

# ALIEN PROPERTY CUSTODIAN

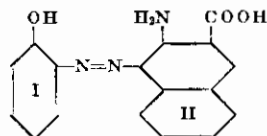
## COMPLEX CHROMIUM COMPOUNDS OF AZO DYE STUFFS

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The present invention relates to complex chromium compounds of azo dyestuffs.

I have found that valuable complex chromium compounds of azo dyestuffs are obtained by treating in substance azo dyestuffs of the formula



wherein the nuclei marked I and II may contain further substituents, with agents capable of yielding chromium.

The starting materials are obtainable by diazotizing an ortho-aminophenol which preferably contains at least one additional substituent, such as halogen or a nitro, alkyl, alkoxy or sulfonic acid group, and combining the diazo compound with 2-aminonaphthalene-3-carboxylic acid or a substitution product thereof being free in the 1-position. Suitable coupling components are for example 2-aminonaphthalene-3-carboxylic acid-5- or 6-sulfonic acid.

The complex chromium compounds may be prepared by heating the metal-free dyestuff with a chromium compound, e. g. chromic formate or chromic chloride, in aqueous or organic solution or suspension, if desired under superatmospheric pressure. The chromium may also be introduced into the azo dyestuffs by heating them in a melt prepared from a carboxylic acid amide or the ammonium salt of a carboxylic acid and containing a compound of trivalent chromium.

The complex chromium compounds thus obtained by chromination in substance, i. e. in the absence of fibres, are characterized in that they contain one atom of chromium for each molecule of the dyestuff. Chromination on the fibre, however, leads to the formation of chromium compounds containing two atoms of chromium for three molecules of the dyestuff.

The new complex chromium compounds, prepared in substance, are capable of dyeing animal fibres, e. g. wool or silk, or synthetic fibres having the properties of animal fibres, clear green shades. The dyeings are very level and excellently fast to light, water, perspiration, steam and hot-ironing. Chrome-tanned leather is dyed deep green shades which are very fast to washing and water. The complex chromium compounds derived from dyestuffs free from sulfonic acid groups or the salts prepared from chromium compounds of dyestuffs containing sulfonic acid groups and organic bases may be used for dyeing cellulose ester

lacquers, plastic compositions or acetate artificial silk spinning solutions.

The following examples will further illustrate how my invention may be carried out in practice.

5 The invention, however, is not restricted to these examples. The parts are by weight.

### Example 1

23.4 parts of 1-amino-2-hydroxy-3-nitrobenzene-5-sulfonic acid are diazotized in the usual manner at 15° C. The diazo solution is then rendered acid to litmus by adding thereto 15 parts of sodium acetate. A weakly alkaline solution of 20.5 parts of 2-aminonaphthalene-3-carboxylic acid, heated to 75° C, is allowed to flow into the diazo solution, the whole being kept at 50° C until coupling is completed. The azo dyestuff formed is salted out, filtered off and washed. The dyestuff is admixed with water to form a paste, the latter being heated with a chromium formate solution, containing 20 parts of chromic oxide and 20 parts of formic acid, for 4 hours to 130° C under superatmospheric pressure. The complex chromium compound deposits in the form of a dark powder when allowing the reaction mixture to cool. It dyes wool and silk clear green shades of good fastness.

A similar dyestuff yielding somewhat bluer shades may be prepared in the same manner from the azo dyestuff derived from diazotized 1-amino-2-hydroxy-5-nitrobenzene-3-sulfonic acid and the same coupling component.

### Example 2

The diazo solution obtained from 15.4 parts of 1-amino-2-hydroxy-4-nitrobenzene is added in acetic acid solution at 50° C to a solution of 30 parts of 2-aminonaphthalene-3-carboxylic acid-6-sulfonic acid. The azo dyestuff formed is salted out, filtered off by suction and dried. It is dissolved in 250 parts of formamide at 110° C. 27 parts of chromic chloride (CrCl<sub>3</sub>·6H<sub>2</sub>O) are added, and the melt is heated to 120° C for 2 hours and to 130° C for one further hour. The whole is then poured into water whereby the chromium containing dyestuff is precipitated out. It dyes chrome-tanned leather clear green shades of good fastness to washing and water. Wool and silk are dyed vivid green shades.

50 Similar dyestuffs are obtained when employing 1-amino-2-hydroxy-4-nitro-5-chlorobenzene or 1-amino-2-hydroxy-3-nitrobenzene-5-sulfonic acid as diazo components.

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