

Strong sulphuric acid dissolves it, with a fine blue color. Reducing agents decolorize the solution.

Azobenzole-sulphocresol.



If an alkaline solution of cresolsulpho-acid be treated with an aqueous solution of diazobenzole nitrate, the result is a deep orange colored, oily liquid. If this be allowed to stand for a short time, and then be treated with an excess of hydrochloric acid, the acid, by this means set free, will, after a short time, crystallize in long, brown needles, with strong metallic lustre. These are quite soluble in alcohol—less so in hot water—to which they impart a fine orange color.

I am now working on the higher diazo-compounds, and have already obtained a series of new colors, far surpassing the previous ones in beauty and durability, and will, on some future occasion, lay before you the results of my work.

XXXV.—ACTION OF BONE BLACK ON SOLUTIONS OF PURE SUGAR.

By P. CASAMAJOR.

In a paper published in the *American Chemist*, for November, 1871, "On the Purification of Sugar Solutions for the Optical Saccharometer," I gave an account of experiments made with dry bone black on sugar solutions. These experiments led me to differ from the conclusion to which Dr. Schiebler had arrived, that bone black absorbs sugar from its solutions, and that, therefore, the use of bone black, even when thoroughly dried, tends to give results with the saccharometer that are lower than they would be without the use of bone black.

My attention was called to this subject again during the latter part of 1878, by several communications of our regretted fellow member, Professor J. M. Merriek, of Boston, to the *Chemical News*. In these communications Professor Merriek cites many authorities who agree in this; that the use of bone black, in tests by the optical saccharometer, tends to give results that are too low. In addition to citing these authorities, he gave experiments, made by himself, which confirm the opinions of the authorities cited by him. He found, in several cases, that when using bone black, the results were from 0.5 to 0.8 per cent. lower than they were previous to using bone black. Professor Merriek wrote to me at the time, calling my attention to these results, and I answered that, being very busy with other things just then, I had not time to go over the subject.

Since that time, during February of this year, I made some experiments to discover whether bone black really absorbed pure sugar from its solutions. In these experiments, I made use, in one case, of a solution of pure sugar, and, in another case, of a solution of good loaf sugar. The pure sugar was obtained by soaking good loaf sugar in alcohol for over twenty-four hours, washing the crystal with more alcohol, and, finally, drying the sugar over a water bath. The object of using pure sugar, or nearly pure, was to eliminate errors which might result from the unequal absorption by bone black of the impurities that exist in commercial sugars. These impurities are of many kinds, but it generally happens, with sugar of high grade, that the impurities are in such a condition that they exert no action on polarized light, so that, on making the correction after inversion, the corrected result is almost always the same as the direct test, when we operate with sugars of 90 per cent. or over.

There being a possibility that the effect of bone black, in lowering the saccharometric degree, was due principally to its power of removing dextro-gyrate impurities in greater quantity than the lævo-gyrate, the use of pure sugar would eliminate any error from this cause.

The experiments were conducted in such a way as to leave no doubt on the question whether bone black took up sugar from its solutions. A weight of bone black was used, which was many times greater than the weight of sugar that filtered over it.

First experiment.—This was made with 125 c.c. of recently and thoroughly dried new bone black. These 125 c.c. weighed 79 grms. The bone black was not washed to remove the salts.

The solution of pure sugar that was filtered over these 79 grms of bone black had a specific gravity of $1.052 = 12.75^\circ$ Balling. In the optical saccharometer it stood at 51.5 per cent. These numbers give a coefficient of purity = 100. A volume equal to 80 c.c. was allowed to run out, the operation taking one hour. This portion that came through had a specific gravity of $1.057 = 13.05^\circ$ Balling. In the optical saccharometer it stood at 49 per cent. These numbers give a coefficient of purity equal to 92.8 per cent.

This fall in the coefficient of purity from 100 to 92.8 cannot be due to absorption of sugar, although the fall in the saccharometric indication from 51.5 to 49 per cent., shows that sugar has been removed from the solution. The decrease in the coefficient of purity can only come from the impurities present in new bone black, and points to the necessity of washing new bone black before passing sugar solutions over it.

As the quantity of bone black was very large in proportion to the weight of sugar that filtered over it, the quantity of sugar removed from the solution may be taken as being very nearly the quantity which 79 grms of new bone black can absorb. To ascertain this quantity of sugar, let us take 80 c.c. of the solution, such as it went on the bone black, its specific gravity being 1.052, the weight of the solution was $80 \times 1.052 = 84.16$ grms.

The degree Balling being 12.75, and the coefficient of purity being 100, the whole weight of sugar in the 84.16 grms of solution was $0.8416 \times 12.75 = 10.73$ grms of pure sugar.

The 80 c.c. of solution which filtered through the 79 grms of bone black in the space of an hour, had a specific gravity of 1.057, which gives for the 80 c.c. of solution a weight of $80 \times 1.057 = 84.56$ grms. The degree Balling of these 84.56 grms being 13.05, and the coefficient of purity 92.8, the quantity of pure sugar in the filtered 80 c.c. is $= 0.8456 \times 13.05 \times 0.925 = 10.25$ grms.

Subtracting this from the quantity of sugar previously obtained, we have $10.73 - 10.25 = 0.48$ grms as the weight of sugar absorbed by 79 grms of new bone black, which is equivalent to 0.606 gm, as the quantity of sugar absorbed by 100 grms of new bone black.

Second experiment.—This experiment closely resembles the first. The bone black used was the ordinary bone black of the refinery. The quantity taken was 125 c.c., weighing 100 grms. The bone black was thoroughly dried, as in the first instance.

The solution of loaf sugar filtered over these 100 grms of old bone black had a specific gravity of 1.051 $= 11.5^\circ$ Balling. In the optical saccharometer it stood at 46 per cent. These numbers give a coefficient of purity equal to 99.5.

As in the previous case, a volume equal to 80 c.c. was allowed to run through the filter in the course of one hour.

The portion that came through had a specific gravity of 1.044 $= 10.95^\circ$ Balling. In the optical saccharometer it stood at 43.3 per cent., which gives a coefficient of purity equal to 98.6.

We may notice here at once that the fall in the coefficient of purity is insignificant when compared to what happened with new bone black. Still it points to impurities absorbed by the old bone black.

If we go over the same calculations as in the first experiment, we find that the weight of the 80 c.c. of solution was $80 \times 1.051 = 84.08$ grms. The total number of grms of sugar in 80 c.c. $= 11.5 \times 0.8408 \times 0.996 = 9.63$ grms.

The 80 c.c., after filtering over 100 grms of old bone black, weighed $80 \times 1.044 = 83.52$ grms, and the total weight of pure sugar in the filtered portion was : $10.95 \times 0.8352 \times 0.986 = 9.01$ grms.

If we subtract this quantity from 9.63, belonging to the 80 c.c., before going on the bone black, we have $9.63 - 9.01 = 0.62$ gm as the weight of sugar absorbed by 100 grms of old refinery black.

CONCLUSIONS.

These numbers : 0.606 gm for the quantity of pure sugar absorbed by 100 grms of new black, and 0.63 gm for the quantity of pure sugar absorbed by 100 grms of old black, are remarkably close, particularly if we consider the degree of approximation compatible with sugar analysis.

The quantity of sugar absorbed by 100 grms of bone black may be put down as 0.6 gm. It is possible that new bone black may generally absorb slightly more or less sugar than the old, but this point can only be settled by a great number of tests.

To return to the action of bone black in sugar tests, there seems little doubt that, in many cases, the addition of bone black seems to lower the saccharometric test. This action must, in part, be due to the comparatively greater absorption of the dextro-gyrate impurities that are found in commercial sugar.

As to the absorbing action of bone black on sugar, if, as the above experiments show, this absorption is about 0.006 of the weight of the black employed, there must be no perceptible error in shaking a small quantity of fine bone black with the solution previous to filtration. A quantity of black equal to 4.5 grms, shaken with 100 c.c. of sugar solution, will almost always produce a marked improvement in the color of the solution. If this quantity of bone black absorbs 0.006 of its weight of sugar, this absorption will be equal to $0.006 \times 4.5 = 0.027$ gm. Now this quantity divided by 26.048 grms (the quantity dissolved in 100 c.c. of solution) will give $\frac{0.027}{26.048}$, which is very approximately $= 0.001$, or $\frac{1}{10}$ of 1 per cent., and this is the error which $4\frac{1}{2}$ grms of bone black may produce from its property of absorbing pure sugar. By absorbing dextro-gyrate impurities a greater error may arise, but I regret to say that I have not tried to solve this problem, which is well worthy the attention of chemists.*

* Dr. Schibler has called attention to the error, committed in saccharometric tests, by using basic acetate of lead. As the precipitate obtained, occupies a certain volume, this volume must be deducted from the 100 c.c. measured by the graduated flask. By using a volume of liquid smaller than 100 c.c., the result obtained is necessarily too high. This being the case, a sufficient quantity of bone black, shaken up with the solution, may act as a corrective by lowering the saccharometric degree. If there was any constancy in the effect of acetate of lead in raising the saccharometric degree, and of bone black in lowering the test, it would be possible to neutralize a certain volume of basic acetate by a corresponding weight of bone black. Unfortunately, if I am not mistaken, experiments are wanting to elucidate this subject.