

BENZYLMETHYLANILINE.

(Preliminary Notice.)

By JAMES H. STEBBINS, JR.

This compound was prepared as follows :

One part of methylaniline and 2 parts of benzylchloride were heated, with return cooler, to boiling, for four hours. The unaltered benzylchloride was then distilled off, and the resulting mixture fractionated. From 192–210°C. some unaltered methylaniline passed over. The temperature then rose to 317°, and from there shot up to 360° C., so that the thermometer had to be removed. The fraction passing over between 317° and 360° has a pale yellow color, and a pleasant, aromatic smell. It is sparingly soluble in aqueous HCl. It forms a nitroso-compound when treated with Na NO₂. This nitrous body is reduced by zinc dust to a colorless solution, most probably owing to the formation of a paraamido-compound. The NO group must, therefore, be in the benzol nucleus, making the substance, therefore, a tertiary amine. If treated with chloranil in the cold, a deep blue coloring matter is formed, which, upon heating, turns green.

This compound is, therefore, probably benzylmethylaniline, mixed with other substances, the exact nature of which has not yet been determined.

ON A SINGULAR PROCESS OF SUGAR ANALYSIS.

By P. CASAMAJOR.

Some dozen years ago there appeared, in the *Journal des Fabricants de Sucre* a letter signed *Un Abonn *, calling attention to an empirical process of sugar analysis, used by Parisian sugar testers. This communication was the occasion of denials of its truthfulness by several sugar testers, and also of counter-statements by chemists who confirmed the accuracy of the statements of *Un Abonn *.

I have reason to believe that such a process was used pretty generally at that time, because, having occasion, shortly after the appearance of the letter of *Un Abonn * above mentioned, to test several marks of a cargo of raw beet sugar from France, I found

that the tests by the empirical process there given agreed more closely with the tests made in Europe than those obtained by the optical saccharimeter.

This empirical process is called "Process of the $\frac{1}{4}$." To test a sugar by it, the water is determined by evaporation, and the ashes by incineration, after addition of sulphuric acid. These are the only tests actually made. They require a very small outlay for apparatus. The return of the analysis of a sugar is made on a blank of this form :

Sugar.....
Glucose
Ashes.....
Water
Organic matters, not saccharine, and loss.....
Total	100

To fill up this blank, the water and the ashes are first put down as found by actual tests. The other constituents are obtained by what may be called *guessing*, within prescribed rules. To get at "organic matters, not saccharine, and loss," the sugar tester takes $\frac{1}{4}$ of the quantity of ashes as determined by actual test, and hence the name of " $\frac{1}{4}$ process." After which there only remain "Sugar" and "Glucose" to fill up, and these are easily got. Beet sugars contain very little glucose (invert sugar), so no serious mistake can occur in guessing at this. The sugar tester, however, is guided by the color of the sugar. If this is dark, compared to most beet sugars, the glucose is estimated at about $\frac{3}{4}$ of 1 per cent. If the sugar is light glucose is put down at about $\frac{1}{10}$ of 1 per cent. Intermediate colors give intermediate percentages of glucose. As the quantity is never very great no important mistake can be made in the item of glucose.

As to the item which figures as "sugar" it is very easily obtained by adding up all the others and subtracting the sum from 100.

Take, as a numerical example, a beet sugar, having 3 per cent. of water and $2\frac{1}{2}$ per cent. of ashes. According to what has been said, the analysis would be given as follows, supposing that the

color of the sample justified guessing that glucose was equal to 0.3 per cent.

Sugar	92.20
Glucose	0.30
Ashes	2.50
Organic matter, not saccharine and loss	2.00
Water	3.00
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Total	100.00

To understand how such a process could ever have come into use, we must remember that most raw beet sugars are of very high grade compared to the generality of raw cane sugars. After deducting water and real sugar, as ascertained by actual analysis, the remainder is comparatively a small percentage. If now the ashes are found generally to be about $\frac{1}{5}$ of the total remainder, it is conceivable that a process like this may give in most cases results which are satisfactory to the buyer and to the seller.

But there remains something else to be considered in relation to this process: It is that beet sugars are sold on the basis of *rendement*, formed on the assumption that 1 per cent. of ashes prevent the crystallization of 5 per cent. of sugar. Now, in the numerical example given, the sugar being stated at 92.20, we would have to deduct $2\frac{1}{2} \times 5 = 12.50$ from 92.20, to obtain, as *rendement*, 79.70 per cent. In doing this we have actually done the same thing as taking the water (equal to 3 p. c.) and glucose (0.30) from 100, which leaves 96.7. Now we take the ashes ($= 2\frac{1}{2}$), add $\frac{1}{5}$ as much and get $2.50 + 2. = 4.50$. We now multiply the ashes by 5 (2.50×5) and get 12.50, which added to 12.50 makes a sum equal to 17. This, deducted from 96.70, leaves as *rendement* 79.70, the number already given. To obtain it we have deducted from 100, the following quantities:

Percentage of water, as determined	3.00
“ “ glucose, guessed at30
“ “ ashes multiplied by	
* (2.50×6.8)	17.00
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	20.30

The percentage of ashes is incomparably the most important element in a sugar analysis by this process.

In the way that a chemist is expected to make a sugar analysis, he is to determine the sugar by the optical saccharimeter, the water by evaporation and the ashes by incineration.

Indeed the water is of no importance and may be left out of the ordinary commercial analysis. From the sugar as given by the saccharimeter, the sugar tester deducts 5 times the weight of the ashes. The result is the *rendement*. If the sugar should be found to be 92 p. c. and the ashes 2.50, the *rendement* would be $92 - (2.5 \times 5) = 79$ p. c. It is on this number, 79, that the sale of beet sugar is based in all European markets, which is a fact of itself more extraordinary to me than the $\frac{3}{4}$ process. The "coefficient 5," which is generally accepted, is based on nothing but assumptions which have no foundations. Those who are willing to buy and sell by the "coefficient 5" need find no fault with the $\frac{3}{4}$ process.

My attention has been called to a description of the $\frac{3}{4}$ process in Wurtz, *Dictionnaire de Chimie*, III., p. 67. After describing Clerget's process, the author of the article says: "Depuis un certain temps, pour éviter un dosage aussi long, on employe, dans les laboratoires, un procédé beaucoup plus simple et qui donne des résultats à peu près exacts." * * * Then follows a brief description of the $\frac{3}{4}$ process.

THE ESTIMATION OF MORPHINE IN OPIUM.

BY J. HOWARD WAINWRIGHT, PH. B.

As one of the chemists connected with the United States Laboratory at the port of New York, my attention has been in a large measure directed to the assaying of opium, samples of which, from every case entered here, are sent to this laboratory for the estimation of morphine.

The literature upon this subject is very voluminous, probably more so than that relating to the assay of any other drug, and comprises the descriptions of many methods.

The requirements of a method adapted to the work of this Laboratory are, rapidity of manipulation, simplicity of the apparatus and, most important of all, accuracy of results. I have, therefore, undertaken an investigation of this very interesting subject, the purpose of which has been, not to test all of the methods published, but simply to try some of the most general and to compare them in order to find one, or the essential features of one, which best meets the above demands.

There are not many different varieties of opium imported at this port, by far the largest amount being Smyrna or Turkish, occasion-