that its density allows it to float on water; and 3d, that the solution has a density slightly different from that of water. I am not prepared at present to develop these ideas, which would require further experiments to establish.

I will confine myself to the well ascertained fact that a piece of camphor wears away much faster when in motion than when at rest. This being established, it appears more than probable that if a piece of camphor is perfectly free from oily matter it will dissolve in water more readily than if protected by a greasy film. The slightest film of this nature, in contact with camphor, becomes a saturated oily solution of camphor, and if any excess exists, over what will cover the camphor, the greasy film will extend over the surface of the water.

When things are in this condition, if an electrified rod is dipped several times in the water, every immersion will remove a portion of oily film from the surface, until finally the film on each piece of camphor becomes so thin that the water reaches the pieces of camphor, and these immediately become gifted with motion.

ON THE METHODS OF INDIGO TESTING.

By Henry M. Rau, Ph. D.

It appears strange, when the high price of indigo and its large consumption in the industrial arts are considered, that the methods commonly employed in this country for its valuation should, from a scientific standpoint, be so crude and inaccurate.

Taking the average price of the various grades of indigo in the market as a basis for calculation, it may be stated that a single per cent. of indigotine represents, to the consumer, from two to two and one-half cents for each pound of goods purchased.

Under these circumstances it would seem highly desirable to employ tests as accurate as possible, even though these should not be as time-saving as the greater number of commercial tests.

Nevertheless, it is a fact, that the ordinary indigo "analyses" are so wide of reliable results, that guess work might quite as well be substituted for them, and this in face of the fact that we have in the gravimetric determination by the reduction methods, a means for a closer valuation of indigo than can be applied in the case of almost any other dyestuff.

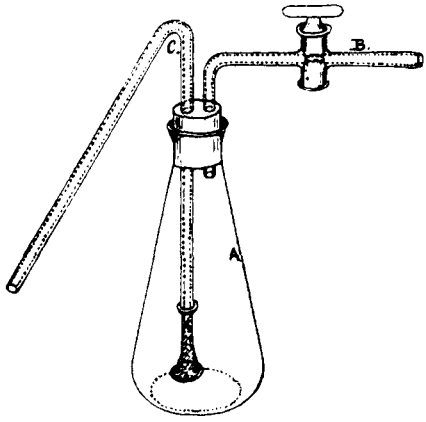
The ordinary mode of judging the quality of an indigo and by which it is commonly bought and sold, is quite superficial; and even in experienced hands allows but a rude estimate of its value. The buyer or broker takes a cake of indigo, breaks it with a knife, and compares it with another of previously known worth as to color, density, hardness, etc. Upon these points of comparison he relies for a correct valuation.

Where a formal analysis is required, the oxidation tests are usually employed; and it is to the inadequacy of these that I particularly desire to call attention. They are based upon the oxidation of the sulpho acid of indigotine in aqueous solution by a variety of agents, with the destruction of the blue color. The different processes in use employ potassium permanganate, potassium bichromate, bleaching powder, or nitric acid, as the case may be. The sample of indigo is dissolved in strong sulphuric acid and the solution brought to a certain volume by the addition of water. A measured quantity is then decolorized with an oxidizing solution which has been standardized upon pure indigotine or some indigo of known value. When the blue color of the indigotine solution has been destroyed and been replaced by a yellow or ochreous shade, the oxidation is complete and the volume of the reagent solution employed, is noted. Now, if indigo contained merely the coloring principle, indigotine, in mixture with inorganic matters, this volumetric estimation would be both practical and reliable, but as its various other ingredients (which are entirely valueless) also enter into the reaction and consume a quantity of the reagent, the method gives these useless matters the same value as the indigotine. The figures obtained are, therefore, always too high, and that in no uniform ratio, but varying with the amount and character of the accompanying impurities. The color which the latter themselves impart to the solution also renders it very difficult to determine exactly when the requisite quantity of the re-agent has been added.

The reason why these methods, although known to be so faulty, have been retained in general practice, is to be found in the high figures obtained by them, which are to the apparent advantage of the dealer. Their scientific appearance also is calculated to impress the unskilled. They are, in reality, anything but scientific. A good Bengal indigo which actually contained 52.5% indigotine, showed

72.5% when tested by the permangate process, a Kurpah of 41.8% (with a larger proportion of organic impurities) by the same test yielded 69.2%. Using potassium bichromate as the oxidizing agent, the figures obtained were nearly the same. In fact, in order to achieve correct results by any of these methods, it would be necessary to determine previously the precise amount of extraneous organic matter accompanying the coloring principle.

I have recently adopted for the analysis of indigo a very convenient modification of Fritsche's reduction test, which is also sufficiently rapid to permit as many as three determinations to be made together within twenty-four hours. I proceed as follows: From 1½ to 2 grammes of the sample, in very fine powder, are carefully weighed and placed in an 8 oz. Erlenmeyer flask (A). The flask is provided with a doubly perforated rubber stopper, through which passes a bent glass tube (B) provided with a stop-cock and reaching just below the stopper, and a second tube (C) of syphon shape which passes nearly to the bottom of the flask and terminates in a small funnel.



In this funnel is placed a wad of glass-wool. Tubes and stoppers must fit air-tight into the flask. The apparatus is accurately tared; from three to four grammes of pure grape sugar in small pieces are then placed in the flask, fifteen to twenty c. c. of a 40%. caustic soda solution, sixty c. c. of water, and sufficient 90%. alcohol (120 c. c.) to bring the whole to about seven fluid ounces, are added. The apparatus is then weighed a second time, so that by deducting the tare of the flask, etc., we obtain the weight of the total contents. A small piece of rubber tubing closed with a pinch cork, is slipped over the syphon tube, the stop-cock is closed, and the flask heated on a water bath for twenty-five to thirty minutes. At short intervals the stop-cock is quickly opened and

closed, to relieve the pressure of the alcohol vapors, the flask being occasionally shaken. The indigo readily dissolves, the liquid assuming a deep yellow color, the insoluble substances settling clearly to the bottom. The solution being completed, the flask is allowed to stand for about an hour, and is then connected with a generator of carbon dioxide gas.

The clear liquid is quickly run off, as far as possible, through the syphon tube, the glass wad retaining any floating particles, and the flask is again weighed quickly, whereby the weight of the liquid thus run off is ascertained. This portion is placed in a beaker glass and a stream of carbon dioxide gas passed through the same for fifteen minutes, causing the indigotine and indirubine to precipitate in crystalline flakes. A current of air is then drawn through, which completes the precipitation. The precipitate, which by this means is obtained in beautiful copper-colored flaky crystals, is collected upon a previously dried and weighed filter, washed repeatedly with boiling water, then with hot dilute hydrochloric acid and finally, with water. Precipitate and filter are then dried at 110% C., and lastly weighed. Deducting the weight of the filter, the indigotine and indirubine contained in that portion of the liquid treated as described, are obtained, and by a simple calculation the percentage contained in the whole sample is ascertained.

The advantages of this process are found in the fact, that the insoluble impurities in the indigo are readily deposited, thereby avoiding the tedious filtration of the liquor, and that the indigotine and indirubine are obtained in a crystalline precipitate which is easily purified by washing.

Schunk has stated (Lit. Phil. Soc., Man. XIV.) that where he employed small quantities of indigo, as compared to the quantities of alcohol, caustic soda and grape sugar, Fritsche's process occasionally yielded him very incorrect results, the indigotine at times not precipitating at all. However this may be, the results which I have obtained by this method have been surprisingly uniform. Thus a sample of artificial indigotine, prepared from propiolic acid, tested 99.96%.; two analyses of pure dry indigotine from indigo showed 99.68%. and 99.74%.; three estimates of a high type Bengal indigo gave 60.35%., 60.78%. and 60.72%.

Few analytical methods can claim greater uniformity than this.