the liquid to be filtered may be gradually poured in the platinum dish, and subsequently hot water is added to wash the precipitate.

After the operation is completed, if the platinum tube is slipped from the rubber tube which connects it with the aspirator, the water held in the platinum tube will fall back in the crucible. This is easily got rid of by subsequent evaporation, but this quantity of water, and that which remains below the platinum plate, may be mostly carried off through the aspirator by carefully removing the precipitate from a point on the edge of the paper disk, and lifting this up with the point of a needle just sufficiently to let air go in, to clear the aspirator tube from liquid.

XXXIII.—A METHOD FOR THE ANALYSIS OF MUSTARD.

BY ALBERT R. LEEDS AND EDGAR EVERHART.

During the past summer a large number of analyses of various articles of food and drink was made by the authors, on behalf of the State of New Jersey. Among the articles analyzed were thirty different kinds of mustards, as sold in shops. The method of analysis followed was that given in all text-books on the subject, and especially recommended by Blythe and Hassall. The process consists, as is well known, in estimating the moisture by drying at 100–110°; in determining the ash for the detection of mineral adulteration, and weighing the oil. The other adulterations are determined only qualitatively.

In this method reliance is placed principally on the estimation of the oil, and from this is calculated the amount of mustard supposed to be present in the mixture. Blythe gives the following formulæ for calculating the amount of mustard in a mixture of flour and mustard: x = amount of mustard and y amount of oil found,

$$\frac{33.9x}{100} + \frac{1.2\,(100 - x)}{100} = y \text{ and } \frac{36.7x}{100} + \frac{2\,(100 - x)}{100} = y.$$

This method of analysis and calculation might do very well if no foreign fat were added, or if none of the original mustard oil were extracted. In fact, however, flour baked in oil or fat is frequently added to a mustard from which a portion of the original oil has been extracted, and furthermore, mustard, before being put on the market, is subjected to hydraulic pressure and loses from 12 to 20 per cent. of its oil. From this method of analysis, it is evident that satisfactory or reliable results cannot be obtained.

So far as is known, there has been only one attempt made to analyze mustard by its separate constituents. Hassall, in his book on "Food, its Adulterations and the Method for their Detection," proposes an analysis, partly direct and partly indirect. His method, and the results obtained by it, are those most quoted both in English and foreign books and journals.

His plan of analysis is as follows : The moisture and ash are determined as ordinarily, and the oil by extracting with ether. The myronate of potash is estimated by taking advantage of its well-known reaction with the myrosine contained in the mustard flour, in presence of water. Forty or fifty grains of the mustard are allowed to digest for twenty-four hours, with about 250 c.c. of water in a well-corked flask. At the end of that time all the myronate of potash will have been decomposed by the ferment myrosine into glucose, sulphate of potash, and mustard oil (allylthiocarbamide), according to the equation $C_{10}H_{18}KNS_2O_{10}=C_6H_{12}O_6+KHSO_4+C_4H_5NS$. The contents of the flask are distilled, and all of the allylthiocarbamide goes over with the water vapor. The end of the condenser dips below the surface of some strong ammonia water to prevent loss of the volatile oil. When no more oily drops come over with the distillate, the receiver and its contents are removed and allowed to stand until the allylthiocarbamide has combined with the ammonia, forming thiosinnamine ($C_4H_5NS.NH_3$). The solution is evaporated to dryness in a tared platinum dish, and from the amount of thiosinnamine found, is calculated the myronate of potash.

So far the method is all that can be desired, but the remaining part is open to very serious objections. For the determination of the myrosine and sulphocyanide of sinapine, a combustion of the mustard is made with soda-lime for the total nitrogen, and another portion is fused with alkaline carbonates and nitrates, to estimate the total sulphur. As much nitrogen and sulphur as is contained in the myronate of potash, is subtracted from the total nitrogen and sulphur, and from the two residues are calculated the amounts of myrosine and sulphocyanide of sinapine. The cellulose is estimated by difference.

The whole of the sulphur residue, and so much of the nitrogen as is necessary, are calculated into sulphocyanide of sinapine, and the remaining nitrogen into myrosine. But as myrosine contains about 1.6 per cent. of sulphur, further calculations are necessary. Such calculations may or may not yield correct results, for one has as much right to calculate all the remaining sulphur first into myrosine, or all the nitrogen into either myrosine or sulphocyanide of sinapine, as the sulphur into the last-named compound. The results cannot be calculated algebraically because there is only one equation, and this equation has two unknown factors. In one of the samples of mustard analyzed by Hassall he finds myrosine 31.686 per cent., sulphocyanide of sinapine 5.714 per cent. Taking his percentages of nitrogen and sulphur, and calculating the total nitrogen first into myrosine, one finds, myrosine 31.43 per cent., sulphocyanide of sinapine 3.95 per cent.

For the reasons above detailed, the following attempt was made to work out a method for the analysis of mustard, which should be direct throughout, and should rest on an actual separation and estimation of the several constituents. The moisture and ash are determined as usual. The mustard oil is extracted with ether in the following manner: A weighed portion of mustard, after drying at 105°, is carefully brushed into a plaited filter. The filter and its contents are placed in a funnel with straight sides. The stem of the funnel is connected, by means of a well-fitting cork, with a small tared flask partially filled with ether, while the funnel itself is connected with an upright condenser. On cautiously boiling the ether, its vapor is constantly condensed, and, falling on the mustard, extracts the oil which is retained in the flask. When all of the oil is removed, the ether is distilled off, and the flask and contents, after drying at 100°, re-weighed. The difference between the weight of the flask alone, and that of the flask and oil, gives the amount of oil. After the ether has evaporated from the mustard residue, a tared flask, containing half water and half alcohol, is substituted for that containing ether, and the contents are boiled and condensed as before. The dilute alcohol dissolves both the sulphocyanide of sinapine and the myronate of potash, while it coagulates the myrosine and leaves both it and the cellulose undissolved. After all the sulphocyanide of sinapine and myronate of potash have been extracted, the contents of the flask are rinsed into a tared platinum dish, evaporated to dryness, dried at 105°, and weighed. The dish and contents are then ignited and weighed. The difference of weight before and after ignition, gives the total amount of sulphocyanide of sinapine and myronate of potash. Subtracting the amount of the latter, the difference is the amount of sulphocyanide of sinapine.

After the extraction with alcohol, the filter contains only the myrosine and cellulose, together with a little coloring matter. The alcohol is allowed to evaporate spontaneously, and then the myrosine and cellulose are treated in the cold with a one-half per cent. soda solution. The solution containing the myrosine is decanted through a weighed filter, and the residue is treated again in the same manner. By this treatment all the myrosine is obtained in solution. The cellulose on the filter is dried, weighed, ignited, and the ash weighed. The difference between the two weights gives the cellulose.

The solution containing the myrosine is just neutralized with dilute hydrochloric acid, and about 50 c.c. of Ritthausen's cupric sulphate solution added. The solution is then *exactly* neutralized with dilute soda, and the heavy green precipitate of the compound of copper and myrosine allowed to settle to the bottom of the beaker. The precipitate is collected on a weighed filter and dried at 110°. After drying, the weight of the precipitate is taken. It is then ignited, and the ash weighed, the difference giving the total amount of myrosine.

The two following analyses of a sample of Brown Mustard Farina, prepared by H K. & F. B. Thurber & Co., New York, were made simultaneously. Afterwards a third analysis was made on the same sample.

ANALYSIS OF BROWN MUSTARD FARINA.

	1	2	3
Moisture	6.78	6.90	6.82
Myronate of potash	0.61	0.61	0.72
Sulphocyanide of sinapine	10.97	11.19	11.21
Myrosine	28.45	28.70	28.30
Mustard oil	29.22	29.21	29.19
Cellulose by difference	20.24	19.55	20.06
Ash	3 73	3.84	3.70
	100.00	100.00	100.00

A combustion was made of the same mustard, and the nitrogen determined. The sulphur was likewise determined by fusing with alkaline carbonates and nitrates, and precipitating with barium chloride.

Nitrogen = 5.337 per cent. Sulphur = 1.489 per cent. Calculating the amounts of nitrogen and sulphur in the myronate of potash, the sulphocyanide of sinapine and the myrosine, we find nitrogen, 5.342 per cent., and sulphur, 1.50 per cent.

If the amounts of the three last named constituents of the mustard be deduced from the total amounts of nitrogen and sulphur according to Hassall, the following will be the percentages :

Myronate of potash	0.61	per cent.
Sulphocyanide of sinapine	10.71	"
Myrosine	28.52	"

In this case, the results obtained by calculation from the percentages of nitrogen and sulphur, are almost identical with those obtained by direct determinations. But the greater ease and certainty of the direct method, and the very considerable errors which are possible when calculations are instituted upon results differing but very slightly from the true ones, must strongly recommend, it appears to us, the direct as compared with the indirect method.

If the mustard is adulterated with starch or flour, the foregoing scheme of analysis may be used as well in the case of pure mustard. After the extraction of the oil with ether, and the sulphocyanide of sinapine and myronate of potash with alcohol, the residue may be treated either with malt extract or with acids under pressure to convert the starch into glucose. The glucose may be estimated as usual.

Fearing that starch was not entirely insoluble in dilute alcohol, and not being able to learn anything on the subject from books, an experiment was made of boiling starch for some time in a mixture of half alcohol and half water. The boiling liquid was filtered hot, and to the filtrate a few drops of iodine solution added. No blue coloration taking place, the starch may be considered insoluble in dilute alcohol, and hence could not interfere in the determination of the sulphocyanide of sinapine and myronate of potash.

XXXIV.—Upon the Compounds of the Aromatic Bases with Metallic Salts, with a Note upon Thiocarbanilide.

By Albert R. Leeds.

Introduction.

The author was led to a study of these compounds by the following considerations :—

I. The energy, in many cases attended by the evolution of much heat, with which a number of them are formed.

II. The hope that a study of the structural formulæ of compounds of so considerable a degree of complexity, and containing both metallic and non-metallic basic radicals in combination with acid radicals, might be attended with theoretic interest.

III. The expectation, which, indeed, first suggested the inquiry, that by removal or replacement of the metallic element in these compounds by reactions, induced by organic compounds of sulphur and related substances, these metallo-aromatic bodies would serve as material in the institution of an interesting line of research. This