

UNITED STATES PATENT OFFICE.

MORITZ ULRICH AND JOHANN BAMMANN, OF ELBERFELD, GERMANY,
ASSIGNORS TO THE FARBENFABRIKEN, VORMALS FR. BAYER &
CO., OF SAME PLACE.

AMIDONAPHTHOLDISULFO ACID.

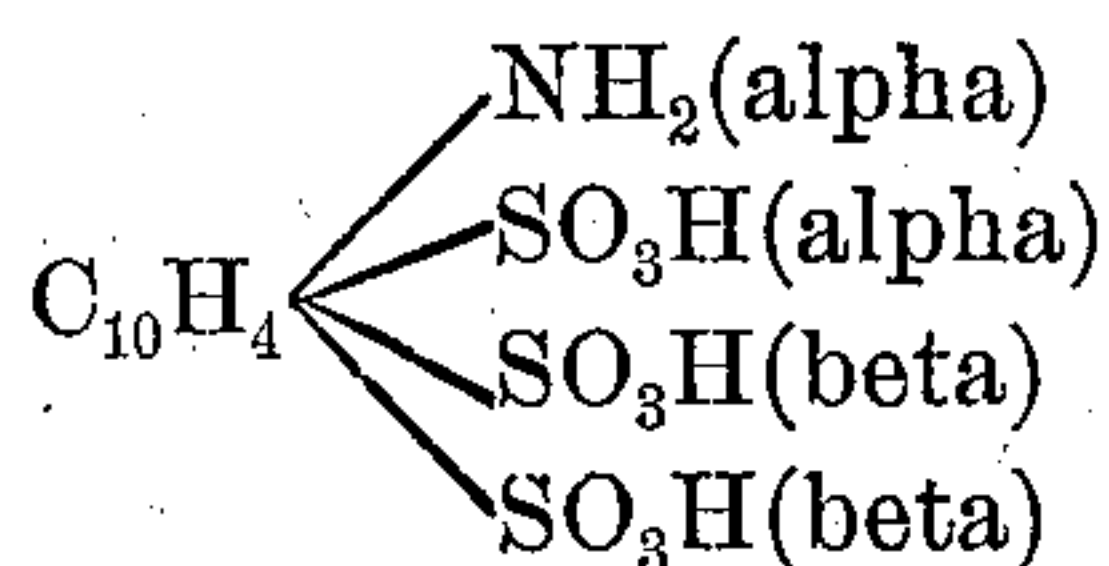
SPECIFICATION forming part of Letters Patent No. 540,412, dated June 4, 1895.

Application filed May 10, 1892. Serial No. 432,495. (Specimens.) Patented in England August 26, 1890, No. 13,443; in Austria-Hungary November 28, 1890, No. 35,494 and No. 58,417; in France December 6, 1890, No. 210,033, and in Italy April 27, 1891, XXV, 29,631, LVIII, 100.

To all whom it may concern:

Be it known that we, MORITZ ULRICH and JOHANN BAMMANN, doctors of philosophy, subjects of the Emperor of Germany, residing at Elberfeld, Prussia, Germany, (assignors to the FARBENFABRIKEN, VORMALS FR. BAYER & Co., of Elberfeld,) have invented a new and useful Improvement in the Manufacture of Amidonaphtholdisulfo Acids, (for which the aforesaid FARBENFABRIKEN have already obtained Letters Patents in England, No. 13,443, dated August 26, 1890; in France, No. 210,033, dated December 6, 1890; in Italy, Vol. XXV, 29,631, Vol. LVIII, 100, dated April 27, 1891, and in Austria-Hungary, No. 35,494 and No. 58,417, dated November 28, 1890,) of which the following is a clear and exact description.

Our invention relates to the production of a new amidonaphtholdisulfo acid which is an alphaamidoalphanaphtholbetadisulfo acid usually termed 1.8 amidonaphtholbetadisulfo acid by melting with caustic alkalies most practically at a temperature from 180° to 190° centigrade the alphanaphthylaminetrisulfo acid which corresponds with the formula—



and is derived from the naphthalenetrisulfo acid obtained at first by Gürke and Rudolph by sulfonating naphthalene or its mono or disulfo acid and prepared first by Koch by nitrating the said naphthalenetrisulfo acid and reducing the alphanitronaphthalenetrisulfo acid thus formed.

The following is a clear and exact description of the manufacture of this new alphaamidoalphanaphtholbetadisulfo acid.

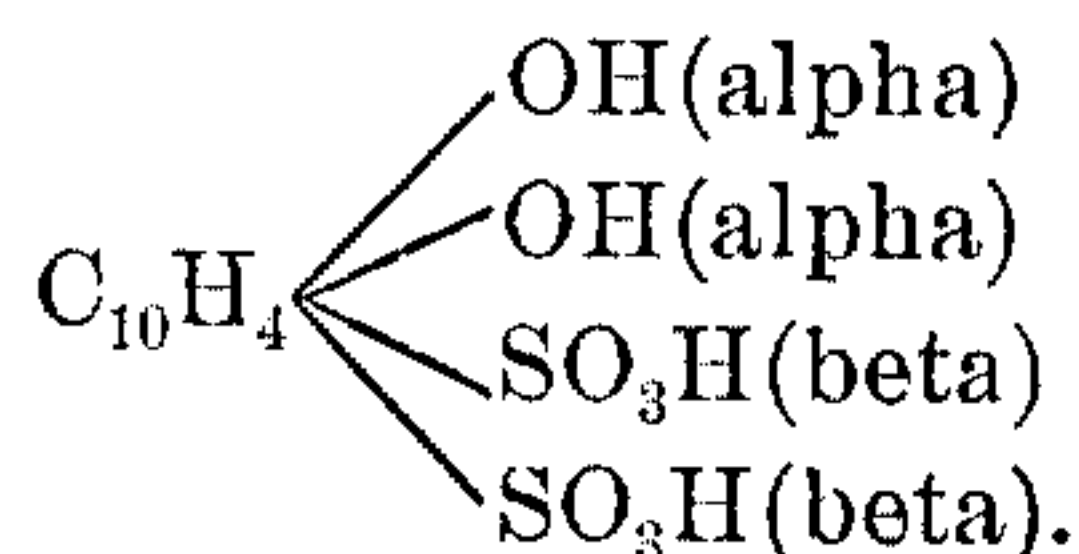
In carrying out our process practically we proceed as follows: One part, by weight, of the above specified alphanaphthylaminetrisulfo acid is melted with two parts, by weight, of caustic soda and a small quantity of water

at from 180° to 190° centigrade in an open vessel, until the melting mass has become clear and of a very liquid consistency and a test-portion thereof when poured into water displays a reddish-violet fluorescence. When the reaction has been found to be complete the resulting melt is decomposed by acidulating with hydrochloric acid. The acid sodium salt of the new amidonaphtholdisulfo acid formed in this process separates in quantitative yield in the form of small thin needles.

Instead of the free alphanaphthylaminetrisulfo acid the corresponding quantity of one of its alkaline salts and likewise in place of caustic soda also caustic potash or a mixture of both may be employed with the same effect. We do not limit our process to the employment of the aforesaid temperature from 180° to 190° centigrade as a somewhat higher temperature may be used, but we have found that the melting process carried out in the above described manner yields very good results. The process may also be effected in closed vessels.

Our new acid thus obtained which we term with regard to its composition and constitution alphaamidoalphanaphtholbetadisulfo acid or usually 1.8 amidonaphthol-beta-disulfo acid possesses the following properties: It separates on decomposing the melt by acids in the form of its acid sodium salt, which crystallizes in small thin white needles moderately soluble in cold, easily soluble in hot water. Its solutions in water show a weak reddish-violet fluorescence which is not altered by the addition of alkalies. Ammoniacal silver solution is reduced on heating it with the salts of this new acid. On adding a solution of ferric chlorid or chlorid of lime to its solutions in water yellowish-brown liquids are obtained which, however, become almost colorless on the addition of an excess of the latter reagent. It is changed by a treatment with nitrous acid, viz: by diazotation in moderately strong hydrochloric acid solution into the muriatic salt of the corresponding diazonaph-

tholdisulfo acid which crystallizes in long light yellow needles and yields by boiling with water,—most practically in the presence of free mineral acids,—a dihydroxynaphthalenedisulfo acid which, as we have found, is identical with the alphadihydroxynaphthalenebetadisulfo acid, usually termed 1.8 dihydroxynaphthalenebetadisulfo acid, described in the Letters Patent No. 458,283, dated August 25, 1891, and having the formula:



By combining the salts of diazobenzene or analogous diazo compounds with our new acid red colors with a strong bluish tinge result, while the tetrazo dye-stuffs obtained by combining a tetrazo salt with our new acid in general produce beautiful blue shades.

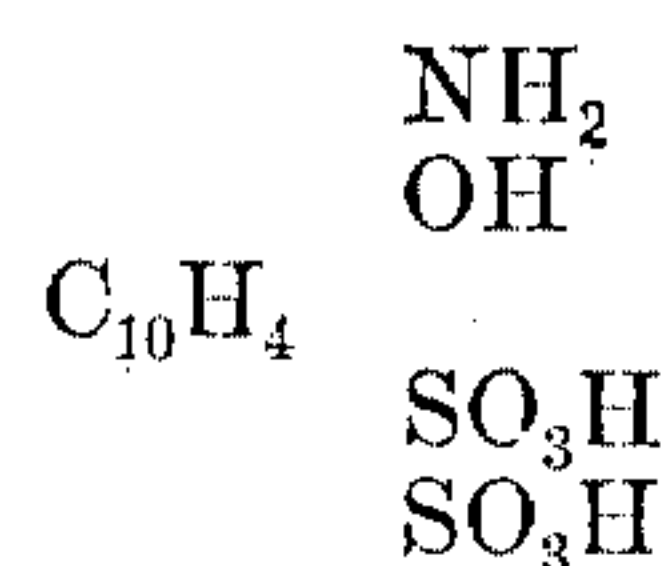
We have found that our new acid is identical with the amidonaphtholdisulfo acid which has been obtained by Leopold Cassella & Co. by changing naphthalene 2.7 disulfo acid into its dinitro compound, reducing the latter and heating the diamidonaphthalenedisulfo acid thus formed with diluted acids under pressure as described in the French Letters Patent No. 210,950, dated January 22, 1891, and the British Letters Patent No. 1,742, dated January 30, 1891.

We are aware of the American Letters Patent No. 458,286, dated August 25, 1891, but our new alphaamidoalphanaphtholbetadisulfo acid or 1.8 amidonaphtholbetadisulfo acid is not identical with the amidooxynaphthalenedisulfo acid claimed in the last named Letters Patent which corresponds as mentioned in the specification with the following patents: in Germany, No. 53,023, dated September 7, 1889; in England, No. 15,175, dated September 26, 1889, and in France, No. 201,742, dated November 5, 1889, wherein only betanaphthylaminetrisulfo acids are subjected to a melting process.

Our new acid chiefly differs from the amidooxynaphthalenedisulfo acid described in the Letters Patent No. 458,286, dated August 25, 1891, by the following properties: The solutions of the former or the salts thereof in water show a weak reddish-violet fluorescence which is not altered by an addition of alkalies while the solutions of the latter or the salts thereof in water show a violet-blue fluorescence which changes into blue-green on the addition of alkalies. The watery solutions of our new acid or the salts thereof assume a yellowish-brown color on adding ferric chlorid or chlorid of lime, while these reagents produce in the watery solutions of the above amidooxynaphthalenedisulfo acid or the salts thereof a reddish-brown color.

We disclaim the subject-matter stated in

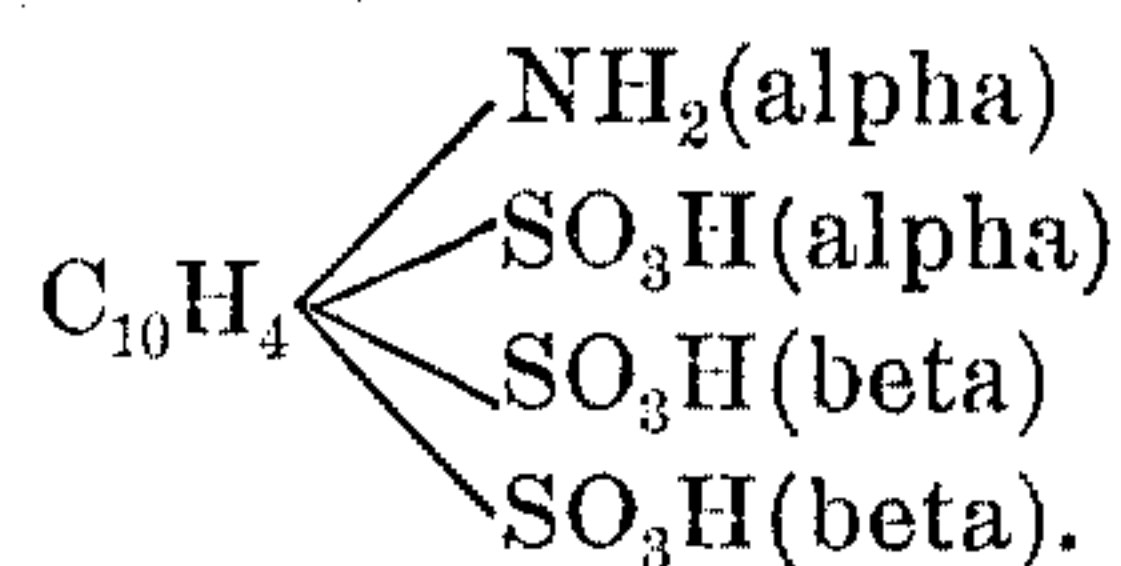
the following words: A new acid, which has the formula—



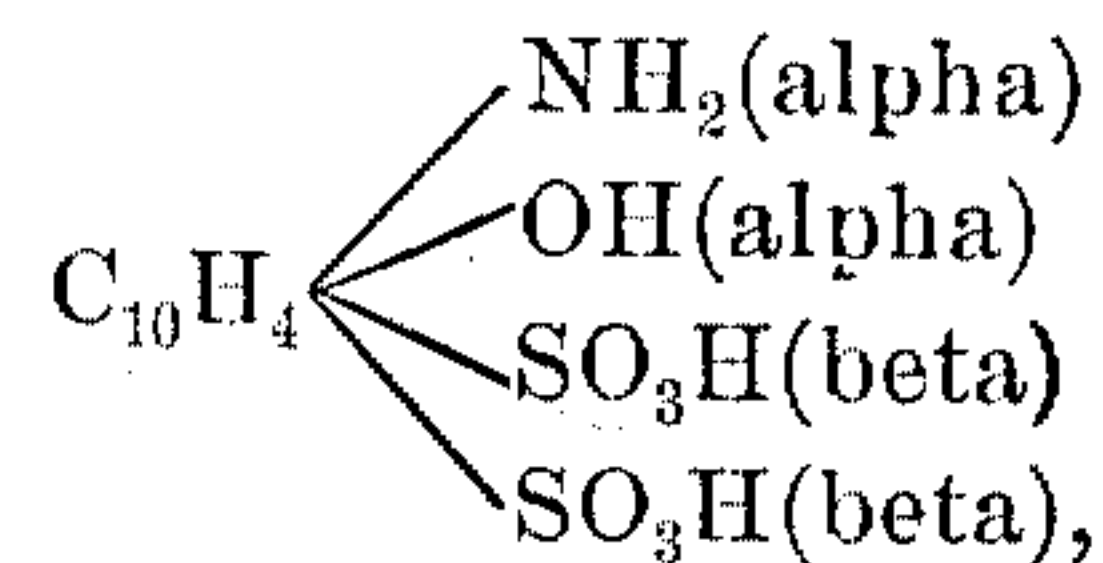
forming needle-like crystals, soluble in water with violet fluorescence, turning brown on the addition of ferric chlorid or calcium hypochlorite, and decolorized by an excess of the latter reagent, and becoming diazotized by treatment with nitrous acid, substantially as described, our invention being fully and distinctly stated in the claims of this application.

Having thus described the nature of this invention and in what manner the same is to be carried out, we declare that what we claim as new, and desire to secure by Letters Patent, is—

1. The process of producing the salt of a new alphaamidoalphanaphtholbetadisulfo acid, which consists in melting with alkalies the alphanaphthylaminetrisulfo acid, which corresponds with the formula—



2. As a new product, the alphaamidoalphanaphtholbetadisulfo acid, having the composition:



forming an acid sodium salt, which crystallizes in small, thin, white needles; moderately soluble in cold, easily soluble in hot water, showing in aqueous solution a weak reddish-violet fluorescence, which is not altered by the addition of alkalies; reducing ammoniacal silver solution on heating, and being converted by nitrous acid, namely, by diazotation in moderately strong hydrochloric acid solution into the corresponding diazo naphtholdisulfo acid, which crystallizes in long, light yellow needles, and yields on boiling with water, most suitably in the presence of free mineral acids, a dihydroxynaphthalene beta disulfonic acid, termed in chemical literature 1.8 dihydroxynaphthalene beta disulfonic acid.

In testimony whereof we have signed our names in the presence of two subscribing witnesses.

MORITZ ULRICH.
JOHANN BAMMANN.

Witnesses:
WM. ESSENWEIN,
RUDOLPH FRICKE.