

in an acid solution. The following example gives the quantities to be employed. About 27 kilos. of tetra-methyl-diamido-benzhydrol and 21 kilos. of *m*-methoxy-phenyl-*p*-tolylamine are dissolved in 250 kilos. of alcohol and mixed with 5 kilos. of 33 per cent. hydrochloric acid. After standing for about five hours the mixture is heated for about three hours on the water-bath. The reaction is complete when a test no longer gives a blue colour when boiled with sodium acetate and acetic acid. After distilling off the alcohol, water and soda solution are added till alkaline and the white precipitate of leuco base is filtered off. It can be purified by dissolving in hydrochloric acid and precipitating with an alkali. To obtain the colouring matter, 10 kilos. of the leuco base are dissolved in 250 litres of water and 6 kilos. of hydrochloric acid, cooled with 200 kilos. of ice, and oxidised by quickly adding 10.2 kilos. of a 50 per cent. lead peroxide paste. After stirring for a quarter of an hour 4 kilos. of sodium sulphate are added, the solution filtered, and the colouring matter precipitated from the filtrate by adding salt. The dyestuff so obtained can be sulphonated according to Eng. Pat. 11,275 of 1891. Acid dyestuffs can also be prepared by sulphonating the leuco base with fuming sulphuric acid and subsequently oxidising the sulphonic acid in the manner already described.

—T. A. L.

Improvements in the Manufacture of Colouring Matters derived from Anthraquinone. B. Willcox, London. From the "Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 1657, January 27, 1892.

THIS is an extension of the following Eng. Pats. 12,715 of 1890 (this Journal, 1891, 759), 17,712 of 1890 (this Journal, 1891, 917), 18,729 of 1890 (this Journal, 1891, 917), and 4871 of 1891 (this Journal, 1892, 513), which refer to the oxidation of certain tetrahydroxyanthraquinones in sulphuric acid solution with the formation of so-called "alizarin cyanines." To this class belong alizarin bordeaux, purpurin bordeaux, anthrachrysone, anthrapurpurin bordeaux, and flavopurpurin bordeaux. It has now been discovered that hydroxyflavopurpurin and hydroxyanthrapurpurin, described in Eng. Pat. 18,729 of 1890, when dissolved in sulphuric acid and oxidised with manganese dioxide or arsenic acid, yield dyestuffs of the alizarin cyanine series. About 10 kilos. of dry hydroxyflavopurpurin are dissolved in 200 kilos. of sulphuric acid (66° B.) and 15 kilos. of powdered manganese dioxide added at 60°–70° C. The melt is then heated to 100° C. until a test in concentrated sulphuric acid shows a strong cyanine spectrum. The melt is then poured into water and boiled with an excess of acid sodium sulphite, when the dyestuff which separates is filtered off and washed to remove adherent acid. It is found to be identical with the hexahydroxyanthraquinone described in Eng. Pat. 17,712 of 1890. In a similar manner hydroxyanthrapurpurin gives alizarin hexacyanine identical with that described in Eng. Pat. 4871 of 1891. By employing larger quantities of sulphuric acid and manganese dioxide the conversion takes place at a lower temperature. These cyanines can also be obtained directly from flavopurpurin and anthrapurpurin in one operation, using a somewhat larger quantity of manganese dioxide and conducting the operation at about 120° C. It would appear, however, that this high temperature is not a necessity providing that sufficiently large quantities of sulphuric acid and of the oxidising agent be taken. Colouring matters of this cyanine series may also be obtained not only from tetrahydroxy- and trihydroxy-anthraquinones, but also from dihydroxyanthraquinones providing that these compounds contain at least one hydroxyl group in each benzene nucleus. A further series of colouring matters produced by the oxidation process is obtained from the products formed by the action of ammonia on alizarin bordeaux or alizarin pentacyanine described in Eng. Pat. 8702 of 1891 (this Journal, 1892, 514). The dyestuffs obtained produce greener shades with mordants than the original substances. The following quantities are given: 10 kilos. of the product of the action of ammonia on alizarin bordeaux are dissolved in 200 kilos. of sulphuric acid (66° B.), and 12 kilos. of 90 per cent. manganese dioxide stirred in at 20°–25° C. After stirring

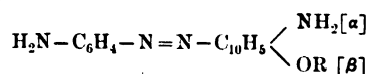
for three hours the product is poured into water and the whole boiled up with 10 kilos. of a 34° B. acid sodium sulphite solution. The precipitate is then filtered off and washed. It dissolves in sodium carbonate and ammonia with a violet colour, in caustic soda with an indigo-blue colour, and in concentrated sulphuric acid with a violet-red colour. The alcoholic solution is bluish-red and exhibits a characteristic absorption spectrum, thus differing from the original substance. With chromium mordants pure blue shades are produced.—T. A. L.

The Treatment of Amido-Guanidine for the Manufacture of a New Compound, and of Products derived therefrom. J. Y. Johnson, London. From the "Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 2194, February 4, 1892. (Second Edition.)

THE preparation of diamidogen from amido-guanidine has already been described in Eng. Pat. 6786 of 1891 (this Journal, 1892, 370). Some further reactions and decompositions of the compound are now published. By treatment with nitric acid, amido-guanidine yields diazo-guanidine, which on treating with sodium acetate forms a new compound called amido-tetrazotic acid. This can be diazotised and employed in the manufacture of colouring matters. Diazo-guanidine also, on treatment with caustic soda, yields azohydric acid (N_3H). The following method is given for producing diazo-guanidine nitrate. One molecular proportion of amido-guanidine nitrate is dissolved in water together with one molecular proportion of nitric acid, and a solution of one molecular proportion of sodium nitrite is run in below 40°–45° C. On evaporation at a low temperature the nitrate is obtained as a crystalline residue. It crystallises from alcohol, forming white crystals melting at 129° C. To obtain amido-tetrazotic acid, the solution of diazo-guanidine nitrate as above obtained is treated with sufficient sodic acetate to form the acetate. The solution is then evaporated, when the diazo-guanidine acetate decomposes into acetic acid and amido-tetrazotic acid, which crystallises from the concentrated solution and can be purified by crystallisation from water. It has the composition $CN_4NH_2 \cdot H_2O$, and on heating loses its water of crystallisation and melts at 199° C. In order to produce azohydric acid, a solution of one molecular proportion of a salt of diazo-guanidine is mixed with rather more than two molecular proportions of caustic soda. This forms sodium azotate, which on distillation with a mineral acid gives off azohydric acid. The same acid is also produced by distilling a salt of diazoganidine with dilute mineral acids. Attention is drawn to the explosive character of azohydric acid and its compounds.—T. A. L.

New Manufacture of Bases and of Colouring Matters therefrom. C. D. Abel, London. From the "Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 2408, February 8, 1892.

NEW bases which contain two amido groups capable of diazotisation are obtained by combining *p*-nitro-diazobenzene with the α -amido- β -naphthol ethers and subsequently reducing the nitro-amido-azo compounds with alkaline sulphides. The constitution of these bases is represented by the general formula—



The preparation is performed by combining equal molecular proportions of diazotised *p*-nitraniline and α -amido- β -naphthol ether, filtering off the reddish-brown precipitate of the nitro-azo-amido compound, suspending it in alcohol, adding sodium sulphide, and heating for some time to 70°–80° C. The base is precipitated from the orange-red solution by the addition of water. These bases are readily soluble in alcohol, ether, or benzene, and dissolve with a deep bluish-red colour in dilute acids. They are converted into tetrazo compounds by the action of nitrous acid and yield valuable colouring matters with the