THE ANALYSIS OF MALT.

BY JOHN A. MILLER, PH.D. Received March 19, 1894.

H AVING been called upon during the past few years to assay a great many samples of malt I have been struck by the variation which existed between the results of my own analyses and those made by other chemists upon the same samples. The variations in some cases were too large to be placed within the possibility of experimental error. With the belief that these variations might, in part at least, be due to a greater or lesser degree of accuracy in the methods employed in the analysis of malt, I undertook a comparative investigation of three methods which are quite universally used.

From the stand-point of the brewer, the largest consumer of malt, what are the important points to be ascertained by the analysis of a malt sample?

1. Moisture.

2. Percentage of extract which the malt will yield when submitted to a miniature mashing process.

3. Diastatic power, that is the rapidity with which the starch contained in the malt is converted into sugar and dextrine.

4. The percentage of acidity (calculated as lactic acid) which the wort contains.

It has been claimed by some that the percentage of sugar formed and the amount of proteids dissolved is of importance in judging of the character of the malt. The amount of sugar is of no practical value to the brewer as the increase or decrease of the percentage of that article is entirely dependent upon the manner in which the malt is handled in the mash tub. The total nitrogen in the malt wort calculated as proteids is of little value since the percentage present in the finished beer will depend upon so many factors as to render the first results of comparatively little value. Amongst the factors which will influence this percentage are: Character of the water used in mashing; the use of a high or low initial mashing temperature; the length of time the wort is boiled in the kettle; the amount and character of the hops used; the character and quantity of yeast used; and the character of the fermentation. The four points mentioned are of about equal importance in the assay of a malt sample.

The determination of moisture shows whether the malt has been properly dried and also whether the purchaser is not paying too high a price for an article which he can obtain very readily from the city water supply.

The determination of lactic acid is of value from the fact that it enables one to judge, within reasonable limits, of the age of the malt under examination. For example, a sample of malt which is low in moisture and high in lactic acid is an old malt which has been redried to bring down the high percentage of absorbed moisture.

In a first-class sample of malt the moisture should not run above five per cent., and the lactic acid 0.7 per cent.

Determination of Moisture.—Two methods were investigated in order to ascertain their relative accuracy. In the one method from two to four grams of ground malt was used; in the other twenty grams. In both cases the samples were heated to 100° C. until the weight was constant. Both methods gave results agreeing to within the third decimal place. The use of from two to four grams, however, is to be recommended:

1. Because it admits of the use of ground watch crystals for drying and weighing, thus preventing the absorption of moisture while the sample is cooling.

2. The time required to obtain a constant weight is much less than when twenty grams are used.

Determination of Extract.—The determination of the percentage of extract which a malt will yield is of very great importance, as upon this percentage, the value of the malt largely depends. It is evident that a sample of malt yielding fifty-five per cent. of extract has not the same commercial value as a malt yielding sixty per cent.

METHOD NO. I.

"Fifty grams of ground malt are weighed out as rapidly as possible (to avoid accession of water) and treated in a weighed beaker with 250 cc. of warm distilled water, of such a temperature that the initial heat of the mixture may be from $50^{\circ}-52^{\circ}$ C. The beaker containing the mash is placed in a water-bath and the contents maintained at the same temperature for a quarter of an hour. The heat is then gradually raised till the immersed thermometer registers 59°-60° C., and the temperature is then kept constant till a drop taken from the liquid ceases to give a blue color with iodine solution and nearly ceased to give a brown. This shows that all the starch and nearly all the erythrodextrine has suffered hydrolysis—a point which will be reached in about twenty minutes. The heat is then increased to about 70° C. in order to complete the saccharification, when the water in the bath is boiled for five minutes. This step which completes the process of mashing should be arrived at in about 100 minutes from the commencement of the operation. The beaker is then cooled and the contents filtered. The insoluble matter is washed with cold water and the filtrate is made up exactly to 400 cc. The density of the clear wort is next taken at 15.5° C. in the usual way by a specific gravity bottle. The excess of density over that of water (taken as 1,000) multiplied by 2.078 will give the percentage of dry extract yield by the malt. Instead of ascertaining the gravity of the infusion, the proportion of solid matter may be determined by evaporating a known measure of the wort to dryness in a flat-bottomed dish, so that the residue may form a thin film. The extract dried at 105° C. till constant in weight."

Two portions of fifty grams each of the same sample of malt treated as indicated above gave the following results:

I. 1I. Specific gravity of the wort.... 1.0284 1.0284 Extract calculated by factor....59.01 per cent. 59.01 per cent.

5.142 grams of the wort were then taken and placed in a wide flat-bottomed platinum dish and the extract dried at 105° C. to almost constant weight. The following was obtained:

I. II. Weight of the extract 0.3496 gram. 0.3488 gram. Average weight 0.3492 gram. Percentage of extract...... 55.87

5.142 grams were again taken and placed in a flat-bottomed dish and this in a water oven, the temperature of which was kept at $70^{\circ}-75^{\circ}$ C., and the extract dried to constant weight. The time required for this was about seventy hours.

METHOD NO. II.

"Fifty grams of ground malt are carefully weighed as rapidly as possible. The ground malt is put into a copper beaker, the weight of which is known, and this beaker is placed in a waterbath. Water is now mixed with the malt to the amount of 200 cc., at a temperature of 38° R. This temperature is held while the malt is continuously stirred for thirty minutes, when the temperature is raised to 58° R., 4° R. each five minutes. When the temperature of 58° R, is reached tests are made in order to find out whether the starch has been converted completely. Usually we find that after the temperature of 58° R. has been reached all the starch is converted. The mash is always held thirty minutes after the temperature of 58° R. has been reached, when it is boiled for five minutes, cooled off, and water enough is added to make the weight of the contents of the beaker, or the weight of the entire mash, 350 grams. The wort is then filtered off. After the wort has been filtered the specific gravity is taken from which the per cent. Balling is ascertained and the amount of extract in the malt computed to the following formula; viz.,"

5.192 grams of wort was then placed in a flat-bottomed dish and dried at 105° C. to almost constant weight. Results; $viz_{...}$

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Weight of extract	0.4520 gr	am.	0.4522 gram.
Average weight		0.4521	gram.
Extract		54.25	per cent.

The same amount of wort dried at $70^{\circ}-75^{\circ}$ C. for about seventy hours gave:

	T -	T1 .
Weight of extract	0,4924	0.4922
Average weight 0.49	923 grain	
Extract 59.0	7 per c	ent.
METHOD NO. III.		

Fifty grams of ground malt are weighed out as rapidly as possible, then placed in a weighed copper beaker and mixed with 200 cc. of water of a temperature of 40° C. The whole mixture is then carefully heated on an asbestos plate until the immersed thermometer registers 60° C. This temperature of 60° C, is maintained for twenty minutes, the mixture being almost constantly stirred during this time. At the end of twenty minutes a few drops of the solution are tested with iodine solution in order to ascertain whether the saccharification is complete. If the iodine gives the starch or erythrodextrine reaction the mash is further heated, the temperature being carefully raised 1° every two minutes until iodine solution ceases to give any reaction. It is very seldom that the temperature will go above 70° C. The flame is then removed, the mash cooled down and enough water added to make the total amount used equal to 400 grams, or the weight of the mash, that is, the malt plus the water equal 450 grams. After thoroughly mixing, the mash is thrown upon a plaited filter. The first half of the wort which filters through is thrown back upon the filter and then all which filters through collected. The specific gravity of this filtrate, or wort, is then taken by means of the Westphal balance. From this gravity the percentage given by Schultze's tables is ascertained and that number multiplied by 8.75, which gives the percentage of dry extract yield from the malt. The percentage can also be calculated by the use of the following formula:

> $(800 - \text{per cent. water}) \times \text{per cent. Schultze}$ 100 - per cent. Schultze.

The Schultze tables are so arranged that they give the amount of extract in 100 grams of wort of the specific gravity obtained. It would be a natural conclusion that the percentage represented by Schultze tables should be multiplied by 8 in order to obtain the amount of extract in 100 grams of malt, but this factor gives results which are below the absolute amount of extract which may be obtained from the malt. As the result of actual

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brewing experience I am of the opinion that the percentage Schultze multiplied by the factor 8 represents the amount of extract which the average brewer obtains from his material in actual practice, although the absolute amount obtainable on a small scale is higher. Two mashes of fifty grams each gave:

	ſ.	II.
Specific gravity of wort	1.0285	1.0285
Extract calculated from Schultze's) table. Factor 8	58.96 per cent.	5 8 .96 per cent.
Factor 8.75	64.487 '' ''	64.487
Extract calculated by formula	64.24	64.24

5.1425 grams of this wort were placed in a flat-bottomed dish and dried at 105° C. to almost constant weight. Results; *viz.*,

	1.	11.
Weight of extract	0.3511 gram.	0.3494 gram.
Average weight of extract.	····· 0.3502	gram.
Extract	56.03	per cent.

The same amount dried at $70^{\circ}-75^{\circ}$ C. for about seventy hours to constant weight gave :

	1.	11.
Weight of extract	0.3700 gram.	0.3681 gram
Average weight	J.3690	gram.
Extract	59.04	per cent.

In order to ascertain what variation, if any, existed between these results and the absolute amount of extract obtainable I made another mash from the same sample of malt which had been used for the preceding experiments. Method No. III was used with this exception that it was not made up to 400 grams, but was at once thrown upon a filter and washed with water at a temperature of 58° C. until the filtrate gave no reaction for sugar with Fehling's solution. This filtrate was then made up to 1,000 cc. and an aliquot part dried at 70°-75° C. to constant weight. Two portions of ten cc. each gave:

	Ι.		I1.	
Extract by weight in ten cc	0.3237 g	ram.	0.3236	gram.
" in 1,000 cc	32.37	" "	32.36	" "
Percentage of extract from malt,	64.74		64.72	
Average percentage			64.7	3.

The variations existing between the results obtained is apparent in the following table:

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No. I.	METHODS. No. II.	No. III.
Per cent. of extract calculated accord-		
ing to the directions of the method, 59.01	63.85	64.487
Method III using factor 8		58.96
" " " formula	• • • •	64.24
Extract dried at 105° C 55.87	54.25	56.03
$10^{\circ} - 75^{\circ} \text{ C. } \dots 58.99$	59.07	59.04
Absolute per cent. of extract obtainable,		64.73

A comparison of these results show that methods II and III give us figures agreeing closer to the actual amount of extract obtainable from the malt than method No. I, but even these are a little below the actual content. No. III, however, only slightly so. The results obtained by drying at 105° C. are too low and untrustworthy owing to a decomposition during the process of drying, as will be shown later in this paper. Looking at the results of the three methods as obtained by drying the extract at $70^{\circ}-75^{\circ}$ we find an excellent agreement between the maximum and minimum results, but a wide variation from the absolute amount of extract. This would indicate that a marked amount of starch was not saccharified and consequently would be lost as extract. It was only obtained by the washing of the grains with warm water.

Extract dried at 105° C.—In order to ascertain why the extract dried at 105° C. was so much lower than when calculated or dried at 70°-75°, a malt analysis was made with the following results:

The percentage of extract obtained by drying at 105° is over four per cent. lower than the percentage of extract obtained by the other methods. This would indicate that either the percentage of extract was actually lower than indicated by the other methods of determination, or that some substance had undergone decomposition and occasioned a loss which reduced the percentage. I was of the belief that the maltose had suffered decomposition at the temperature of 105° and therefore lowered the percentage of extract.

Determinations of sugar were consequently made:

1. In the wort obtained by method III and calculated to the total amount of extract obtained.

2. The maltose or sugar contained in the extract dried at $70^{\circ}-75^{\circ}$ was determined and a calculation made for the total from these results.

3. The sugar contained in the extract dried at 105° was determined and a similar calculation made.

Method	No. I	ΙI	· · · · · · · · · · · · · · · · · · ·	37.50	per	cent.
Extract	dried	at	70 [°] -75 [°] · · · · · · · · · · · · · · · · · · ·	37.50	11	••
"	* *	" "	105 [°]	31.50	• •	••

It will be seen that the percentage of maltose obtained from the extract dried at 105° is six per cent. lower than that obtained from the original wort and from the extract dried at $70^{\circ}-75^{\circ}$. This rather clearly indicates that a decomposition of maltose has taken place with a consequent loss.

In every case the extract dried at 105° was almost black in appearance and had a distinct burnt sugar odor.

CONCLUSIONS.

1. Methods II and III give results almost equal in accuracy. No. III giving nearer the absolute amount of extract obtainable when the factor 8.75 is used. It is furthermore a preferable method as it requires less time for the analysis than No. II. And where a number of samples are handled, time is an important item.

2. Method I is inaccurate as the results obtained are much below the actual amount of extract.

3. The extract can not be determined by drying at 105° owing to the decomposition of the maltose at that temperature. This applies to the determination of extract in beer as well as in unfermented worts.

4. The washing of the grains until no sugar reaction is obtained and the subsequent drying of the extract at $70^{\circ}-75^{\circ}$ C. is impracticable, except for scientific purposes, as the amount of time required for the completion of an analysis is too great.

I would recommend method III as the simplest; it is accurate when the factor 8.75 is used, and requires the least time. It is the method upon which a subsequent paper on diastatic power will be based.

I wish, here, to express my sincere thanks to my assistant, Mr. W. I. Tibbals, for the very able and enthusiastic manner in which he has assisted me in this investigation.

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