

The following papers by H. A. Mott, Jr., were announced for the next meeting: 1. "Absorption of Sugar by Bone Black." 2. "Practical Determination of the Sugar of Commerce."

The meeting adjourned.

P. CASAMAJOR,

Recording Secretary, pro tem.

XXXIV.—SOME NEW AZO-COLORS.

BY JAMES H. STEBBINS, JR., B. S.

When I first began the study of the azo-compounds, I selected as my starting point the action of diazobenzole upon the amines, amides, and phenolls, reserving the privilege of experimenting with the higher diazo-compounds at some future period.

The following compounds are the results of my experiments:

Azobenzole-trinitrooxybenzole.— $C_6H_5N=NC_6H(NO_2)_3OH$.

This compound was obtained by treating an alcoholic solution of picric acid with an aqueous solution of one molecule of diazobenzole nitrate.

After standing a short time the liquid became filled with long, brown needles. These had then to be filtered rapidly, as only a short contact with the mother liquor was sufficient to decompose them. They were then washed several times with cold alcohol, and dried in the air pump, over sulphuric acid.

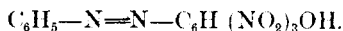
When dry, they appear as long, brown, prismatic needles, with a strong metallic lustre. They are very explosive, a temperature of about 70° C. being sufficient to explode them. The melting point could not be obtained, owing to this circumstance.

They are insoluble in cold, but slightly soluble in boiling water, under partial decomposition. On the other hand, they are readily dissolved in warm alcohol, with a pretty yellow color.

The analysis of the above compound leads to the following result:

THEORY.		FOUND.
C ₁₂	144.... 43.21	43.33
H ₇	7.... 2.10	2.59
N ₅	70.... 21.02	20.93
O ₇	112.... 33.67	—
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	333 100.00	

These figures prove the correctness of the assumed formula, viz. :



Azobenzole-pyrogallol.— $C_6H_5-N=N-C_6H_2(OH)_3$.

This was obtained by treating an alkaline solution of pyrogallol, with an aqueous solution of 1 mol. of diazobenzole nitrate. Immediately a brick-red precipitate is formed, which increases on standing.

It was then collected on a filter, washed with a little alcohol, and dried.

It dissolves in glacial acetic acid and nitrobenzole, from which it crystallizes in dark, red-brown needles. An alcoholic solution of these dyes silk and wool of a pretty old-gold color, without mordants.

The analyses gave the following results :

THEORY.		FOUND.	
Anal. 1.		Anal. 2.	
C ₁₂	144 62.60	62.36	62.28
H ₁₀	10 4.34	5.23	4.64
N ₂	28 20.86	—	—
O ₃	48 12.20	—	—
	<hr/> 230 100.00	<hr/>	<hr/>

These figures evidently lead to the following formula :



Azobenzole-oxycarboxylbenzole.— $C_6H_5-N=N-C_6H_3\begin{matrix} \diagup OH \\ \diagdown COOH \end{matrix}$.

This pretty orange dye was obtained by the action of diazobenzole nitrate, on an alkaline solution of salicylic acid.

It crystallizes in orange-red needles, which are insoluble in water, but easily soluble in alcohol or ether.

If this compound be treated with strong sulphuric acid it dissolves, forming a sulpho-acid which, to me, has all the appearances of the compound obtained by P. Griess from diazosulphanilic and salicylic acids.

Azosulphoxylbenzole-phloroglucin.

This was produced by the action of diazosulphanilic acid on an alkaline solution of phloroglucin.

A heavy orange precipitate is thus formed, which is collected on a filter, dissolved in water, and precipitated as a soda salt, by means of common salt. The compound thus obtained has the appearance of an orange crystalline powder, and is easily soluble in water. The free acid may be obtained by treating a concentrated aqueous solution of

the soda salt with an excess of strong hydrochloric acid. It crystallizes in thin, orange tablets, with a beetle-green lustre.

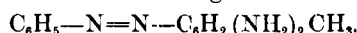
Animal fibre is dyed a fine orange with these.

Azobenzole-diamidotoluole Nitrate.

This pretty orange dye stuff is obtained by treating an aqueous solution of diazobenzole nitrate with a solution of alpha toluylendiamine, m. pt., 99°.

Immediately the mixture assumes an orange-red color, and, after an hour's standing, the liquid becomes filled with fine, red needles. These are then thrown on a filter and allowed to drain; they are then dissolved in boiling water, and the base is set free with ammonia, in the form of a yellow colored, crystalline precipitate. These crystals are then washed, and dried at 100°.

The analysis leads to the following formula:



C ₁₃	68.35 per cent.
H ₁₄	7.61 “
N ₄	24.60 “

The base unites readily with acids to form salts; but the best form of salt is the chloride, as it is by far the most easily soluble and convenient to handle.

If a concentrated solution of the chloride be treated with an aqueous solution of zinc chloride, a heavy, crystalline, orange-red precipitate of the zinc double salt is formed.

Platinic chloride produces a precipitate of beautiful crimson needles, which constitute the platinum salt.

If this dye be dissolved in strong H₂SO₄, and heated for a short time, and then thrown in cold water, a heavy precipitate is formed, which constitutes the sulpho-acid.

This dissolves in water readily, but has a browner tinge than the previous dye.

Reducing agents decolorize the solution, regenerating toluylendiamine.

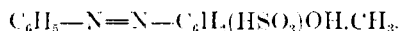
Diamidoazonaphthalene Hydrochlorate.



I obtained this brown dye by the action of diazonaphthalene hydrochlorate in aqueous solution, on a solution of naphthalene diamine dissolved in alcohol. If a little of the solution be evaporated, the coloring matter will crystallize out in long, brown needles. These are insoluble in water, but quite readily soluble in alcohol.

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. Schiedler 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. Merrick, of Boston, to the *Chemical News*. In these communications Professor Merrick 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 Merrick 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.