after fermentation is complete. The conclusions are then the same as with the wines poor in sugar.

For polarization only large, exact instruments are to be used.

The rotation is to be calculated in degrees Wild according to Landolt (Zeitschr. f. analyt. Chemie, 7. 9):

$$1^\circ \text{ Wild} = 4.6043^\circ \text{ Soleil.}$$

$$1^\circ \text{ Soleil} = 0.217189^\circ \text{ Wild.}$$

$$1^\circ \text{ Wild} = 2.89005^\circ \text{ Ventzke.}$$

$$1^\circ \text{ Ventzke} = 0.346015^\circ \text{ Wild.}$$

*Gum (arabic).*—For establishing the addition of any considerable quantities of gum 4cc. wine are treated with 10cc. of 96 per cent. alcohol. If gum is present, the mixture becomes milky, and only clears up again after several hours. The precipitate which occurs adheres partly to the sides of the tube, and forms hard lumps. In genuine wine flakes appear after a short time, which soon settle, and remain somewhat loose. For a more exact test it is recommended to evaporate the wine to the consistency of a sirup, extract with alcohol of the strength given above, and dissolve the insoluble residue in water. This solution is treated with some hydrochloric acid (of specific gravity 1.10) heated under pressure two hours, and the reducing power ascertained with Fehling's solution, and calculated to dextrose. In genuine wines no considerable reduction is obtained in this way. (Dextrin is to be detected in the same way.)

*Mannite.*—As the presence of mannite in wines has been observed in a few cases, it should be considered when pointed crystals make their appearance in the extract or the glycerine.

*Nitrogen.*—In the estimation of nitrogen the soda-lime method is to be used.

*Mineral matters.*—For their estimation 50cc. of wine are used. If the incineration is incomplete, the charcoal is leached with some water, and burned by itself. The solution is evaporated in the same dish, and the entire ash gently ignited.

*Chlorine estimation.*—The wine is saturated with sodic carbonate, evaporated, the residue gently ignited and exhausted with water. In this solution the chlorine is to be estimated volumetrically according to Volhard, or gravimetrically. Wines whose ashes do not burn white by gentle ignition usually contain considerable quantities of chlorine (salt).

*Sulphuric acid.*—This is to be estimated directly in the wine by the addition of barium chloride. The quantitative estimation of the sulphuric acid is to be carried out only in cases where the qualitative test indicates the presence of abnormally large quantities. (In the case of viscous or very muddy wines a previous clarification with Spanish-earth is to be recommended.)

If in a special case it is necessary to investigate whether free sulphuric acid or potassium bisulphate are present, it must be proved that more sulphuric acid is present than is necessary to form neutral salts with all the bases.

*Phosphoric acid.*—In the case of wines whose ashes do not react strongly alkaline the estimation is made by evaporating the wine with sodic carbonate and potassic nitrate, the residue gently ignited and taken up with dilute nitric acid; then the molybdenum method is to be used. If the ash reacts strongly alkaline the nitric-acid solution of it can be used directly for the phosphoric-acid determination.

The other mineral constituents of wine (also alum) are to be determined in the ash or residue of incineration.

*Sulphurous acid.*—One hundred cubic centimeters wine are distilled in a current of carbonic acid gas after the addition of phosphoric acid. For receiving the distillate 5cc. of normal iodine solution are used. After the first third has distilled off, the distillate, which must still contain an excess of free iodine, is acidified with hydrochloric acid, heated and treated with barium chloride.

*Adulteration of grape wine with fruit wine.*—The detection of this adulteration can only exceptionally be carried out with certainty by means of the methods that have so far been offered. Especially are all methods untrustworthy which rely upon a single reaction to distinguish grape from fruit wine; neither is it always possible to decide with certainty from the absence of tartaric acid or from the presence of only very small quantities that a wine is not made from grapes.

In the manufacture of artificial wine together with water the following articles are known to be sometimes used: Alcohol (direct or in the shape of fortified wine), cane sugar, starch sugar, and substances rich in sugar (honey), glycerine, bitartate of potash, tartaric acid, other vegetable acids, and substances rich in such acids, salicylic acid, mineral matters, gum arabic, tannic acid, and substances rich in the same (e. g., kino, catechu), foreign coloring matters, various ethers and aromas.

The estimation or rather the means of detecting the most of these substances has already been given above with the exception of the aromas and ethers, for which no method can as yet be recommended.

The following substances may be mentioned here in particular, which serve for increasing the sugar, extract, and free acid: Dried fruit, tamarinds, St. John's bread, dates, figs.

B.—*Rules for judging of the purity of wine.*

I. (a) Tests and determinations which are, as a rule, to be performed in judging of the purity of wines: Extract, alcohol, sugar, free acids as a whole, free tartaric acid qualitative, sulphuric acid, total ash, polarization, gum, foreign coloring matters in red wines. (b) Tests and determinations which are also to be carried out under special circumstances: Specific gravity, volatile acids, bitartate of potash, and free tartaric acid quantitative, succinic acid, malic acid, citric acid, salicylic acid, sulphurous acid, tannin, mannite, special ash constituents, nitrogen.

The Commission considers it desirable, in giving the estimations generally performed, to adhere to the order of succession given above (under (a)).

II. The Commission cannot regard it as their province to give a guide for judging of the purity of wine, but thinks it advisable, in the light of its experience, to call attention to the following points:

Wines which are made wholly from pure grape juice very seldom contain a less quantity of extract than 1.5 grams in 100cc. wine. If wines poorer in extract occur they should be condemned, unless it can be proven that natural wines of the same district and vintage occur with a similar low content of extract.

After subtracting the "fixed acids" the remaining extract (*extractrest*) in pure wines, according to previous experience, amounts to at least 1.1 grams in 100cc., and after subtracting the "free acids," at least 1.0 gram. Wines which show less *extractrest* are to be condemned, in case it cannot be shown that natural wines of the same district and vintage contain as small an *extractrest*.

A wine which contains appreciably more ash than 10 per cent. of its extract content must contain, correspondingly, more extract than would otherwise be accepted as a minimum limit. In natural wines the relation of ash to extract approaches very closely 1 to 10 parts by weight. Still a considerable deviation from this relation does not entirely justify the conclusion that the wine is adulterated.

The amount of free tartaric acid in pure wines, according to previous experience, does not exceed one-sixth of the entire "fixed acids."

The relation between alcohol and glycerine can vary in pure wines between 100 parts by weight of alcohol to 7 parts by weight of glycerine; and 100 parts by weight of alcohol to 14 parts by weight of glycerine. In case of wines showing a different glycerine relation an addition of alcohol or glycerine can be inferred.

As sometimes during its handling in cellars small quantities of alcohol (at most 1 per cent. by volume) may find their way into wine this fact must be borne in mind in judging of its purity.

These proportions are not always applicable to sweet wines.

For the individual ash constituents no generally applicable limits can be given. The opinion that the better kinds of wine always contain more phosphoric acid than others is unfounded.

Wines that contain less than 0.14 gram of mineral matter in 100cc. are to be condemned, if it cannot be shown that natural wines of the same kind and the same vintage, which have been subjected to like treatment, have an equally small content of mineral matter.

Wines which contain more than 0.05 gram of salt in 100cc. are to be condemned.

Wines that contain more than 0.092 gram sulphuric acid ( $SO_3$ ) corresponding to 0.20 grams potassie sulphate ( $K_2SO_4$ ) in 100cc., are to be designated as wines containing too much sulphuric acid, either from the use of gypsum or in some other way.

Through various causes wines may become viscous, black, brown, cloudy, or bitter; they may otherwise change essentially in color, taste, and odor. The color of red wines may also separate in a solid form; still all these phenomena in and of themselves would not justify the condemnation of the wine as not genuine.

If during the summer time an energetic fermentation commences in a wine, this does not justify the conclusion that an addition of sugar or substances rich in sugar, *e. g.*, honey, &c., has taken place, for the first fermentation may have been hindered in various ways or the wine may have had an addition of a wine rich in sugar.

The methods adopted by the "Union of Bavarian Chemists" differ considerably from the above in many particulars, so they are given also, together with the methods adopted by the same body for the examination of beer<sup>1</sup> in somewhat condensed form.

### WINES.

#### METHODS OF INVESTIGATION.

**I. Determination of specific gravity.**—This is to be done by means of a Westphal's balance or a piconometer, and always at 15° C.

**II. Determination of extract.**—Ten to 50cc. wine at 15° C. are evaporated in a platinum dish on the water bath to the proper consistence and then dried in a drying oven at 100° C. to constant loss of weight. Constant loss of weight is assumed when three weighings, with equal intervals between the first and second and second and third give equal differences between the successive weighings.

Weighings are to be made at intervals of fifteen minutes.

**III. Inorganic matter.**—This is the incombustible ash obtained by burning the extract. Repeated moistening, drying, and heating to redness are advisable to entirely get rid of all organic constituents.

**IV. Acidity.**—After shaking vigorously, to drive off carbonic acid, the wine is to be titrated with an alkali solution and the acidity expressed in terms of tartaric acid.

**V. Glycerine.**—(1) This is determined in dry wines as follows: The alcohol is driven off from 100cc. wine, lime or magnesia added, and the mass evaporated to dryness. The residue is boiled with 90 per cent. alcohol, filtered, and the filtrate evaporated to dryness. This residue is dissolved in 10–20cc. alcohol, 15–30cc. ether added, and the mixture allowed to stand until it is clear. It is then decanted from the sticky precipitate into a glass-stoppered weighing bottle, evaporated to constant loss of weight, and weighed.

(2) The following method is employed for sweet wines: 100cc. wine are measured into a porcelain dish and evaporated on the water bath to a sirupy consistence, mixed with 100–150cc. absolute alcohol, poured into a flask, ether added in the proportion of  $1\frac{1}{2}$  volumes to each volume of alcohol used, the flask well shaken, and allowed to stand until the liquid becomes clear. This is then poured off and the residue again treated with a mixture of alcohol and ether. The liquids are mixed, the alcohol and ether driven off, the residue dissolved in water, and treated as in (1).

(3) In all glycerine determinations it is necessary to take into consideration the loss of glycerine due to its volatility with water and alcohol vapor, and accordingly to add to the glycerine found 0.100 gram for each 100cc. of liquid evaporated.

(4) It is necessary to test the glycerine from sweet wines for sugar, and if any is present it must be estimated by Soxhlet's or Knapp's method and its weight subtracted from that of the glycerine.

**VI. Alcohol.**—The determination must be made by distillation in glass vessels and the results stated as follows: 100cc. wine at 15° C. contain x grams or cubic centimeters alcohol.

**VII. Polarization.**—(1) The wine is decolorized with plumbic subacetate.

<sup>1</sup> Hilger, Vereinbarungen u. s. w., p. 154.

(2) A slight excess of sodic carbonate is added to the filtrate from (1). Two cubic centimeters of a solution of plumbic subacetate are added to 40cc. white wine and 5cc. to 40cc. red wine, the solution is filtered and 1cc. of a saturated solution of sodic carbonate added to 21 or 22.5cc. of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and results estimated in equivalents of Wild's polaristrometer with 200mm. tubes.

(4) All samples rotating more than  $0.5^{\circ}$  to the right (in 220mm. tubes, after treating as above), and showing no change, or but little change, in their rotatory power after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotatory power of less than  $0.3^{\circ}$  to the right shows that impure glucose has not been added.

(6) Wines rotating between  $0.3^{\circ}$  and  $0.5^{\circ}$  to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented and their optical properties then examined.

**VIII. Sugar.**—This is to be determined by Soxhlet's or Knapp's method. The presence of unfermented cane sugar is to be shown by inversion, &c.

**IX. Potassic bitartrate.**—The determination of potassic bitartrate as such is to be omitted.

**X. Tartaric, malic, and succinic acids.**—(1) According to Schmidt and Hiepe's method.

(2) Determination of tartaric acid according to the modified Berthelot-Fleury method.

(3) If the addition of 1 gram finely powdered tartaric acid to 100 grams wine produces no precipitate of potassic bitartrate, the modified Berthelot-Fleury method must be employed to determine free tartaric acid.

**XI. Coloring matter.**—(1) Only aniline dyes are to be looked for.

(2) Special attention is to be paid to the spectroscopic behavior of rosaniline dyes, as obtained by shaking wines with amyl alcohol before and after saturation with ammonia.

(3) A qualitative test for alumina is not sufficient evidence of the addition of alum.

**XII. Nitrogen.**—To be determined according to the ordinary method.

**XIII. Citric acid.**—Presence to be shown by a qualitative test, as baric citrate.

**XIV. Sulphuric acid.**—To be determined in the wine after adding hydrochloric acid.

**XV. Chlorine.**—To be determined in the nitric-acid solution of the burnt residue by Volhard's method.

**XVI. Lime, magnesia, and phosphoric acid.**—These are determined in the ash fused with sodic hydrate and potassic nitrate, the phosphoric acid by the molybdenum method.

**XVII. Potash.**—Either in the wine ash, as the platinum double salt, or in the wine itself, by Kayser's method.

**XVIII. Gums.**—Presence shown by precipitation by alcohol; 4cc. wine and 10cc. 96 per cent. alcohol are mixed. If gum arabic has been added, a lumpy, thick, stringy precipitate is produced; whereas pure wine becomes at first opalescent and then flocculent.

#### METHODS OF JUDGING PURITY—(Beurtheilung).

##### Part I.

**I.** Commercial wines may be defined as follows: (a) The product obtained by the fermentation of grape juice with or without grape skins and stems. (b) The product obtained by the fermentation of pure must, to which pure sugar, water, or infusion of grape skins has been added. It must contain not more than 9 per cent. alcohol and 0.3 per cent. sugar, and not less than 0.7 per cent. acid, estimated as tar-

taric. (c) The product obtained in southern countries by the addition of alcohol to fermented or partly fermented grape juice. French wines are not included, however. (d) The product obtained by fermenting the expressed juice of more or less completely dried wine grapes.

II. The above definitions do not apply to champagnes.

III. The following include the operations undergone by wines in cellars (*Keller-mässige Behandlung*): (a) Drawing and filling. (b) Filtration. (c) Clarification by the use of kaolin, isinglass, gelatine or albumin, with or without tannin. (d) Sulphuring. Only minute traces of sulphurous acid may be contained in wine for consumption. (e) Adulteration of wine. (f) Addition of alcohol to wine intended for export.

IV. Wines, even if plastered, must not contain more sulphuric acid than that corresponding to 2 grams potassic sulphate ( $K_2SO_4$ ) per liter.

V. Medicinal wines are those mentioned in Parts I and IV, with the following restrictions: (a) They must not contain more sulphuric acid than corresponds to 1 gram potassic sulphate per liter. (b) They must contain no sulphurous acid. (c) The percentage of alcohol and sugar to be given on the label. (d) These restrictions apply only to wines expressly recommended or sold for medicinal use.

### Part II.

I. Improperly gallized wines are preparations of grape juice, pure sugar and water, or grape-skin infusion, that contain more than 9 per cent. alcohol or less than 0.7 per cent. acid, or both, and preparations in which impure glucose has been used. The following facts enable us to detect them: Small quantity of inorganic matter (phosphoric acid and magnesia), and right rotation if impure glucose is used. If the rotation exceeds  $0.2^\circ$  to the right, the wine is to be concentrated, freed from tartaric acid as far as possible, and again polarized.

II. Addition of alcohol is to be assumed if the ratio of alcohol to glycerine is greater than 10 to 1 by weight.

III. Addition of water and alcohol is recognized by the diminution in the quantity of inorganic matter, especially magnesia, phosphoric acid, and usually potash. Addition of water alone is recognized in the same way.

IV. Scheelization, *i. e.*, addition of glycerine, is assumed if the ratio of glycerine to alcohol exceeds 1 to 6 by weight.

V. The presence of cane sugar is ascertained by a determination of sugar (by Soxhlet's or Knapp's method), before and after inversion.

### BEER.

#### A.—METHODS OF INVESTIGATION.

By beer is to be understood a fermented and still fermenting drink, made from barley (or wheat) malt, hops, and water, and which was fermented by yeast.

I. *Determination of specific gravity*.—For this as well as all other determinations the beer is freed from carbonic acid, as far as possible, by half-filling bottles with it and shaking vigorously. It is then filtered. The specific gravity is then determined either by Westphal's balance or by a piconometer at  $15^\circ C$ .

II. *Determination of extract*.—Seventy-five cubic centimeters of beer are carefully weighed and evaporated in a suitable vessel to 25cc., care being taken to prevent boiling. After cooling, water is added until the original weight is reached, and the specific gravity of the liquid taken as in I. The per cent. of extract is obtained from this specific gravity by the use of a table constructed by Dr. Schultz, and is given as "per cent. extract, Schultz."<sup>1</sup>

<sup>1</sup> Hilger, p. 123.

III. Alcohol is determined by distilling the beer. A piconometer of about 50cc. capacity and with a graduated neck is used as a receiver. The piconometer is carefully calibrated. Seventy-five cubic centimeters of beer are distilled until the distillate reaches about the center of the scale on the neck of the piconometer. This is then cooled to 15° C., dried, and weighed, and the alcohol determined by means of Baumé's table.

$$A = \frac{D \cdot d}{g}$$

The percentage of alcohol by weight is to be given. In very acid beers it is necessary to neutralize before distilling.

IV. *Original gravity of wort*.—This may be ascertained, approximately, by doubling the per cent. by weight of alcohol found as above, and adding the per cent. of extract. As this procedure is not exact, it may be made more nearly so by using the formula

$$\frac{100 (E + 2.0665 A)}{100 + 1.0665 A}$$

V. Degree of fermentation. This is estimated by using the formula

$$V_1 = 100 \left( 1 - \frac{E}{c} \right).$$

VI. *Sugar determination*.—This is to be determined directly, in the beer previously freed from carbonic acid, by Soxhlet's method of weighing the reduced copper; 1.13 parts of copper correspond to 1 part anhydrous maltose.

VII. Determination of dextrin is seldom required, and if required is to be performed by Sachsse's method.

VIII. *Nitrogen*.—Twenty to thirty cubic centimeters are evaporated in a Hofmeister "schälchen" or on warm mercury, and the extract burned with soda-lime. The nitrogen may also be determined by Kjeldahl's method.

IX. *Acids*.—(a) Total acids: The carbonic acid is driven off from 100cc. of beer by heating in beakers for a short time to 40° C. and the beer then titrated with baryta water (one-fifth to one-tenth normal). The saturation point is reached when a drop of the liquid has no longer any action on litmus paper. The acidity is to be given in cubic centimeters normal alkali required for 100 grams beer and as grams per cent. of lactic acid. The indication "acidity" or "degree of acidity" is insufficient.

(b) Normal beer contains but a very small quantity of acetic acid. The determination of fixed acid in the repeatedly evaporated extract is to be cast aside. The acetic acid produced by souring of the beer is shown by the increase in total acids. A qualitative test of the presence of acetic acid in the distillate from beers containing acetic acid is sufficient. Neutralized beer is to be acidified with phosphoric acid and distilled. Weigert's method is recommended.

X. *Ash*.—Thirty to fifty cubic centimeters of beer are evaporated in a large tared platinum dish and the extract carefully burned. If the burning takes place slowly, the ash constituents do not fuse together.

XI. *Phosphoric acid*.—This is to be determined in the ash obtained by evaporating and burning in a muffle 50 to 100cc. of beer to which not too much baric hydrate has been added. The phosphoric acid is determined in the nitric acid solution of the ash by the molybdenum method.

XII. *Sulphuric acid*.—The direct determination is not permissible. The determination is to be made by using the ash prepared by burning with sodic hydrate and potassic nitrate or baric hydrate and proceeding in the ordinary way.

XIII. *Chlorine*.—This is to be determined in the ash prepared with sodic hydrate.

XIV. *Glycerine*.—Three grams calcic hydrate are added to 50cc. of beer, evaporated to a sirupy consistence, about 10 grams coarse sea-sand or marble added, and dried. The dry mass is rubbed up, put into a capsule of filter paper, placed in an extraction apparatus, and extracted for six to eight hours with 50cc. alcohol. To the light colored extract at least an equal volume of ether is added, and the solution, after stand-

ing a while, poured into or filtered into a weighed capsule. After evaporating the alcohol and ether, the residue is heated in a drying oven at  $100^{\circ}$  to  $105^{\circ}$  C. to a constant loss of weight. In beers rich in extract the ash contained in the glycerine can be weighed and subtracted. In case the glycerine contains sugar, this can be determined by Soxhlet's method and subtracted.

XV. Hop substitutes are to be determined by Dragendorff's method. Picric acid is to be determined by Fleck's method. In examining for alkaloids, check experiments with pure beer must in all cases be made.

XVI. *Sulphites*.—One hundred cubic centimeters of beer are distilled after the addition of phosphoric acid and the distillate conducted into iodine solution. After one-third has distilled over, the iodine-colored distillate is acidified with hydrochloric acid and baric chloride added. If sulphites were not contained in the beer no precipitate is observed, but at the utmost a turbidity.

XVII. *Salicylic acid*.—This may be shown qualitatively by shaking with ether, chloroform, or benzine. The solution is allowed to evaporate, the residue dissolved in water, and a very dilute solution of ferric chloride added. The addition of too much acid and too violent shaking is to be avoided. The smallest trace of salicylic acid may also be shown by dialysis, as it passes very readily through membrane.

NOTE.—All the results of an investigation are to be stated in percentages by weight.

#### B.—METHODS OF JUDGING PURITY OF BEERS.

I. It is unjust to demand in a fermented beer an exact ratio of alcohol to extract, as the brewer cannot regulate the degree of fermentation within narrow limits. As a rule, Bavarian draft and lager beers contain from 1.5 to 2 parts of extract for each part of alcohol, but a smaller proportion of extract would not necessarily prove the addition of alcohol or glucose (the former to the beer, the latter to the wort).

II. The degree of fermentation of a beer must be such that at least 48 per cent. of the original extract has undergone fermentation.

III. If glucose or other bodies poor in nitrogen have been used in appreciable quantity as substitutes for malt, the nitrogen contents of the beer extract will fall below 0.65 per cent.

IV. The acidity of a beer should not be greater than 3cc. normal alkali to 100cc. beer. Acidity of less than 1.2cc. normal alkali to 100cc. beer indicates previous neutralization. If the acids are composed principally of lactic acid a larger quantity may be present.

V. The ash of normal beer is not above 0.3 grams to 100 grams beer.

VI. The amounts of phosphoric and sulphuric acids and chlorine in beer extract vary within such wide limits that their determination signifies nothing as to the purity of the beer.

VII. The amount of glycerine in pure beer is not greater than 0.25 gram to 100 grams beer.

VIII. The following methods of clarifying beer are legal: (a) Filtration. (b) Well-boiled hazel or beech shavings. (c) Isinglass.

IX. The following methods of preserving beer are legal: (a) Carbonic acid. (b) Pasteurizing. (c) Salicylic acid, this only for beers intended for export to countries where the use of salicylic acid is not forbidden by law.

NOTE.—The preceding methods are also to be used in the examination of imported beer.

#### C.—ADMINISTRATIVE NOTE.

It is absolutely necessary that the beer be preserved in well-corked green-glass bottles. Stone jugs and such vessels are not to be used.

The beer samples are to be protected from light and kept at a low temperature. Care in making tests is, above all, necessary.

## APPENDIX C.

The following bill, recently adopted in New York, constitutes the only instance I have been able to find of legislation directed specifically against the adulteration of any form of fermented liquors in this country. The manner in which such bills are drawn has considerable influence upon their efficiency in preventing and punishing the fraud against which they are directed, and while it is not exactly within the province of this publication to make suggestions as to the proper form for such legislation, I feel impelled to venture the assertion that the crudity of this bill affords little evidence that any chemist was consulted or concerned in its composition:

AN ACT to define pure wines, half wines, made wines, and adulterated wines, and to regulate the manufacture and sale of half wines and made wines and to prohibit the manufacture or sale of adulterated wines within the State of New York.

[Passed June 18, 1887, three-fifths being present.]

*The people of the State of New York, represented in Senate and Assembly, do enact as follows :*

§ 1. All liquors denominated as wine, containing aleohol, "except such as shall be produced by the natural fermentation of pure undried fruit juice," or compounded with distilled spirits, or by both methods, except as permitted by section two of this act, whether denominated as wine or by any other name whatsoever, in the nature of articles for use as beverages, or for compounding with other liquors intended for such use, and all compounds of the same with pure wine, and all preserved fruit juices compounded with substances not produced from undried fruit, in the character of, or intended for use as beverages, or for use in the fermentation or preparation of liquors intended for use as beverages, and all wines, imitation of wines or other beverages produced from fruit which shall contain any alum, baryta salts, caustic lime, carbonate of soda, carbonate of potash, carbonic acid, salts of lead, glycerine, salcylic\* acid, or any other antiseptic, coloring matter, other than that produced from undried fruit, artificial flavoring, essence of ether, or any other foreign substance whatsoever which is injurious to health, shall be denominated as adulterated wine, and any person or persons who shall manufacture with the intent to sell, or shall sell, or offer to sell, any of such wine or beverages shall be guilty of a misdemeanor, and shall be punished by a fine of not less than two hundred dollars, or more than one thousand dollars, or imprisonment in the county jail for a term of not less than six months, or more than one year, or by both such fine and imprisonment in the discretion of the court, and shall be liable to a penalty of one dollar for each gallon thereof sold, offered for sale, or manufactured with intent to sell, and such wine or beverage shall be deemed a public nuisance and forfeited to the State, and shall be summarily seized

\* So in original.

and destroyed by any health officer within whose jurisdiction the same shall be found, and the reasonable expense of such seizure and destruction shall be a county charge.

§ 2. For the purpose of this act the words "pure wine" shall be understood to mean the fermented juice of undried grapes or other undried fruits, provided, however, that the addition of pure sugar to perfect the wine, or the addition of pure distilled spirits to preserve it, not to exceed eight per centum of its volume, or the using of the necessary things to clarify and fine the wine, which are not injurious to health, shall not be construed as adulterations, but such pure wine shall contain at least seventy-five per centum of pure grape or other undried fruit juice.

§ 3. For the further purpose of this act, should any person or persons manufacture with the intent to sell, or sell or offer to sell, any wine which contains less than seventy-five per centum and more than fifty per centum of pure grape or other undried fruit juice, and is otherwise pure, such wine shall be known, branded, marked, labeled and sold as "half wine," and upon each and every package of such wine, which shall contain more than three gallons, there shall be stamped upon both ends of such package, in black printed letters, at least one inch high and of proper proportion, the words "half wine," and upon all packages which shall contain more than one quart and up to three gallons, there shall be stamped upon each of such packages, in plain, printed black letters at least one-half inch high, and of proper proportion, the words "half wine," and upon all packages or bottles of one quart or less, there shall be placed a label, securely pasted thereon, on which label the words "half wine" shall be plainly printed in black letters at least one-fourth of an inch high and of proper proportion. Should any number of such packages be inclosed in a larger package, as a box, barrel, case, or basket, such outside package shall also receive the stamp "half wine," the letters to be of the size according to the amount of such wine contained in such outside package: *Provided, further,* That any person or persons who shall sell, offer for sale, or manufacture with the intent to sell any wine which shall contain less than fifty per centum of pure grape or other undried fruit juice, and is otherwise pure, such wine shall be known, stamped, labeled, and sold as "made wine," and shall be stamped, marked, and labeled in the same manner as prescribed in this section, except the words shall be in this case "made wine."

§ 4. If any person or persons shall sell, or offer for sale, or manufacture with intent to sell any wine of the kind and character as described in the third section of this act, which shall not be stamped, marked, or labeled after the manner and mode therein prescribed, such person or persons shall be guilty of a misdemeanor, and shall be punished by a fine of not less than two hundred dollars, or more than one thousand dollars for each and every offense, or by imprisonment in the county jail, not less than three months, or more than one year, or by both fine and imprisonment in the discretion of the court, and in addition thereto shall be liable to a penalty of one-half dollar for each gallon thereof so sold, offered for sale, or manufactured with the intent to sell or offer for sale. All penalties imposed by this act may be recovered with costs of action by any person, in his own name, before any justice of the peace in the county where the offense was committed; where the amount does not exceed the jurisdiction of said justice, or when such action shall be brought in the city of New York, before any justice of the district or of the city court of said city; and such penalties may be recovered in the like manner in any court of record in the State, but on recovery by the plaintiff in such case for a sum less than fifty dollars, the plaintiff shall only be entitled to costs to an amount equal to the amount of such recovery. It shall be the duty of any district attorney in this State, and he is hereby required to prosecute or commence actions in the name of the people of this State, for the recovery of the penalties allowed herein, upon receiving proper information thereof, and in all actions brought by such district attorney, one-half of the penalty recovered shall belong to and be paid over to the person or persons giving the information upon which the action is brought, and the other half shall be paid to the

treasnrer of the county in which said action is brought within thirty days from the time of its collection, and the said one-half shall be placed to the credit of the poor fund of the town or city in which the cause of action arose. All jndgments recovered in pursuance of the provisions of this act, with the interest thereon, may be collected and enforced by the same means and in the same manner as a judgment rendered in an action to recover damages for a personal injury. Two or more penalties may be included in the same action.

§ 5. The provisions of this act shall not apply to medicated wines, such as are pnt up and sold for medical purposes only.

§ 6. This act shall take effect on September first, eighteen hundred and eighty-seven.

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