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NONDUSTING COMPOSITIONS FOR STABILIZING DIAZO SALTS

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This invention relates to a process for reducing dusting in powders, more particularly in powders of organic color compounds, chromogens and auxochromic compounds and to dustless compositions of the same.

It is customary in the manufacture of organic color compounds, i. e., dyestuffs and organic pigments, and of chromogens and auxochromic compounds to prepare these materials in powder form as in this way economy and convenience in shipping and handling of the products is to be obtained. These advantages in the powder form of the products are offset to some extent by the tendency of the products to become objectionably dusty in handling, a condition brought about by air-lifting of very fine product particles which are always present in the powders. The degree of the dusting will vary depending on the concentration of very fine particles in the powders and on the conditions surrounding the handling of the powders, such as the presence and magnitude of neighboring air-currents, the length of fall in pouring, or the vigor of the packing in packaging, etc. The dusting in some instances may be so slight as to be negligible in effect. On the other hand, in extremely pronounced cases it is a health hazard, causing pneumoconiosis or skin irritations among the workers, as well as a noticeable loss of material. In pronounced cases of dusting, packaging, pouring and measuring are difficult while in extreme cases accurate measurement is sometimes a practical impossibility due to loss of material.

I have now found that dustiness in pulverulent organic color compounds, chromogens and auxochromic compounds may be overcome in an advantageous manner by incorporating therewith a minor proportion of an alkyl monohaloacetate in which the alkyl group contains not more than 4 carbon atoms.

These alkyl monohaloacetates are liquid compounds having the general formula:



wherein R is a lower alkyl group having 1 to 4 carbon atoms, for example, methyl, ethyl, propyl, isopropyl, butyl, isobutyl, etc., and X is halogen which may be chlorine, bromine, fluorine or iodine. These alkyl monohaloacetates exhibit toward the organic color compounds, chromogens and auxochromic compounds, non-reactivity, but a slight solvent action, non-interference with their use in the role of colorant, chromogen or auxochromic compound as the case may be, and a high degree of permanency in respect to the

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dust-binding effect. They do not have an unpleasant odor and have low volatility at ordinary temperatures. The preferred embodiment of the invention resides in the use of these compounds wherein the halogen is chlorine, due chiefly to price and availability.

The amount of the liquid alkyl monohaloacetates used in treating the powders in accordance with the invention may be varied. Positive results in dust reduction may be obtained with as little as 0.5% of one of the alkyl monohaloacetates on the weight of the powder. Optimum amounts range from about 1 to 3% by weight, although in some instances for best results up to about 5% by weight may be required. Where economically feasible, still larger amounts may be used with excellent results, for example between about 5 and 10% by weight. Amounts should not be employed, however, as would set up a continuous liquid phase in the powder bringing about separation of the alkyl monohaloacetate therefrom by drainage. The incorporation of the liquid alkyl monohaloacetates with the powders may be accomplished in any suitable and convenient way, for example, by the use of powder blending machines having provision for spraying or otherwise distributing the liquid uniformly over the mass of the powder so as to avoid formation of pastes in the mass. For best results, a thorough mixing of the powder and liquid should be obtained.

The term chromogen is used herein in the customary sense of the term to mean those organic compounds which, in themselves not dyestuffs, are capable of being converted to dyestuffs by an agency such as oxidation or by coupling with a compound containing one or more auxochrome groups, i. e., auxochromic compounds. Under the term chromogen as defined herein are included leuco vat dyestuffs and their derivatives such as the leuco sulfuric acid esters, which compounds on oxidation are converted to the vat dyestuffs, and the water-soluble stabilized diazo compounds which in aqueous medium, acid, neutral or alkaline as the case may be, couple with auxochromic compounds.

The term auxochromic compound is used herein in the customary sense of the term to mean those organic compounds which contain one or more auxochromes which principally are hydroxy, amino and substituted amino groups. Compounds of this kind are for example, resorcinol, salicylic acid, β -naphthol, β -naphthol-3,6-disulfonic acid (sodium salt), dehydrothio-p-toluidine, β -naphthylamine, β -naphthylamine-

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3,6-disulfonic acid and the like. A class of auxochromic compounds with which the invention is particularly concerned are those known in the art as Naphthol-AS compounds. The Naphthol-AS compounds are arylides of carboxylic acids capable of coupling with a diazo compound to form an azo dyestuff. Examples of this type of coupler compounds are, the anilides or anisidides of 2,3-hydroxynaphthoic acid, 2,3-hydroxyanthroic acid, 2-hydroxy-11-benzo(a)-carbazole-3-carboxylic acid, 2-hydroxy-3-dibenzofurancarboxylic acid, etc., and the bisbenzidides of acetoacetic acid, terephthaloyl- α,α' -diacetic acid and the like.

The water-soluble stabilized diazo compounds may be prepared by reacting the diazonium salt in aqueous solution with metal salts such as zinc chloride, cadmium chloride and tin chloride or with compounds such as the alkali metal fluoroborates, alkyl or aryl sulfonic acids, e. g., naphthalene-1,5-disulfonic acid, and acid sulfates. These stabilized diazo compounds exhibit the properties of true salts in that they ionize immediately in neutral or acid solution and show the reaction of diazo ions. Amines which carry one or more water-solubilizing groups such as the sulfonic or carboxylic acid groups to render the stabilized compound soluble in water may also be used for the preparation of stabilized diazo compounds. Amines of this character are, for example, sarcosine, methyl taurine, N-ethyl or N-methyl-5-sulfo-anthranilic acid, proline and N-methyl glucamine. Depending on whether the amine employed is primary or secondary, the resulting stabilized compound is a diazo-amino or a diazo-imino compound. In the art and herein also, these stabilized diazo-amino and diazo-imino compounds are contained under the generic or collective term, water-soluble stabilized diazo-amino compounds.

The invention and the application of the process thereof to the preparation of dustless powders of organic color compounds, chromogens and auxochromic compounds is illustrated in more detail in the following examples, to which, however, it is not intended that the invention be limited. Parts are by weight.

Example 1

50 parts of a powder of a stabilized diazo compound constituted by the zinc chloride double salt of diazotised 4-chloro-o-nitroaniline and 0.75 part of ethyl chloroacetate are well mixed by milling together in a small mixing device. The product has outstanding non-dusting properties and good storage stability.

The zinc chloride double salt of diazotised 4-nitro-o-anisidine similarly treated with ethyl chloroacetate acquires excellent non-dusting properties.

Example 2

50 parts of a powder of the zinc chloride double salt of diazotised 2,5-dichloroaniline and 1 part of ethyl chloroacetate are mixed in the manner of the previous example. The product has excellent non-dusting properties.

Example 3

50 parts of a powder of the azo dye prepared by coupling diazotised p-nitroaniline and dihydroxyethyl-m-toluidine is mixed as in Example 1 with 1 part of ethyl chloroacetate. The product is a composition of excellent non-dusting properties.

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Example 4

50 parts of a powder of dimethoxydibenzanthrone is well mixed with 1 part of ethyl chloroacetate in the manner of the preceding examples. The product has outstanding non-dusting properties.

Example 5

50 parts of a powder of an azoic dye mixture comprising Naphthol AS-G (bisacetoacet-o-toluidide) and diazotised 5-chloro-o-anisidine stabilized with sarcosine is well mixed with 2.5 parts of ethyl chloroacetate by milling together in a small mixing device. The composition is a non-dusting powder.

In like manner, the process may be applied for the reduction of dusting in powders of chromogens, auxochromic compounds and dyestuffs generally, and for the preparation of non-dusting powders of organic pigments, such as azo pigments per se, metal complexes of azo dyestuffs such as the copper or cobalt complexes of the azo dyestuffs obtained by coupling tetrazotised dianisidine or tetrazotised benzidine with 2-naphthol-6-sulfonic acid (Schaeffer's salt), phthalocyanine pigments, and color lakes such as those prepared by the deposition on water-insoluble substratum such as barium sulfate of azo dyestuffs, for example, the yellow pigment from the coupling of diazotised o-nitro-p-chloroaniline with acetoacet-o-chloroanilide. Non-dusting powders of dyestuffs such as Methyl Violet, Crystal Violet and the rhodamines, acridine dyestuffs and vat dyestuffs, for example indanthrones, pyranthrones, flavanthrones, dibenzanthrones, anthranthrones, dibenzopyrenequinones, anthrimidecarbazoles, naphthacridones, indigo, thioindigo, indirubin, etc., and their leuco and leuco ester derivatives.

The invention is applicable to the preparation of dustless powder compositions which contain a water-soluble stabilized diazo compound in admixture with a Naphthol-AS type coupler. In its broader aspect the process may be applied in many instances to the binding of dust in powder mixtures of organic color compounds, chromogens or auxochromic compounds which contain other powdered materials useful in the coloring art, such as dyeing or printing assistants, for example, surface-active agents, e. g., aliphatic acid condensation products of taurine. The same relative proportions of the liquid alkyl monohaloacetate are applicable in such cases, the total quantity thereof being taken on the weight of the powder mixture.

I claim:

1. A composition of matter comprising a pulverulent water-soluble stabilized diazo compound which is a salt admixed with an amount of an alkyl monohaloacetate in which the alkyl group contains not more than 4 carbon atoms, which is not below about 0.5% by weight but is less than would result in liquid separation by drainage.

2. A composition of matter comprising a powder of a water-soluble stabilized diazo compound which is a salt admixed with from about 1 to 5% by weight of an alkyl monochloroacetate in which the alkyl group contains not more than 4 carbon atoms.

3. A composition of matter comprising a powder of a water-soluble stabilized diazo compound which is a salt admixed with from about 1 to 5% by weight of ethyl chloroacetate.

4. A composition of matter comprising a pul-

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verulent water-soluble double salt of a metal salt and a diazo compound admixed with from about 1 to 5% by weight of an alkyl monochloroacetate in which the alkyl group contains not more than 4 carbon atoms.

5 A composition of matter comprising a pulverulent water-soluble double salt of zinc chloride and a diazo compound admixed with from about 1 to 5% by weight of an alkyl monochloroacetate in which the alkyl group contains not more than 4 carbon atoms. 10

6. A composition of matter comprising a pulverulent water-soluble stabilized diazo compound which is a salt of an aryl sulfonic acid admixed with from about 1 to 5% by weight of an alkyl monohaloacetate in which the alkyl group contains not more than 4 carbon atoms. 15

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