

TABLE VI.—READINGS OF MILK SERUM ON THE IMMERSION REFRACTOMETER REDUCED TO 20°.

| Temp. | Scale readings. | | | | | | | | | |
|-------|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 36. | 37. | 38. | 39. | 40. | 41. | 42. | 43. | 44. | 45. |
| 15° | 34.75 | 35.75 | 36.75 | 37.75 | 38.75 | 39.75 | 40.70 | 41.70 | 42.65 | 43.65 |
| 16 | 35.05 | 36.05 | 37.05 | 38.05 | 39.05 | 40.05 | 41.00 | 41.95 | 42.90 | 43.85 |
| 17 | 35.25 | 36.25 | 37.25 | 38.25 | 39.25 | 40.25 | 41.20 | 42.20 | 43.15 | 44.10 |
| 18 | 35.50 | 36.50 | 37.50 | 38.50 | 39.50 | 40.50 | 41.45 | 42.45 | 43.40 | 44.40 |
| 19 | 35.75 | 36.75 | 37.75 | 38.75 | 39.75 | 40.75 | 41.75 | 42.75 | 43.70 | 44.70 |
| 20 | 36.00 | 37.00 | 38.00 | 39.00 | 40.00 | 41.00 | 42.00 | 43.00 | 44.00 | 45.00 |
| 21 | 36.25 | 37.25 | 38.25 | 39.30 | 40.30 | 41.30 | 42.30 | 43.30 | 44.30 | 45.30 |
| 22 | 36.50 | 37.50 | 38.50 | 39.55 | 40.60 | 41.60 | 42.60 | 43.60 | 44.60 | 45.60 |
| 23 | 36.80 | 37.80 | 38.80 | 39.85 | 40.90 | 41.90 | 42.90 | 43.90 | 44.90 | 45.90 |
| 24 | 37.10 | 38.10 | 39.10 | 40.15 | 41.20 | 42.20 | 43.20 | 44.20 | 45.20 | 46.20 |
| 25 | 37.40 | 38.40 | 39.40 | 40.45 | 41.50 | 42.50 | 43.50 | 44.50 | 45.40 | 46.50 |

Example.—If at 25° the milk serum reads 42.3, the correct reading at 20° is $43.5 + 0.3 = 43.8$.

[CONTRIBUTION FROM THE DEPARTMENT OF FOOD AND DRUG INSPECTION
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COMPOSITION AND ADULTERATION OF GROUND MUSTARD.

BY ALBERT E. LEACH.

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MUSTARD has long taken the lead as the most extensively adulterated of all the spices, especially in localities where no system of food inspection prevails. The custom of deliberately mixing cereal flour or starch with ground mustard was practiced for many years, and, in fact, continued long after the serious adulteration of other spices had been held in check by the increased enforcement of pure food laws.

Coloring with turmeric was also a time-honored custom, and, in fact, still prevails, except in localities where the public have been educated to view with suspicion the deep yellow color of the product. Of late the employment of other colors than turmeric, such, for example, as various of the oil-soluble coal-tar dyes, has arisen to claim the attention of the analyst.

All the above forms of gross sophistication can, without doubt, be dealt with by almost any food commission or health board having in charge the enforcement of pure food laws, acting under the general food laws of their state, and without the fixing of definite standards. It is, however, in connection with other and perhaps milder forms of adulteration of mustard that the necessity of carefully fixed standards are of greater importance. The questions of how much starch, if any, should be allowed in ground mustard, due to weed seeds, and whether mustard hulls have been mixed with the mustard "flour" in excess of the amount found in the whole seed, should be largely settled by appropriate standards, if possible.

Commercial mustard "flour," unlike the other spices, is not ground from the whole seed, but is a manufactured product, being deprived, as a rule, of a large portion of the fixed oil and most of the hulls, so as to insure a fine, even powder. Hence in fixing standards for what the trade understands as mustard flour, difficulties are met with not encountered in the ground whole spices.

Very complete analyses of nearly all the spices of known purity, excepting mustard and turmeric, have been made by Winton, Ogden and Mitchell,¹ and the same analysts have made partial analyses of a large number of commercial mustard flours found on sale in Connecticut,² as follows:

TABLE I.

| | Total ash. | Ether extract. | | Reducing matters by acid conversion as starch. | Starch by diastase method. | Crude fiber. | Nitrogen $\times 6.25$. |
|--------------|------------|----------------|---------------|--|----------------------------|--------------|--------------------------|
| | | Volatile. | Non-volatile. | | | | |
| Maximum.... | 7.35 | 1.90 | 28.10 | 6.12 | 2.08 | 4.87 | 43.56 |
| Minimum.... | 4.81 | 0.00 | 17.14 | 1.85 | 0.28 | 1.58 | 35.63 |
| Average..... | 5.99 | 0.56 | 20.61 | 4.33 | 1.07 | 2.58 | 39.57 |

Richardson³ made the following analyses of whole seed flour, prepared by himself, without the removal of the fixed oil.

¹ Twenty-second Ann. Rep. Conn. Expt. Sta., 1898, p. 184.

² *Ibid.*, page 169.

³ U. S. Dept. of Agric., Div. of Chem., Bull. 13, Part 2, page 181.

TABLE II.

| | Water. | Ash. | Volatile oil. | Fixed oil. | Starch. | Crude fiber. | Albu- minoids. | Un- deter- mined. | Nitro- gen. |
|-------------------|--------|------|---------------|------------|---------|--------------|----------------|-------------------|-------------|
| White seed..... | 5.57 | 4.29 | 0.97 | 33.56 | 0.00 | 5.40 | 28.88 | 21.33 | 4.62 |
| White flour..... | 3.33 | 5.23 | 1.84 | 34.83 | 0.00 | 9.05 | 25.56 | 20.16 | 4.09 |
| Seed husk..... | 6.17 | 4.99 | 0.55 | 28.12 | 0.00 | 9.50 | 23.44 | 27.23 | 3.75 |
| California yellow | 4.83 | 5.96 | 1.27 | 31.96 | 0.00 | 8.50 | 31.13 | 16.35 | 4.98 |
| California brown | 4.11 | 4.88 | 1.35 | 36.63 | 0.00 | 16.18 | 24.69 | 12.16 | 3.95 |
| English yellow | 3.11 | 4.07 | 2.06 | 31.51 | 0.00 | 6.90 | 30.25 | 22.10 | 4.84 |
| Trieste brown .. | 4.62 | 5.61 | 0.63 | 39.55 | 0.00 | 10.84 | 25.88 | 18.87 | 4.14 |

Table III, which follows, shows the results of analyses made in the writer's laboratory of a number of the commoner varieties of mustard, both yellow and brown, acknowledgment being given to Mr. Arthur D. Smith for assistance in the analytical work. The mustard "flours" were prepared in the same manner as the commercial article, from seed separated as completely as possible from the hulls, and having the usual proportion of fixed oil removed by pressure. The "hulls" analyzed were some that had been separated from the seed in the ordinary process of sieving, in the manufacture of the flour, and afterwards reduced by grinding to a fine powder. The six varieties of whole seed examined were each ground directly in a porcelain power mill of the type known as a "chaser," yielding in each case a fine, oily mass, hardly suited for a commercial product, but very useful in serving as a basis for standards in that all the components of the seed were present in their natural proportions.

Methods of Analysis.—The methods of analysis employed were, in the main, those adopted provisionally by the Association of Official Agricultural Chemists.¹

In the case of the brown seed, the developed volatile oil was determined by the following method:

*Determination of Mustard Oil in Mustard Flour. Roeser's Method.*²—Mix 5 grams of the sample with 60 cc. of water and 15 cc. of 60 per cent. alcohol, and let stand for two hours. Distil into a flask containing 10 cc. of ammonia, and, after about two-thirds of the solution have been distilled off, mix the ammoniacal distillate with 10 cc. of tenth-normal silver nitrate solution, and allow the mixture to stand for twenty-four hours, after which make up with water to 100 cc. Filter, and treat 50 cc. of the

¹ U. S. Dept. of Agric., Bur. of Chem., Bull. 65, Part X, "Spices."

² *Jour. Pharm. Chim.*, 15, 361-364 (1902).

filtrate with 5 cc. of tenth-normal potassium cyanide solution. Titrate the excess of cyanide with the tenth-normal silver nitrate, using as an indicator a 5 per cent. solution of potassium iodide made slightly ammoniacal.

The percentage of mustard oil present is found by multiplying by 2 the number of cubic centimeters of silver nitrate solution taken up by the oil, and multiplying this product by the factor 0.3137.

Starch in Mustard.—Pure mustard contains absolutely no starch, yet mustard hulls of all varieties by the diastase method show considerable copper-reducing matter, which is not due to starch, and should not be attributed to it.

One sample of mustard hulls showed, by the diastase method, 7 per cent. of reducing matter, reckoned as dextrose,¹ in the moisture- and fat-free substance.

The material of the seed itself is comparatively free from such reducing matter, and when the separation from the hulls is complete, should show hardly a trace of reducing material by the diastase treatment. The mustard flours of the above table were practically free from contaminating starch, as shown by the microscope, the small amount of reducing action under the diastase treatment being undoubtedly due to the hulls which were left in them.

Hence it is often of considerable importance to ascertain whether the reducing matter is due to starch or to hulls, and this can readily be accomplished by a microscopical examination, which furnishes the very best, if not the only means of judging the character and extent of starch contamination, when the amount of starch is small.

In the standards recently adopted by the Secretary of Agriculture for certain food products,² standard ground mustard is defined as "containing not more than 2.5 per cent. of starch by the diastase method." It would seem as if some modification of this standard should be made.

From the fact that among the samples recorded in the above table 1.82 per cent. is the maximum of reducing material reckoned as dextrose found in the air-dry substance for mustard *ground from*

¹ The name dextrose is retained in place of the correct scientific name, *d*-glucose, because the former is always used in the publications of the A. O. A. C.—EDITOR.

² Office of the Sec. of Agric., Circular No. 10, p. 11.

the entire seed, and found by the microscope to contain only the minutest traces of contaminating weed starch, it would seem as if 2.5 per cent. of reducing matter in the air-dry powder was excessive.

Any actual starch present in ground mustard is due either to fraudulent admixture, or to the presence of starch-containing weed seeds common in some of the cheaper varieties of mustard seed. If contamination with foreign seed were to be legalized to the extent of 2.5 per cent. of actual starch, this would mean a percentage of foreign material far in excess of that figure, since starch is but one ingredient of these foreign seeds.

In view of the fact that starch is foreign to pure mustard, and that copper-reducing materials, other than starch, exist in mustard hulls under the diastase treatment, it would seem as if the standard should rule out more than mere traces of starch, as shown by the microscope. Furthermore, since the removal of the fat in more or less degree is customary, it would be better to express constants on the moisture- and fat-free basis, limiting the reducing matter by the diastase method in the moisture- and fat-free substance to, say, 2.5 per cent. not of actual starch, according to the present wording of the standard, but to "reducing matter reckoned in terms of dextrose." or, if desired, the equivalent "in terms of starch."

Typical of the cheap varieties of mustard high in foreign weed seed, which, it is claimed by manufacturers, cannot be removed from the mustard by sieving or by winnowing, are the Dakota mustard and some of the lower grade German seeds. By reason of the cheapness of these seeds there is constant temptation to use them. Such varieties were for obvious reasons not included in the above table, though the varieties analyzed were intended to cover a tolerably wide range. It is, in fact, questionable whether seed so highly contaminated with foreign material as the Dakota mustard should be allowed at all.

Following is the analysis of a sample of Dakota mustard flour, found by the microscope to be free from more than traces of hull tissue. It was found to contain over 3 per cent. of actual starch in the water- and fat-free substance, since, in the absence of hulls, practically all the reducing matter under the diastase treatment was due to starch.

TABLE IV.—DAKOTA MUSTARD FLOUR.

| | Air-dry powder. | Moisture- and fat-free substance. |
|---|-----------------|-----------------------------------|
| Moisture | 7.42 | |
| Total ash | 7.80 | 9.71 |
| Water-soluble ash | 0.46 | 0.57 |
| Ash insoluble in hydrochloric acid. | 0.75 | 0.93 |
| Ether extract..... | 12.23 | |
| Alcohol extract..... | 23.81 | 29.64 |
| Total nitrogen..... | 6.84 | 8.51 |
| Volatile oil..... | 3.76 | 4.68 |
| Crude fiber..... | 2.28 | 2.84 |
| Reducing matter, by acid conversion, as dextrose..... | 14.05 | 17.48 |
| Reducing matter, by diastase, as dextrose | 2.87 | 3.57 |
| Reducing matter, by diastase, as starch..... | 2.58 | 3.21 |

Numerous varieties of starch-containing weed seed were found in the whole Dakota mustard seed from which the above flour was prepared. Three of the most common varieties were picked out and analyzed for starch. One of these, evidently an inferior grade of broken wheat, was found to contain 70.93 per cent. of starch; another, a small circular seed, contained 70.65 per cent.; and a third, a small angular seed, contained 67.5 per cent. of starch, all three starch determinations being made by the diastase treatment.

Added Mustard Hulls.—Another method of producing a cheap mustard flour consists in the addition of ground mustard hulls. While it is true that the higher grades of mustard flour are deprived almost entirely of hulls, some are occasionally found that contain added hulls to an extent exceeding the amount of hulls in the whole seed. This practice should obviously be considered as a form of adulteration. Such an excess of hulls is rendered obvious by a microscopical examination, and may be confirmed by a chemical analysis. For this purpose certain additional standards would be helpful.

The main points of difference between hulls and seed substance of pure mustard are indicated chemically by such constants as the total nitrogen, the crude fiber and the reducing matter by diastase. The following table shows this in summarized form:

TABLE V.—IN THE WATER- AND FAT-FREE SUBSTANCE.

| | Mustard flour. Per cent. | Mustard hulls Per cent. | Whole seed. Per cent. |
|--|-----------------------------|----------------------------|--------------------------|
| Total nitrogen | Maximum | 9.61 | 5.00 |
| | Minimum | 8.27 | 3.40 |
| | Mean | 9.09 | 4.14 |
| Crude fiber | Maximum | 4.26 | 22.20 |
| | Minimum | 2.31 | 13.74 |
| | Mean | 3.24 | 18.11 |
| Reducing matter as dextrose, by diastase | Maximum | 0.93 | 7.06 |
| | Minimum | 0.00 | 1.51 |
| | Mean | 0.37 | 4.27 |

One sample of commercial mustard flour comparatively free from starch, but condemned by the writer on account of added hulls, as shown unmistakably by the microscope, exhibited the following constants in the water- and fat-free substance:

| | Per cent. |
|---|-----------|
| Total nitrogen | 6.97 |
| Crude fiber | 7.69 |
| Reducing matter as dextrose by diastase | 2.20 |

In this sample the microscope showed the fragments of hull to clearly form the chief feature of the mass, largely exceeding the material of the seed tissue.

Suggested Standards.—Based on the above analyses, the following standards are suggested for ground mustard, expressed on the moisture- and fat-free substance: Reducing material by diastase treatment should not exceed 2.5 per cent., expressed as dextrose; crude fiber should not exceed 5 per cent.; and total nitrogen should not be less than 8 per cent. As shown by the microscope, the sample should be free from more than minute traces of starch, and should not exhibit an excess of hulls over seed tissue.

THE COMPOSITION OF TURMERIC.

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TURMERIC (*Curcuma longa* of the family *Zingiberaceae*), while of chief interest to the analyst as an adulterant of other spices, possesses some value as a condiment in itself, forming, as it does, the chief ingredient of curry powder. Turmeric is a material especially adapted by its deep yellow color to intensify the color of mustard and ginger, especially when these spices are also