

allow of any determination of the conductivity, though this would be an interesting point to examine, in view of the high conducting power of the commercial metal, and the probability that the value would be notably raised by the removal of impurities. An imperfect attempt was made to measure the expansion by change of temperature, using for the purpose determinations of specific gravity in water at widely different temperatures, and correcting for expansion of water and glass, but the quantity of material was too small to make the result trustworthy.

It was also attempted to estimate roughly the fusibility, by placing equally heavy beads of the pure and the commercial aluminum in front of a fine blowpipe jet, this jet being fixed in position and fed with air by a steady water blast. From the difference of distance at which the beads could be just fused it seemed probable that the pure metal was a little less fusible than that of commercial character.

Under the same conditions of heating the former seemed to oxidize on the surface with rather more ease than the latter. On the other hand, the pure metal seemed to present perceptibly greater resistance to the prolonged action of solvents—acids and alkalies—than the impure.

UNIV. OF VIRGINIA,

*July 15, 1882.*

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CONTRIBUTIONS FROM THE LABORATORIES OF THE  
SCHOOL OF MINES, COLUMBIA COLLEGE.

XXXI.—ON NITRO-SACCHAROSE.

By ARTHUR H. ELLIOTT, Ph. B., F, C. S.

This curious body has already been investigated by Sobrero in 1847,\* Schoenbein,† Ad & W. Knop‡ and others; and no definite conclusions as to its constitution appear to have been brought out by their labors. Having to make some experiments upon this body I thought it would not be uninteresting to give an account of some results which I obtained in the course of my work. These experiments include the action of a number of solvents, the action

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\* Comptes Rendus Vol. 24, p. 247.

† Pogg. Ann. Vol. 70, p. 100.

‡ Jour. für prak. Chem. Vol. 56, p. 334.

of ammonium sulphhydrate, and also the action of a strong solution of ferrous chloride.

In the preparation of nitrosaccharose, pure crystals of cane sugar were used and reduced to a powder fine enough to pass through a sieve with 100 holes to the linear inch, after which it was dried in a water-bath. A number of methods were used to nitrate the sugar, and a brief description of these may prove useful. In the first case the following method was used. 50 grms. of sulphuric acid (sp. gr. 1.84) and 25 grms. of nitric acid (sp. gr. 1.53) were mixed together, and the mixture cooled to 15° C.—25 grms. of the finely powdered sugar were now slowly stirred into the acid mixture, the temperature rose to 30° C., the mixture being constantly stirred for 15 minutes. The clot thus formed was thrown into cold water (about 1 litre) and the acid mixture was also added. The nitrosaccharose thus formed was collected together into one mass, and after washing and squeezing in plenty of cold water it weighed 24.4 grms.

The material thus obtained was a soft, silky looking, wax-like body, plastic, and adhering to the fingers when removed from water. At 30° C. it is soft enough to flow like melted pitch. It could not be washed free from acid. When heated on a water-bath it melted and began to decompose giving off red fumes. An attempt was made to neutralize it with potassic carbonate, but the action was very energetic and most of the material dissolved in water.

Another lot of nitrosaccharose was made using 25 grms. of sugar 400 grms. of sulphuric acid (sp. gr. 1.84) and 150 grms. of nitric acid (sp. gr. 1.53) following the same method of adding the sugar as mentioned before. The mixture had a temperature of 15° C. before adding the sugar and rose 10° C. during the experiment. In this case the acid mixture containing the clot of nitrosaccharose was poured into cold water and the mixture of acids and water became so hot that red fumes were evolved. The resulting mass after washing and squeezing weighed only 22 grms. The mass was melted on a water-bath under water, and while in this condition potassinn carbonate was added and the whole well stirred. It was then washed from excess of alkalis and allowed to stand in water several days, changing the water daily. After a time it began to give an acid reaction, showing decomposition. It was washed with a little alcohol and allowed to dry.

A third lot of nitrosaccharose was made, using the same acids and sugar in the following proportions: sugar, 22 grms., sulphuric

acid, 176 grms., nitric acid, 66 grms. The sugar was added as usual, and the resulting mixture was poured into water in small portions, cooling the water after each addition; the nitrosaccharose thus obtained after washing and squeezing weighed 27 grms. It was washed with water and a weak solution of potassic carbonate.

The rest of the treatment was exactly like that followed in the second case.

Yet another quantity of nitrosaccharose was made, using a large quantity of sugar. Eight hundred grms. of sulphuric acid (sp. gr. 1.84), and 300 grms. of nitric acid (sp. gr. 1.53), were used with 95 grms. of sugar. When half the sugar had been added to the acid mixture, the whole was cooled, the rest of the sugar added and the completed mixture allowed to stand one hour with frequent stirring. The acids were now poured off the nitrosaccharose, and the latter, when washed and kneaded thoroughly with water, weighed 135 grms. When the mass was first removed from the acid mixture it had the consistence of soft butter, but as the washing and squeezing progressed it became more and more resinous and white. A further kneading in water hot enough to bear the hand in it, using two lots of about one litre each, and finally in water containing one per cent. of potassic carbonate, reduced the well-squeezed mass to 109 grms.

This last method appears to give the best result in the matter of washing, but the material slowly decomposes, the water in which it is kept becomes acid and smells faintly of hydrocyanic acid. Nevertheless the nitrosaccharose made by either of the methods changes so slowly that I have kept it under water for over a year without any great change in appearance or bulk, beyond the acid reaction mentioned above.

*Action of Solvents on Nitrosaccharose.*—The following solvents were used upon nitrosaccharose, both at ordinary temperature and also by heating in a water-bath:

Water—*Cold*, soluble; *hot*, soluble.

Absolute alcohol—*Cold* and *hot*, soluble.

Alcohol, 93 per cent.—Same as absolute.

Alcohol, 80 per cent.—*Cold*, slowly soluble; *hot*, soluble.

Alcohol, 50 per cent.—*Cold*, insoluble; *hot*, soluble.

Ether—*Cold* and *hot*, soluble.

Chloroform—*Cold* and *hot*, soluble.

Benzole—*Cold*, insoluble; *hot*, slowly soluble.

Carbon disulphide—*Cold*, insoluble; *hot*, insoluble.

- Sulphuric acid (sp. gr. 1.84)—*Cold* and *hot*, soluble.  
 Acetic acid (glacial)—*Cold* and *hot*, soluble.  
 Turpentine—*Cold* and *hot*, insoluble.  
 Wood naphtha—*Cold* and *hot*, soluble.  
 Amylic alcohol—*Cold*, partly soluble; *hot*, soluble.  
 Carbolic acid—*Cold*, partly soluble; *hot*, soluble.  
 Nitrobenzole—*Cold* and *hot*, soluble.  
 Tolnol—*Cold*, insoluble; *hot*, partly soluble.  
 Petroleum naphtha ( $\hat{\gamma}1^{\circ}$ – $\hat{\gamma}6^{\circ}$  B'e.—*Cold* and *hot*, insoluble.  
 Astral oil—*Cold* and *hot*, insoluble.  
 Mineral sperm—*Cold* and *hot*, insoluble.  
 Olive oil—*Cold*, insoluble; *hot*, soluble.  
 Sperm oil—*Cold*, insoluble; *hot*, soluble.  
 Potassic hydrate (1 in 10)—*Cold*, insoluble; *hot*, partly soluble.  
 Ammonia hydrate—*Cold*, insoluble; *hot*, partly soluble.  
 Nitric acid (conc.)—*Cold* and *hot*, soluble.  
 Hydrochloric acid (conc.)—*Cold*, partly soluble; *hot*, slowly soluble.  
 Stannous chloride, decomposes it.  
 Ferrous sulphate, decomposes it.  
 Ferrous chloride, decomposes it.  
 Ammonia sulphhydrate, decomposes it.  
 Borax (5 per cent. solution — *Cold* and *hot*, insoluble.  
 Acetic ether—*Cold* and *hot*, soluble.  
 Acetone—*Cold* and *hot*, soluble.  
 Glycerine—*Cold*, insoluble; *hot*, soluble.  
 Nitroglycerine—*Cold*, slowly soluble; *hot*, readily soluble.

While trying the action of these solvents the following facts were noticed. When heated with water it decomposes as it dissolves. On heating with turpentine a slightly yellow fluid is obtained. Carbolic acid is reddened, both cold and hot. Tolnol, both cold and hot, gives a red color. With olive oil mutual decomposition takes place on heating. Sperm oil acts like olive oil. Potassic hydrate gives a brown solution when heated. Ammonic hydrate, even in the cold gives a yellowish-brown color. Nitric acid, decomposes it on heating giving red fumes. Hydrochloric acid, both cold and hot decomposes nitrosaccharose. Stannous chloride is decomposed giving a white precipitate, even in the cold. Ferrous sulphate, also chloride, decomposes nitrosaccharose, even in the cold, giving brown precipitates. Ammonic sulphhydrate, both hot and cold, decomposes this body, giving a liver colored solution, with separation of sulphur.

*Action of Ferrous Chloride upon Nitrosaccharose.*—Solution of ferrous chloride was prepared 10 c.c. of which contained 1.4 grms of iron in the ferrous state. Nitrosaccharose was dissolved in 93 per cent. alcohol and the solution mixed with ferrous chloride solution mentioned above. After heating on a water-bath excess of potassichydrate was added, the solution filtered and the precipitate washed. Two samples of this solution were taken; one was used to estimate glucose direct with Fehling's solution; the other was heated with acid to invert the sugar, and then, after neutralizing, the glucose was determined as in the first case. They both gave 11.7 per cent. of glucose on the original amount of nitrosaccharose taken, showing that, as might have been expected, heating with ferrous chloride solution inverts all the sugar in nitrosaccharose. This action should be compared with the reducing action of ammoniac sulphhydrate mentioned further on. It should be mentioned here that the ferrous chloride contained no free acid, since it was made by adding a large excess of iron to hydrochloric acid and heating till action ceased, a large quantity of iron remaining undissolved which was left in the solution to keep it saturated.

To determine the amount of oxidation produced by the nitrosaccharose upon the ferrous chloride, several experiments were made. In the first case the nitrosaccharose was treated with ferrous chloride and hydrochloric acid in an atmosphere of carbonic acid. The excess of ferrous chloride remaining after the action, was determined with standard solution of potassic dichromate. .705 grm. of nitrosaccharose oxidized .583 grm. of iron, which is equal to 30.6 per cent. of  $\text{NO}_2$  in nitrosaccharose. In another experiment the nitrosaccharose was dissolved in ether and the ether solution was shaken up with pure calcic carbonate. After filtering, the calcic carbonate was washed with ether and the filtrate and washings evaporated to dryness. The residue was washed with water and contained only a trace of calcic nitrate, hence nitrosaccharose contained little free acid. The water-washed residue was now dissolved in ether and treated with ferrous chloride solution and hydrochloric acid on a water bath, the excess of ferrous salt being determined with potassic dichromate as in previous experiment. .572 grm. of nitrosaccharose oxidized .645 grm. of iron which is equal to 41.6 per cent. of  $\text{NO}_2$  in nitrosaccharose. Another experiment in which the treatment with calcic carbonate was omitted and the ether solution treated at once with ferrous chloride and hydrochloric acid, gave the following results: .563 grm. of nitrosaccharose oxi-

dized .633 gm. of iron which is equal to 41.3 per cent. of  $\text{NO}_3$  in nitrosaccharose.

From these experiments it appears that the body under consideration acts as a nitrate and contains but a very small quantity of free acid when thoroughly washed and squeezed. In treating with ferrous chloride direct without solution in ether, some of the nitrosaccharose is decomposed without oxidizing the iron solution.

*Action of Ammonic Sulphhydrate upon Nitrosaccharose.*—For these experiments the nitrosaccharose was dissolved in 93 per cent. alcohol and treated with ammoniac sulphhydrate, which was made by saturating ammoniac hydrate with sulphuretted hydrogen. In each case a weighed quantity of nitrosaccharose was dissolved in the alcohol, and a large excess of ammoniac sulphhydrate was added to the solution. The mixture was then placed on a water bath and heated till a dry residue was left, which was treated with water, and filtered. The insoluble matter was examined and found to be sulphur. The solution was heated directly with Fehling's solution and gave only a trace of cuprous oxide, showing that the sugar had not been inverted during the reduction. This fact was established by several such experiments. Several experiments were now made by reducing the nitrosaccharose in alcoholic solution with ammoniac sulphhydrate, as before stated, and then inverting the sugar by heating two hours with a little sulphuric acid. By this means the nitrosaccharose gave 28.4, 28.7, 26.5, 24.9 per cent. of cane sugar. The first and second results are probably correct, as the third and fourth results gave solutions which were colored, apparently from formation of caramel from over-heating. The acid solution obtained by pouring the mixture of sulphuric and nitric acids, after treating the sugar, in water, was kept and a special examination was made for oxalic acid. For this purpose 50 c.c. of the diluted solution were neutralized with ammoniac hydrate and solution of calcic chloride added. After a time a precipitate was formed which proved to be calcic sulphate, but no calcic oxalate could be detected in it.

From a consideration of the foregoing experiments the following conclusions may be drawn: Ferrous chloride in presence of hydrochloric acid reduces nitrosaccharose and converts the sugar into glucose. Ammonic sulphhydrate reduces nitrosaccharose without inverting the sugar, or only to a very slight extent. In regard to the experimental determination of sugar and  $\text{NO}_3$  in nitrosaccharose: if we take the percentage of sugar found and add it to the

percentage of  $\text{NO}_3$  found by oxidizing ferrous chloride, we obtain the following results: The average of the first and second determinations of sugar by reducing with ammoniac sulphhydrate (28.4 and 28.7) gives 28.55 per cent. of cane sugar in nitrosaccharose. The average of two determinations of  $\text{NO}_3$  in nitrosaccharose (41.6 and 41.3) gives 41.45 per cent. of  $\text{NO}_3$  in that body. Adding these together we have 70.00 per cent. as a total; the balance of the percentage consists of water, some free acids, and probably other compounds resulting from the action of the acids upon the sugar. Calculating the determined constituents to per cent., we have cane sugar, 40.78, to  $\text{NO}_3$  59.21, or a ratio of 1 molecule of cane sugar to 8 molecules of  $\text{NO}_3$  (exactly 1 to 8.009). From these figures it would appear that nitrosaccharose is formed by replacing eight atoms of hydrogen in a molecule of sugar by eight molecules of  $\text{NO}_3$ , giving the formula  $\text{C}_{12} \text{H}_{14} (\text{NO}_3)_8 \text{O}_{11}$ . Assuming that this is the correct formula for nitrosaccharose, the theoretical percentage yield of its constituents would be sugar 41.20,  $\text{NO}_3$  59.75. When we compare these figures with those actually obtained above, and remember that the methods used are not capable of great exactness in results, it appears most probable that the above is the true formula for nitrosaccharose.

### XXXII.—NOTES ON BONE OIL.

BY ARTHUR H. ELLIOTT, Ph. B., F. C. S., AND FERDINAND SANDS, A. B., Ph. B.

During the Spring of 1881, Messrs. C. P. Sawyer and W. W. Share, two students of the class of 1881, of the School of Mines, at the suggestion of Prof. C. F. Chandler, undertook to investigate bone oil, and repeat some of the work of Anderson,\* C. G. Williams,† Vohl,‡ Church, and Owens§, Schwanert,|| Unverdorben¶ and Runge.\*\*

\**Phil. Mag.*, (3) vol. xxiii. p. 174; (4) vol. ii. p. 457; (4) vol. ix. pp. 145 and 214. *Trans. Roy. Soc. Edin.*, vol. xvi. p. 4; vol. xx. (2) p. 247; vol. xxi. (1) p. 219; vol. xxi. (4) p. 571.

†*Chem. Soc. Jour.*, (Lon.), vol. vii. p. 97. *Proc. Roy. Soc.*, vol. xiii. p. 305; vol. xii. p. 311.

‡*Jahresb.*, 1859, p. 742.

§*Jahresb.*, 1850, p. 859. *Phil. Mag.*, (4) vol. xx. p. 110.

¶*Am. Ch. Pharm.*, vol. cxx. p. 279.

¶¶*Pogg. Ann.*, vol. xi. pp. 59, 67; vol. viii. pp. 25f, 480.

\*\**Pogg. Ann.*, vol. xxxi. pp. 65, 67. *Chem. Gaz.*, 1855. p. 308.