saccharine powder, easily soluble saccharine in gravel form, and saccharine tablets. The latter two preparations contain a small percentage of bicarbonate of soda and are more available for cooking purposes.

The factory—and, so far as I am informed, it is the only one of its kind existing—is at Salbke-Westerhusen, on the Elbe.

Patents have been obtained in most civilized lands, and monopolies of sale (which debar the manufacturers from selling direct) exist in many countries, including the United States. The American agents are J. Movius & Son, New York. Up to the present, however, the American trade has been inconsequential. In 1891, the export to New York was only about 800 kilogrammes (1,800 pounds approximately), while during the same period 7,200 kilogrammes was shipped to England. I understand that the owners would erect a factory in the United States should the state of the trade ever warrant it. ALBERT H. WASHBURN,

United States Commercial Agent.

Magdeburg, September, 1892.

THE MANUFACTURE OF PURE NAPHTHALENE.

NAPHTHALENE was for many years a source of serious trouble to gas managers, and is so to some extent even to-day. It has, however, of late years taken an important place in technical chemistry as the starting point of a series of coloring matters of great importance in the tinctorial industries. It is not that naphthalene is itself capable of yielding colors by any simple, direct treatment, but because—in a manner similar to benzene in the manufacture of aniline—the naphthalene is first converted into a nitro-compound—nitro-naphthalene, which is then reduced to the amine naphthylamine, by the action of nascent hydrogen; or, again, by “sulphonation”—i. e., the action of sulphuric acid of a specific gravity not less than 1.85, whereby naphthalene is converted into alpha-naphthalene-disulphonic acid, which by fusion in a special manner with caustic soda produces the naphthols. It is, therefore, by the preliminary processes of converting naphthalene into substances bearing such formidable titles as alpha-

spheric thermal changes, which would cause the naphthalene to solidify; it is therefore best to cover it with non-conducting composition to prevent blocking. The worm, K, may, of course, be a coil-shaped one, or of any of the forms in vogue in other distilling processes. The illustration shows a form which is in use in America, in which the vapors take a zigzag direction along the pipes, K. These pipes are cast, or made sound with borings to the ends of the water tank, thus avoiding internal joints and bringing leakages to the outside, facilitating inspection and repair. The ends of the pipes, K, and K₂, are joined by cast iron caps, S, fastened on with set screws, and the remaining pipes are similarly joined, thus making a complete worm with all joints outside.

To prevent the exposed ends, S S, becoming too cool and thus being blocked with naphthalene, a sheet iron casing is fastened in the position shown by the dotted lines, and the intervening space filled with sand or iron borings. A thermometer, L, is immersed in the cooling water. The condensed liquid flows down the shoot, M, into a cylindrical receiver, N, provided with a manhole and escape tap, U, a run-off tap, R, a sludge tap, P, fitted flush with the bottom for cleaning purposes, and a steam coil, O, by which the contents are maintained in a liquid condition. This receiver is 7 ft. in diameter and 4 ft. deep. In some cases horizontal cylindrical tanks are used, but they are not so convenient, and in stock taking their contents are not so readily ascertained by direct gauging. The crude naphthalene is charged into the still through the manhole, B, and a small fire is put under the still to liquefy the charge. The still is filled so that the liquid contents reach to the level shown or thereabout, thus leaving boiler room. This would correspond to over five tons of crude naphthalene. A quantity of caustic soda solution at 70° Twaddell corresponding to about 30 lb. of 70 per cent. caustic soda is added, and the distillation proceeded with. Care is taken that the safety valve, F, is in good order, the pockets, F and H, are filled with oil to insure the thermometers responding promptly to varying temperatures, and steam is passed through the pipe, I J, to prevent solidification of naphthalene and consequent blocking, the steam being conducted into the water which surrounds the

spects similar to that shown. Here a quantity of 70° Twaddell caustic soda solution corresponding to about 50 lb. of solid 70 per cent. caustic soda is added, and the naphthalene distilled as before. In this distillation the caustic soda reacts at a temperature less than 160°, and to prevent the reaction reversing itself at a higher temperature, the fire is drawn as soon as the thermometer, E, shows 165° to 170°, and the contents of the still are allowed to settle for one hour, at the end of which time the caustic soda and the fixed impurities are drawn off by opening the 2 in. tap Y in the lid of the mudhole C. The tap is then closed and the distillation proceeded with as in the previous cases, separating “first runnings” and finishing at 235° C. The finished naphthalene in the receiver, N, is run off into galvanized iron cans, which hold 100 lb. each; these are allowed to stand all night to solidify, and when emptied and the lumps broken on the following day, the result is a beautifully white crystalline product—naphthalene of the highest commercial purity, melting point 79.2° to 79.5° C., free from oily matters and suitable for the preparation of naphthylamines, naphthols, and tinctorial products.—*Industries.*

THE CONSTITUTION OF THE ALKALOIDS.*

By ALFRED R. L. DOHME.

DURING the centuries that disease has been combated by drugs and medicines, no person, be he doctor, alchemist, or chemist, until the beginning of the nineteenth century, had any idea as to what the efficacy of any given drug was due. It was at the time when chemistry was receiving its first rays of enlightening sunshine, cast upon it from the scrutinizing and far-seeing brains of a Lavoisier, a Dalton, and a Berzelius; when it was still germinating in the dark confines of a chaotic earth, but just prepared to burst its enveloping shell and blossom into that beautiful and attractive hardy plant, *scientia chemia*. It was in 1806 that the German pharmacist, Serturmer, isolated from opium morphine and meconic acid. No one attached any importance to the discovery, and it was not until 1817 his investigations were republished under the title, “Morphine a New Vegetable Base and Meconic Acid, the Principal Constituents of Opium,” that public attention was aroused. Serturmer pointed out the decided alkaline properties of morphine, characterizing it definitely as a vegetable base very much resembling ammonia. The idea dawned upon the minds of chemists that perhaps other drugs might contain similar bases, and they went to work to test the truth of their surmise. The result was that many such bases were soon isolated, and between the years 1817 and 1835 no less than twenty-five new alkaloids sprang into existence, and in the following order:

1806, morphine, by Serturmer; 1817, narcotine, by Robiquet; 1818, veratrine, by Meissner; 1818, strychnine, by Pelletier and Caventou; 1819, brucine, by Pelletier and Caventou; 1819, piperine, by Oersted; 1819, delphinine, by Brandes; 1820, cinchonine, by Pelletier and Caventou; 1820, quinine, by Pelletier and Caventou; 1820, solanine, by Desfosses; 1824, chelidone, by Godefrey; 1826, corydaline, by Wackenroder; 1826, berberine, by Chevallier and Pelletan; 1827, conicine, by Gieseecke; 1828, nicotine, by Poselt and Reimann; 1829, aricine, by Pelletier and Corriol; 1829, sanguinarine, by Dana; 1832, codeine, by Robiquet; 1832, narceine, by Pelletier; 1833, quinine, by Henry and Delondre; 1833, atropine, by Geiger and Hesse; 1833, hyoscyamine, by Geiger and Hesse; 1833, aconitine, by Geiger and Hesse; 1833, colchicine, by Geiger and Hesse; 1835, pseudomorphine, by Pelletier and Thiboumery; 1835, thebaine, by Pelletier and Thiboumery, etc.

These alkaloids were, in most cases, analyzed by Liebig, Gerhardt, Laurent, and Regnault, and their empirical formulas given, as in the case of morphine, C₁₇H₁₉NO₃, etc.

As in the case of nearly every chemical compound during this early period of chemistry, only the composition was determined, no one troubling himself much about the constitution of compounds. In a short time, however, the fertile brain of Berzelius evolved a theory of the constitution of chemical compounds based upon the electrical conditions and properties of the various elements, generally known as the electro-chemical theory. Other theories followed soon after, accompanied, each and every one of them, by a long and often lively discussion in the journals of the time. Most of these compounds were, however, inorganic, while some of the simpler organic compounds also were considered. No one, however, made an onslaught on the numerous classes of alkaloids, despite their value, importance, and apparent similarity in composition. This is quite natural if we consider that fundamental questions of the constitution of compounds had first to be settled—a firm basis had first to be established before such complex bodies as alkaloids could be attacked. As a result, the simpler bodies, such as alcohol, benzene, ether, chloroform, aniline, etc., were first studied thoroughly and their mutual relation established. Berzelius explained alkaloids as compounds containing ammonia united to an indifferent group, while Liebig explained them by saying they were amidogen compounds, i. e., compounds containing amidogen, NH₂, united to another radical, which latter varied, then, for the various alkaloids; or, in other words, they were all derived from ammonia by having an atom of the latter substituted by some other radical. The first great step in advance in this chapter of the alkaloids was taken in 1848, when Hofmann, the late lamented professor of chemistry in the University of Berlin, in Germany, and Wurtz, in Paris, by their brilliant work upon the substituted ammonias, showed that the latter were all derived from ammonia by the substitution in the latter of the hydrogen atoms by various radicals. They showed conclusively that the three hydrogen atoms of ammonia are all capable of being substituted by other elements and radicals, and that they all bear the same relation to the nitrogen atom. Besides this, they showed the distinction between primary, secondary, and tertiary amines, and especially the fact that all alkaloids were tertiary amines.

In 1846, Anderson, an English chemist, had obtained as a product of the dry distillation of bones an oil which he termed bone oil, containing a number of sub-

* From the *Pharmaceutical Review*, November.

THE MANUFACTURE OF PURE NAPHTHALENE—SECTIONAL ELEVATION OF PLANT.

naphthylamine, beta-naphthylamine, alpha-naphthol, beta-naphthol, mono and tri-sulphonic acids of beta-naphthol, and other polysyllabic substances, that the more immediate color-producing operations are reached.

As will be inferred from the great variety of colors produced, and the great delicacy of the shades, purity in the raw materials used is a *sine qua non*. It is, therefore, at the very outset of prime importance that the naphthalene, the radical of the industry, should be of the highest commercial purity, and it is found in practice that a product having a melting point of 79.5° C., which only shows slight discoloration when dissolved in excess of strong sulphuric acid, and is free from the slightest trace of tar oils, is necessary to secure good results.

The manufacture of this article is conducted as follows: The crude naphthalene which crystallizes from the heavy oils in the fractionation of coal tar is subjected to distillation in the apparatus shown in sectional elevation herewith. In this plant A represents the body of a still, 7 ft. in diameter and 7 ft. deep, with a dished bottom having an inclination toward the mudhole, C, so that the still can be as nearly as possible emptied by a 2 in. tap Y shown in the mudhole cover. The still is provided with a manhole cover of the improved self-locking type, as used on gas retorts, by which rapidity of closing and opening and a tight joint are secured. A delicate pressure gauge, D, and an iron tube or “pocket” to carry the thermometer, E, are also fitted in the top of the still as shown. The still is fitted with a safety valve, F, which is set to blow off at a very slight pressure. The vapor passes from the still head, by the 6 in. pipe, G, to the condensing worm, K.

This pipe, G, has a ½ in. steam pipe, I J, passing down its center, fitted with valves at I and J, and terminating in the cooling water which surrounds the worm, K. A small thermometer, H, is inserted in a pocket at G, and serves as a check to the indications of the thermometer, E, in the still itself. To minimize damage in case of fire, the still and its brick setting are erected under light shedding some 10 ft. or more clear of the building where the condensation takes place, and the pipe, G, is consequently exposed to atmo-

worm, K, so as to maintain a temperature of 80°, and steam is passed through the coil, O. The first runnings consist of water and oily matters, with a little naphthalene, the latter commencing to come over at 210°, and the former gradually ceasing as the distillation proceeds. The stillman takes small samples at the worm end from time to time, and as soon as the aqueous matters have ceased, the condensed products, which up to this time have been run into a receptacle not shown in the illustration, are led by the shoot, M, into the receiver, N, and the distillation carried on until a temperature of 235° is reached, when the distillation finishes and the fire is drawn. The liquid naphthalene is thickened to the consistency of bricklayers' mortar by mixing with ground naphthalene residues from previous operations. The pasty mass is transferred to strong bags and pressed while hot by hydraulic pressure up to three tons per square inch—a well known operation needing no special description. The oily matters are thus expressed, collected, and sometimes used as liquid fuel, and the naphthalene is left as a hard, somewhat gray cake, which, when struck, should give a good ringing sound. This is known as “pressed material,” and each lot is carefully tested in the laboratory before further treatment.

A second distilling apparatus, similar to and in close proximity to the first still, is charged with the pressed material together with about 1 cwt. of “best thirds” commercial sulphur, fire is applied and the distillation conducted as above described. The “first runnings” are kept separate, and together with those from other distillations, worked up with fresh crude material. The same observations as to temperature apply in this distillation as in the former one. In this case, however, a considerable quantity of sulphureted hydrogen gas is evolved, and this is drawn off at the end of the worm through the pipe shown by the dotted lines at V into a box fitted with wire gauze “baffles,” or similar means which arrest the naphthalene unavoidably accompanying the gas. The sulphureted hydrogen passes thence to the chimney or to an oxide of iron absorber. The naphthalene collects in a receiver similar to N, and is maintained in a liquid condition until the following morning, when it is forced by compressed air or “blown over” into a third still in all re-

stances, mostly of a basic character. From this bone oil he isolated, in particular, a basic substance having the formula C_8H_7N , to which he gave the name of pyridine. Some years previously Runge discovered in coal tar, by subjecting it similarly to dry distillation, a base which he named quinoline, and of which he found the formula to be C_8H_7N , but whose relation to pyridine was not discovered until later. Gerhardt had already, in 1842, obtained quinoline as a product of the distillation of strychnine, cinchonine, and quinine with alkalies.

Later on it was found that the same quinoline is obtained when morphine, berberine, and brucine are similarly treated. From this time the intimate relation of nearly all alkaloids with quinoline or pyridine becomes more and more apparent, and chemists began to think that alkaloids were derivatives of these two hydrocarbons. Even the product of oxidation of alkaloids, usually acids, when distilled with lime or when simply heated yielded pyridine or quinoline. In order to verify or disprove the theory that alkaloids are derivatives of pyridine, chemists now applied the principles of synthesis, and by starting with pyridine strove to build up alkaloids. It is not many years ago that this work was done, and that artificial alkaloids, notably coniine, atropine, and piperidine, were made and found to be identical in every way with the naturally occurring alkaloids.

The German chemist Ladenburg, now professor at the University of Breslau, has become especially prominent in this chapter of chemistry, and his successful syntheses of alkaloids well merited the universal admiration they received. It was, of course, necessary to determine exactly the constitution of the alkaloid in question before any successful attempt to make it could be made. For simplicity sake, let us take the case of the first artificially prepared alkaloid, and trace up as closely as possible just how the work was done. The alkaloid is coniine. Hofmann had, in 1884, elucidated the constitution of coniine after a long series of investigations, resulting in the formation of hosts of new compounds, whole classes of them, such as the conyrynes, the coniceines, etc. He showed that coniine was α -propyl piperidine—which, since piperidine was known to be hexahydropyridine, means that coniine is α -propyl-hexahydropyridine, which the two following formulas make plain:

Starting with α -picoline, which is α -methyl pyridine, he treated this with acetic aldehyde (CH_3CHO), and thus formed α -allyl pyridine. On reducing this by means of hydrogen generated by the action of metallic sodium on alcohol containing the α -allyl pyridine in solution, he obtained coniine, as the following equations make plain:

This product was identical with natural coniine in every way, save that it was optically inactive, *i. e.*, did not turn the plane of polarized light when the latter was passed through it. Ladenburg now converted his optically inactive coniine into optically active coniine by taking advantage of the well known fact that the mixture in equal parts of the dextro-rotatory and levo-rotatory compounds produces optically inactive compounds. The problem then resolved itself into the splitting of the inactive coniine into dextro and levo-rotatory coniine. This he did by making a coniine dextro-tartrate, *i. e.*, combining coniine with dextro-rotatory tartaric acid. On carefully and fractionally recrystallizing his salt he obtained two coniine tartrates of different solubility, which he separated. From these he obtained two coniines, one of which was dextro-rotatory coniine, and the other levo-rotatory coniine. Thus we see how, starting with pyridine, Ladenburg obtained a coniine identical in every respect with natural coniine. This was the beginning of the alkaloid era, the era in which we now are, and the era which has already given us the constitution of nicotine, trigonelline, atropine, cocaine, pilocarpine, papaverine, narcotine, cotarine, hydrastine, and berberine. Nicotine was attacked and its constitution elucidated by Cahours and Etard,† and that of trigonelline was similarly elucidated by Jahns‡ and Hantzsch.§

The constitution of atropine we owe to Ladenburg,||

* The α , β , γ , etc., refer to the position of the substituting group with reference to the nitrogen atom; α is the position next to it, β second from it, and γ opposite to it.

† Cahours and Etard, *Comptes Rendus*, '88-97.

‡ Jahns, *Berichte der Deutschen Chem. Gesell.*, xx., p. 2840.

§ Hantzsch, *Berichte der Deutschen Chem. Gesell.*, xix., p. 31.

|| Ladenburg, *Berichte der Deutschen Chem. Gesell.*, xxii., p. 2593.

while Einhorn* showed that cocaine was methyl-benzoyl-ecgonine, and that ecgonine was methyl-tetrahydropyridyl- β -oxypropionic acid. The constitution of pilocarpine we owe to Hardy and Calmels,† that of papaverine to Goldschmidt,‡ and that of narcotine to Wegscheider.§ Hydrastine, the so-called white alkaloid of *Hydrastis canadensis*, owes its formula to the excellent work of Freund,|| and finally berberine, its sister alkaloid, is indebted for its existence as a definitely known compound to W. H. Perkin, Jr.,¶ the young English chemist who has but recently been elected professor of chemistry at Manchester University to succeed the late Professor Schorlemmer. In a later article I propose to trace in detail as far as that is possible the work of Dr. Martin Freund on hydrastine, in order to show exactly how the problem of the determination of the constitution of alkaloids is attacked and successfully finished.

* Einhorn, *Berichte der Deutschen Chem. Gesell.*, xxii., pp. 399, 1495, and 2570.

† Hardy and Calmels, *Comptes Rendus*, 102, 103, 105.

‡ Goldschmidt, *Monatshefte für Chemie*, 4-10.

§ Wegscheider, *Berichte*, xvi., p. 1235.

|| Freund, *Berichte*, 22 and 23.

¶ Perkin, *Jour. Chem. Soc.*, 1890, p. 992.

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