

adulterated with the lighter colored cereal starches; hence it is very commonly found in these spices, both with and without other adulterants.

It is sometimes used, moreover, in small quantities in adulterated cayenne, mace, and various other spices, to counteract the colors of other dyestuffs, such, for instance, as ground redwood, which in itself would be too intense, if used as the sole artificial coloring agent in cayenne.

The chief ingredients of turmeric are starch, a slightly fluorescent, orange-yellow, volatile oil, a deep yellow coloring-matter (curcumin), soluble in alcohol, but insoluble in cold water, cellulose and a gum.

There are few, if any, records of the satisfactory analysis of turmeric, with the exception of two varieties of Bengal and Madras turmeric recorded in the recently issued 4th edition of König's *Nahrungs und Genussmittel*.¹

The following are results of analyses made in the writer's laboratory of three varieties of turmeric commonly used in this country, the samples being ground in each case from large fragments of the root-stock.

Variety.	Moisture.	Total ash.	Ash soluble in water.	Ash insoluble in hydrochloric acid.	Total nitrogen.	Protein. N X 6.25.	Total ether extract.	Volatile ether extract.	Non-volatile ether extract.	Alcohol extract.	Crude fiber.	Reducing matters by acid conversion in starch.	Starch by diastase method.
China . .	9.03	6.72	5.20	0.11	1.73	10.81	10.86	2.01	8.84	9.22	4.45	48.69	40.05
Pubna . .	9.08	8.52	6.14	...	0.97	6.06	12.01	4.42	7.60	7.28	5.84	50.08	29.56
Alleppi.	8.07	5.99	4.74	...	1.56	9.75	10.66	3.16	7.51	4.37	5.83	50.44	33.03
Average	8.73	7.07	5.36	...	1.42	8.88	11.17	3.19	7.98	6.96	5.37	49.73	34.21

MALT ANALYSIS.

BY H. AUG. HUNICKE.

Received July 13, 1904.

I. DETERMINATION OF EXTRACT.

WHILE great quantities of malt are evaluated by inspection, it cannot be said that any considerable amount is purchased by analysis. The purchaser, while largely guided by the color, justly

observes more particularly the physical character of the endosperm. For the determination of the latter characteristics several simple tests are in use which can all be replaced, in a qualitative sense, by the simple biting test. However, where brewing operations are conducted on a rational basis it is preferable to determine quantitatively the extract-yielding properties of the malt.

The result of the mashing process depends in the main upon the quality of the malt used, but with the same grain results differ very widely with changes of temperature. This fact is so well established that one is not surprised to find that the first condition of uniformity, agreed to by chemists, was that of well defined working temperatures varied at definite intervals.

The method of analysis used is as follows: Fifty grams of malt are ground very finely into a copper beaker, 200 grams of water of 45° C. added and placed in a water-bath. The mash is allowed to remain at 45° for thirty minutes, when the temperature is raised 1° every minute, so that after twenty-five minutes the mash will be at 70° C., where it is kept for one hour. During the whole process, which lasts one hundred and fifteen minutes, the mash should be stirred. Upon completion the beaker is cooled down to about 10° to 15° C., placed on a balance and cold water added until the total weight of the contents is 450 grams. Stirring vigorously, so as to mix well, the whole is thrown upon a dry filter and allowed to drain into a dry flask. The first 75 cc. are returned and 150 cc. of the filtrate taken for a specific gravity determination. The German standard method prescribes that the first 100 cc. be returned to the filter and the *whole filtrate*¹ used for the determination of specific gravity. The method adopted by the subcommittee of the United States Brewers' Association differs from the above methods in that 250 cc. water are added at the outset. After the mash reaches 70° the iodine test is applied "every five minutes until inversion has taken place. The mash is then cooled to about 15° C. and its net weight is then increased to 450 grams by the addition of water. The mash is then carefully and thoroughly mixed and a *quantity of clear wort, sufficient for the saccharimeter determination,*² is filtered through a coarse filter."

Those who have had occasion to make many extract determinations of malt are conscious of the lamentable fact that the causes

¹ The italics are the writers. The bearing of these differences will be discussed more fully in another paper.

affecting the results are so manifold that even with the most conscientious work discordant results are easily obtained. So, for example, it is generally known that the physical character of the grain affects the yield very materially, so that one can, with certainty, predict a higher yield when the grain is mealy, while, on the other hand, a lower yield may be expected when the grain is hard and glassy, or else has failed to germinate. This difference of property finds expression in the extract determination of malt ground to varying degrees of fineness. The same grain will yield very nearly the same amount of extract, whether malted well or poorly, within reasonable limits, when ground to a very fine powder. When, however, it is ground coarsely the extract determination gives a higher yield the more thoroughly the malting process has rendered the grain soluble. It is for these reasons that some chemists are in the habit of making for each malt analysis at least two grindings, one being fine and the other coarse. The result of the finely ground sample indicates the amount of extract the grain might yield in practice with perfect malting, while the coarse grinding indicates the amount of extract obtainable in practice. The coarse grinding should be so adjusted as to give results comparable with those obtainable in the brew-house.

It is evident that if the amount of extract yielded by any malt depends upon the degree of fineness to which it is ground, that in order to obtain comparable results in the laboratory, a mill of accurate construction must be used. The absolute lack of a laboratory mill, possessing the property of reliability as to the degree of comminution has left the matter of extract determination of malt in a state unworthy of an analytical method. However, since the newly designed Seck mill can be had it has become possible to produce a meal giving fairly uniform results. Unfortunately, the mill can be adjusted only to the coarser grades. Very fine grinding is impossible. The same firm has more recently built a mill for very fine grinding, which the writer has, however, not yet used.

After a season of experience with the first-mentioned mill, the writer observed, in making extract determinations, both from fine and coarse grindings, that the result of the coarse grinding did not vary as heretofore, apparently, with the physical properties of

the malt only, but seemed to vary also with the amount of water contained, the results being calculated to dry substance. This somewhat unexpected result was observed with sufficient regularity to make it comparatively certain. The writer, therefore, resolved to systematically investigate the effect of the variation of moisture on the yield of extract. For this purpose a sample of Wisconsin malt of the 1903 season was prepared by mixing twenty-four samples which had been analyzed during the previous winter and which varied in moisture from about 4 per cent. to nearly 6 per cent., the average being 4.64 per cent. The length of aeropire showed the following average results:

	Per cent.
0 to $\frac{1}{4}$	1.96
$\frac{1}{4}$ to $\frac{1}{2}$	0.08
$\frac{1}{2}$ to $\frac{2}{3}$	0.67
$\frac{2}{3}$ to $\frac{3}{4}$	2.83
$\frac{3}{4}$ to 1.....	66.46
1 and over.....	28.00

Otherwise, the physical properties may be said to have been of the average quality characteristic of Wisconsin grain when malted fairly well. Two determinations of extract had always been made, a finely ground one from a coffee-mill of French manufacture and a coarser one from the product of the Seck mill, set on the "25" mark. The former gave as an average result 70.64 per cent., extract, being 74.09 per cent., calculated to dry substance. The latter gave as an average result 66.60 per cent., extract, being 69.95 per cent., calculated to dry substance.

The mixed sample was treated with a current of fairly dry air for two days, which reduced the moisture to 4.44 per cent. At the same time the whole was thoroughly mixed, placed in a bottle, well stoppered and set aside for use. Portions of about 300 grams were filled into quart bottles and varying amounts of water added, the bottles being well corked, repeatedly shaken and allowed to stand a few days so as to allow the moisture to become uniformly diffused throughout the mass. It appears from the results given below that the preparation of the samples answered its purpose. This sample was marked "Malt I."

Six series of tests were made by varying the amount of moisture in the malt on the one hand and on the other hand grinding to 4° of fineness. The finest meal was ground in the coffee-

mill, already referred to, while three coarse grades, 0, 25, 50, were passed through the Seck mill, which was set at the marks indicated respectively.

SERIES I.

<i>a.</i> Moisture : — 4.46, 4.39, 4.48 and 4.42 = 4.44 per cent. average.				
	Fine.	0.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	70.82	68.92	67.14	54.32
Extract dry	74.11	72.12	70.26	56.84
<i>b.</i> Moisture : — 6.24, 6.23, 6.14 and 6.11 = 6.18 per cent. average.				
	Fine.	0.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	69.81	66.50	65.03	48.79
Extract dry	74.41	70.88	69.31	52.00
<i>c.</i> Moisture : — 7.89, 7.86, 7.85 and 7.79 = 7.85 per cent. average.				
	Fine.	0.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	67.58	64.78	62.82	45.40
Extract dry	73.24	70.30	68.17	49.27
<i>d.</i> Moisture : — 9.60, 9.49, 9.67 and 9.48 = 9.56 per cent. average.				
	Fine.	0.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	66.26	62.44	59.49	40.55
Extract dry	73.26	69.04	65.78	44.84
<i>e.</i> Moisture : — 11.12, 10.95, 11.33 and 10.69 = 11.02 per cent. average.				
	Fine.	0.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	64.87	59.35	45.61	31.19
Extract dry	72.90	66.70	51.26	35.05
<i>f.</i> Moisture : — 12.92, 12.65, 12.82 and 12.88 = 12.82 per cent. average.				
	Fine.	0.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	64.20	59.29	52.29	31.94
Extract dry	73.64	68.01	59.98	36.64

It seemed desirable to extend the investigation to unground malt and accordingly one test of each of the watered samples of "Malt I" was made with the following results:

SERIES II.

Moisture.	Extract.	Extract, dry.
Per cent.	Per cent.	Per cent.
4.44	5.30	5.55
6.18	5.31	5.66
7.85	5.56	6.03
9.56	5.42	5.99
11.02	5.58	6.27
12.82	5.46	6.26

These values should all be equal, which is practically the case. A slight increase of extract with increase of water is, however, discernible, though not too much value should be attached to this observation because the differences are evidently not greater than would be expected from the nature of the process.

Another sample was similarly prepared. Being of similar origin as the first sample, the results are directly comparable. This was marked "Malt II."

SERIES III.

<i>a.</i> Moisture: — 4.43, 4.46, 4.51 and 4.41 = 4.45 per cent. average.				
	Fine.	o.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	70.96	69.87	68.06	51.24
Extract dry	74.26	73.12	71.23	53.63
 <i>b.</i> Moisture: — 6.36, 6.26, 6.32 and 6.27 = 6.32 per cent. average.				
	Fine.	o.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	69.25	67.59	65.58	65.61
Extract dry	73.91	72.13	69.99	70.02
 <i>c.</i> Moisture: — 8.06, 8.00, 7.97 and 7.91 = 7.99 per cent. average.				
	Fine.	o.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	68.33	65.73	63.10	39.85
Extract dry	74.26	71.66	68.58	43.31
 <i>d.</i> Moisture: — 9.70, 9.69, 9.66 and 9.69 = 9.69 per cent. average.				
	Fine.	o.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	66.75	63.74	59.13	34.04
Extract dry	73.91	70.58	65.47	37.69
 <i>e.</i> Moisture: — 11.56, 11.45, 11.40 and 11.41 = 11.45 per cent. average.				
	Fine.	o.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	65.27	61.46	53.95	27.24
Extract dry	73.71	69.41	60.70	30.76
 <i>f.</i> Moisture: — 13.29, 13.21, 13.24 and 13.14 = 13.22 per cent. average.				
	Fine.	o.	25.	50.
	Per cent.	Per cent.	Per cent.	Per cent.
Extract	64.46	59.06	48.21	22.10
Extract dry	74.28	68.06	55.55	25.47

In the two principal series, I and III, the decrease of yield of extract with increase of moisture becomes so pronounced that there can be little doubt of its reality. While the finest grades indicate a certain degree of uncertainty in this respect, a general agreement is, however, observable. The apparent discrepancy in the results containing 12.82 per cent. and 13.22 per cent. water,

respectively, is due to the difficulty of passing malt of such high water content through the coffee-mill without subjecting it to a long-continued grinding action, the damp particles adhering at times. The coarser grades all prove the rule. This comes out very clearly in Series III, where special care was exercised in stirring uniformly at equal intervals.

There may be several reasons for this variation of yield with variation of water content. The amount of moisture in a sample of malt affects the degree of fineness obtained with any given mill by making the individual grains softer and, therefore, more grinding, causing them, upon compression, to behave much like a little bag containing water, the more so the higher the water content. Such softened grains yield very little dust in the process of grinding. The starch cells are not disrupted to any great extent when the water content is high. Just to what extent a higher water content will affect the yield of extract will always depend more or less upon the quality of the malt. The latter always affects the degree of fineness resulting from any disintegrating process. Evidently then the degree of fineness of the ground product depends upon the brittleness or lack of coherence of the internal structure of the grain. Anything which decreases the brittleness either by causing greater toughness or greater softness, which may either be due to an increased toughness of the starchy content or softening of the cell wall and their contents, will lower the yield of extract.

The lower yield of extract from increased percentage of water may perhaps also be accounted for in a measure by the increased dilution of the charge, thereby retarding the reaction both as to rate and quantity. This is shown by the following results:

"Malt I" was used. It was ground in the coffee-mill to the finer grade and four tests made by varying the charge of malt; otherwise all conditions remained as usual. The amounts of malt taken were 50, 40, 30 and 20 grams respectively, giving:

SERIES IV.

	Fine. Per cent.	40. Per cent.	25. Per cent.	50. Per cent.
Extract	71.04 ¹	70.39	70.07	67.87
Extract dry . . .	74.34 ¹	73.65	73.31	71.02

¹ Strictly these two figures should be 70.82 per cent. and 74.11 per cent. respectively as in Series I. This difference corroborates the repeatedly observed variation of the results from the coffee-mill which is not absolutely reliable in the uniformity of results.

This certainly shows that the yield of extract is lowered to a appreciable extent by dilution and within the errors of the method it would almost appear to explain the decreased yield of Series I and III, but it is by no means quite clear whether dilution can account for all of the loss of yield, especially as results are wanting on coarser grades. The entire supply of the prepared sample of malt having been used, it was impossible to extend the research to the coarser grades.

The results given thus far prove conclusively the relation of yield to water content. The various results given are by no means as concordant in other respects as might be desired. It is hardly possible to enumerate all the various causes tending to disturb the results. The writer has set out to discover some of the many influences affecting the results. Of course, the matter of working temperatures can be dismissed at once, as under a circumstances great care must be observed in maintaining the prescribed temperatures. This, however, is by no means easily accomplished and, therefore, remains as a variable factor to some extent. But one factor which appeared to need some inquiry was that of the influence of stirring. To investigate this, three series of extract determinations were made.

Samples of "Malt I" were ground in the Seck mill set at 25 and 50 marks respectively. The first sample was treated four different ways, the first being stirred every minute, the second every five minutes, the third every fifteen minutes and the fourth not at all.

SERIES V.

	Fine. Per cent.	0. Per cent.	25. Per cent.	50. Per cent.
Extract	55.68	54.00	53.81	53.43
Extract dry	58.27	56.57	56.31	55.91

The second sample was also treated in four different way. The first was stirred during the entire time of mashing, one hundred and fifteen minutes, the second during the first eighty-five minutes, the third during the first fifty-five minutes and the fourth during the first thirty minutes.

SERIES VI.

	Fine. Per cent.	0. Per cent.	25. Per cent.	50. Per cent.
Extract	56.60	55.65	55.96	53.73
Extract dry	59.23	58.24	58.56	56.23

The third sample was again treated in four different ways, corresponding to those of the first sample.

SERIES VII.

	Fine. Per cent.	o. Per cent.	25. Per cent.	50. Per cent.
Extract	67.31	67.09	67.45	67.03
Extract dry	70.44	70.21	70.58	70.14

It will be observed that for coarse grindings a very perceptible decrease of yield is obtained when the mash is not stirred at all, and stirring even at intervals of five minutes will not give results as high as stirring every minute. So also does it become apparent from the results given that good stirring during the rising temperature is most essential. In finer grades of grindings the differences of yield, due to want of stirring, do not become great. In fact, it might be inferred from the results that very finely ground malt will yield up its entire extract without stirring at all or at most merely occasionally stirring. It follows from this that in the process of extract determination the mash should be stirred occasionally when working on finely ground malt, but when the test as applied to coarsely ground malt a well regulated method of stirring should be adopted. After the method of making two extract determinations of malt becomes more general it will be found necessary to agree on a uniform method of stirring. This applies particularly to the coarser material. It should not be unferred that the more vigorous the stirring the better the result. The danger lies rather in the opposite direction. The greater yield on the coarser grindings from a more vigorous stirring may be largely due to the disintegrating effect of the stirring, which must be greater with the coarsest grades of malt and the information sought by coarse grinding may thus be obscured.

Such disturbing factors as lack of uniformity of samples must always be an element of uncertainty in all extract determinations. Even with thorough mixing the variability of the individual rains and their weight, as compared with the charge, probably makes it impossible to expect a greater accuracy than 0.1 per cent., other errors being eliminated, for 1 grain of malt weighs about 0.025 gram, which, on a charge of 50 grams, is 0.05 per cent.

A loss, perhaps almost unavoidable, is that due to the formation of dust in the process of grinding and that, of course, applies

more particularly to the finer grades. It has been suggested to add from 1 to 3 grains of malt in excess of the 50 grams, taken for the test to cover the losses due to brushing off the parts of the mill. While this suggestion is a good one in a general way it cannot be applied with equal justification to all degrees of fineness or malts of high percentage of moisture. The better the malt and the lower the percentage of moisture the greater the loss from the formation of dust. Another method practiced to some extent is to weigh a charge somewhat in excess of 50 grams to grind the latter and weigh the exact amount afterward. This would appear acceptable if it were not for the hygroscopic properties of many samples of malt, more particularly those which have been dried to a high degree. The method is recommended by the German conventional method for fine grinding; for the coarse grindings 50 grams are weighed and then ground. If the loss due to the formation of dust is variable, there is no justification for adding a constant correcting factor in the form of one or more grains, hence this method is objectionable.

The rate of grinding must affect at least the coarser grade as the energy applied to each grain as it drops in between the rolls depends upon the speed of the rolls. The greater the speed of grinding the greater is the energy applied to the grain; the greater the latter the greater the comminution. It follows from this that at least for the coarser grades the mill should be adjusted to some standard speed, at least approximately, and the the feeding hopper be filled and grinding then begun with a regulated speed.

A source of error lies in the method of filtration. It is well known that the first drippings of the malt indicate a lower density, due to the adsorption of the extract by the filter-paper. The error is very generally obviated by returning the first running to the filter. Just to what extent this phenomenon of separation of solvent and solute during filtration affects the results of extra determination will be the subject of another paper.