STABILIZED DIAZONIUM SALTS AND PROCESS OF EFFECTING SAME

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10 Claims. (Cl. 260—141)

1. Alkali metal Soaps Such as Sodium oleate, CH₃(CH₂)₂CH:CH(CH₂)COONa, and sodium stearate CH₃(CH₂)₁₆COONa

2. Sulfates of higher fatty acids such as sulfonated castor oil (triglyceride of ricinoleic acid)
   OSO₄Na
   CH₂(CH₃)₃CHCH₂CH(CH₂)COOCH₃
   OSO₄Na
   CH₂(CH₃)₃CHCH₂CH(CH₂)COOCH₃
   OSO₄Na
   CH₂(CH₃)₃CHCH₂CH(CH₂)COOCH₃

3. Sulfonated higher fatty alcohols such as the octyl sulfonic acid and the sodium salt of dodecyl sulfonic acid of USP 1,966,187.

4. Sulfated higher fatty alcohols such as sodium lauryl sulfate
   C₁₂H₂₅OSO₄Na

5. Phosphated higher alcohols such as the product known as W.A.35A.
   (CH₃)₃PONa


7. Sulfosuccinic acid esters such as a dibutyl ester of sodium sulfosuccinate and the diethyl ester of sodium sulfosuccinate.

8. Acylamidoalkylsulfonates such as the product known as Igepon T
   CH₁₅H₂₃CON(CH₃)₂SO₄Na

9. Acylaminooxyalkyl carboxylates such as the sodium salt of oleyl sarcosine
   CH₁₈H₃₇CONH—CH₂COONa

10. Alkylsulfonamidoalkyl sulfonates such as the product known as Emulphor STU
    CH₃(CH₂)₁₂–₁₄SO₄NHCH₂CH₃SO₄Na

11. Alkylsulfonamido acetates such as the product known as Emulphor STH
    CH₃(CH₂)₁₂–₁₄SO₄NHCH₂COONa

12. Mixed fatty acid monoglyceride sulfates such as the product known as Arctic Syntex M (Young and Coons' Surface Active Agents, page 119, Chemical Publ. Co., Brooklyn, N. Y., 1945).
13. Alkylphenoxalkyl sulfates such as the sodium sulfuric ester of isooctyl phenyl monoglycol ether

\[ (\text{C}_8\text{H}_{17})_2\text{CH}_2\text{O}CH_2\text{OSO}_3\text{Na} \]

The sodium sulfuric ester of dodecylphenyl monoglycol ether

\[ (\text{C}_{12}\text{H}_{25})\text{CH}_2\text{O}CH_2\text{OSO}_3\text{Na} \]

and the sodium sulfuric acid ester of tetramethylbutylphenoxy ethanol

\[ (\text{CH}_3)_4\text{CH}\text{CH} = \text{CH}\text{CH}_3\text{O}CH_2\text{OSO}_3\text{Na} \]

14. Amino soaps of fatty acids such as the stearic acid soap of 2-amino-1-butanol

\[ \text{CH}_3\text{CH} = \text{CH}
\]

The diazonium compounds stabilized by the surface active agents and the salts or acids generally employed as stabilizing agents in Fast Color Salts such as metallic chlorides, particularly the mercury, iron, zinc, cadmium, tin, antimony, platinum, gold, copper and arsenic chlorides, naphtalene sulfonic acids such as naphtalen-1,5-disulfonic acid, and fluoroboric acid are prepared by introducing a solution of the surface active agent into the diazotation solution. The combined diazo solution and surface active agent solution is then mixed with a solution containing the additional stabilizing agent. The exact order of mixing the reactants is not material, although it is preferable to initially mix the diazonium compound and the surface active agent. Thus the diazo and surface active agent can be combined first and the combined solutions added to a solution of the Fast Color Salt stabilizer (such as ZnCl₂); the Fast Color Salt stabilizer can be added to the mixture of the diazo plus the surface active agent; the diazo and the surface active agent may be run into the solution of the Fast Color Salt stabilizer simultaneously; or the latter may be added to the diazo solution prior to the surface active agent provided that the diazo solution is kept sufficiently dilute to prevent precipitation of the stabilized diazo before the surface active agent can act on it. When partial stabilization is effected with the surface active agents and stabilization completed with metallic chlorides or other known stabilizers, the stabilized diazonium salts precipitate out with a crystal structure which is distinguished from that of the crystal structures of diazonium salts produced from the same diazonium compounds but stabilized only with the usual or conventional stabilizers. This new crystal growth results in most cases either in the formation of larger crystals which may be different in structure from the crystal structure of the corresponding conventionally stabilized diazonium salts or in a change of degree of crystal aggregation, usually forming a greater degree of aggregation. In either case the product can be handled with greater ease and the solutions more efficiently filtered. It is also characteristic of the diazonium compounds stabi-

The stabilization of the diazonium salts by means of the surface active agents of this invention thus results in an appreciable reduction of the water content of the filtered diazo cake, as a direct result of the change in crystal pattern or formation of larger crystals or crystal aggregates.

As compared with ordinary Fast Color Salts which are stabilized only with the usual metallic salts, less water is held by the stabilized diazonium salts of this invention than is held by the Fast Color Salts, the comparative ratio being in the range of 25 to 50% for the conventionally stabilized diazonium salts to 5 to 15% for the diazonium salts stabilized in accordance with this invention. In order to obtain these results appreciable amounts of the surface active agent are used. Preferably about 5 to 20% of the surface active agent based on the amount of the diazo compound is employed. After filtering and dry mixing of the filter cake it may be noted that the dusting characteristic of the dried powdered product is decreased to a considerable degree and in many cases the resulting Fast Color Salts are rendered completely non-dusting. The resistance of the resulting stabilized diazonium salts or Fast Color Salts to decomposition during storage is also greatly improved by the use of the surface active agents. In order to inhibit decomposition of stabilized diazonium salts during storage, it is necessary that they be kept as dry as possible. This is accomplished generally by adding with the stabilized diazonium salts anhydrous agents, such as the anhydrous sodium sulfate, anhydrous magnesium sulfate and partially dehydrated aluminum sulfate mentioned above. These compounds combine with water to form water of crystallization and thus keep the diazonium salts dry. The less water initially present in the filter cake the smaller the amount of dehydrating agent which must be used. In view of the large crystal structure or increased crystal aggregate formation resulting from the partial surface active compound stabilization of this invention, the filter cake contains only a fraction of the water which is present in the same diazonium salt stabilized by the usual complex salt formation with such compounds as ZnCl₂ and hence requires smaller amounts of the dehydrating agents. Also, the necessity for rigorous oven drying is eliminated in those cases where the Fast Color Salts stabilized with the usual stabilizers retain a large amount of water. In such cases it is normally necessary not only to hydraulically press them, but also to pre-dry them in ovens heated to approximately 40° C. for from 12 hours to 2 days in order to bring down the water content. Only after such rigorous drying treatment can they be dry mixed in the usual way. In the case of the surface active agent stabilized diazonium compound of this invention the stabilized diazonium salt will be in such a form that even a secondary drying treatment can be effected immediately with the dehydrating agents and the danger of at least partial decomposition of the diazo compound due to exposure to excessive heat for long periods of time is averted.

The following examples will serve to more fully illustrate this invention. It is understood, however, that the invention is not limited to the specific conditions obtaining in these examples which are intended to be illustrative of the best
manner to effect the products. Unless otherwise stated, the parts are by weight.

Example 1

The following mixture is slurried for 2 to 3 hours:

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<thead>
<tr>
<th>Substance</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>76 cc. water</td>
<td></td>
</tr>
<tr>
<td>50.6 cc. hydrochloric acid (21° Bé.)</td>
<td></td>
</tr>
<tr>
<td>33.6 gr. 4-nitro-o-anisidine</td>
<td></td>
</tr>
</tbody>
</table>

This mixture is then cooled to −10° C. and 13.8 gr. of 100% sodium nitrite are added as a 53% solution, care being taken that the temperature does not rise above +10° C. An excess nitrite test is maintained for 1/2 hour. Charcoal is added and the diazo solution filtered. A solution containing 6.6 gr. sodium oleate per 20 cc. of water is run into the diazo solution. The resulting solution is run into a solution containing 57.6 gr. 1.5-naphthalene disulfonic acid in 100 cc. of water. The precipitate is filtered, sucked dry, and dry mixed and standardized with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate.

The product obtained from this example has a coarser crystalline structure than the product obtained in a similar manner but without the addition of the sodium oleate. Because of this coarser crystalline structure the filter cake has a higher degree of initial dryness, thus requiring the addition of smaller amounts of the drying compounds used in dry mixing and lessening the tendency to dust of the dry particles.

Example 2

The following mixture is slurried for 2 to 3 hours:

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</tr>
<tr>
<td>33.6 gr. 4-nitro-o-anisidine</td>
<td></td>
</tr>
</tbody>
</table>

The mixture is cooled to −10° C. and 13.8 gr. of 100% sodium nitrite are added as a 53% solution, care being taken that the temperature does not rise above +10° C. and excess nitrite test is maintained for half an hour. Charcoal is added and the diazo solution filtered. 16 gr. sulfonated castor oil are added to the diazo solution and a solution of 7 gr. of ZnCl₂ in a small amount of water. The filter cake is then dried. The stabilized diazo is salted out with NaCl equivalent to 10% of the volume, filtered, dried, dry mixed and standardized with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate.

The product thus obtained has a coarser crystalline structure than the product obtained in a similar manner, but without the addition of the sulfonated castor oil. Because of this coarser crystalline structure the filter cake has a higher degree of initial dryness, thus requiring the addition of smaller amounts of the drying compounds used in dry mixing and lessening the tendency to dust of the dry particles.

Example 3

32.4 gr. 2,5-dichloroaniline are slurried with 46 cc. of water and then heated to 60° C. While applying fast agitation 78 cc. hydrochloric acid (21° Bé.) are added over a ten minute period. The mixture is cooled to zero degrees and 30 gr. of ice are added. 13.8 gr. of sodium nitrite are added as a 53% solution. The solution is then purified by treating with charcoal followed by filtering. 5.5 gr. of the sodium salt of dodecyl sulfonic acid are then added, followed by 7 gr. ZnCl₂ dissolved in a small amount of water. The resulting stabilized diazo is salted out with NaCl equivalent to 10% of the volume, filtered, sucked dry, dry mixed and standardized with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate.

The product obtained in accordance with this example has properties similar to the compositions of Examples 1 and 2 as compared with the product obtained in a similar manner to that of this example by omitting the sodium salt of dodecyl sulfonic acid.

Example 4

2,5-dichloroaniline is diazotized in the same manner as in Example 3. After purification of the diazo solution with charcoal and filtering, 9 gr. of sodium lauryl sulfate are added as an aqueous solution, followed by the addition of 7 gr. ZnCl₂ in a small amount of water. The resulting stabilized diazonium salt is salted out with NaCl equivalent to 10% of the volume, filtered, sucked dry, dry mixed and standardized with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate. The thus obtained product has properties similar to those of the product obtained in accordance with Example 3.

Example 5

33.6 gr. 5-nitro-o-anisidine are slurried with 100 cc. of water. To this are added 65 cc. hydrochloric acid (21° Bé.). The mixture is cooled to −10° C. and diazotized with 13.8 gr. NaNO₂ as a 53% solution. The solution is agitated for half an hour, treated with charcoal and filtered. There are then added 30 gr. of a phosphated higher fatty alcohol known as W.A.35A, said to have the formula (C₆H₁₃)₇Na₄P₃O₁₀, which is dissolved in a small amount of water, and 7 gr. ZnCl₂ also dissolved in a small amount of water. The thus stabilized diazo compound is salted out with NaCl equivalent to 10% of the volume, filtered, sucked dry, dry mixed and standardized with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate.

The product of this example has a coarser crystalline structure than the product obtained in a similar manner but without the addition of the phosphated higher fatty alcohol. Because of this coarser crystalline structure the filter cake has a higher degree of initial dryness, thus requiring a smaller amount of the drying compounds used in dry mixing and lessening the tendency to dust of the dry particles.

Example 6

5-nitro-o-anisidine is diazotized in the same manner as in Example 5. After filtering the charcoal treated diazo solution such as Twitchell Oil 2X is added in the amount of 10% of the calculated amount of diazo compound or about 8 gr. dissolved in a small amount of water. An additional 7 gr. of concentrated aqueous solution of ZnCl₂ is added. The precipitate is filtered, sucked dry, standardized and dry mixed with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate.

The product thus obtained has a coarser crystalline structure than the product obtained in a similar manner without the addition of the petroleum oil sulfate. Because of this coarser crystalline structure, the filter cake has a higher degree of initial dryness, thus requiring the addition of smaller amounts of the drying compounds used in dry mixing and lessening the tendency to dust of the dry particles.
Example 7
28.3 gr. of 4-chloro-o-toluidine are added with agitation to a solution of 59 cc. (21° Bé.) hydrochloric acid in 80 cc. of water. The mixture is cooled to −5° C. and 13.8 gr. sodium nitrite in a 33% solution are added while maintaining the temperature below +10° C. The resulting diazo solution is purified by charcoal treatment and filtered. The diazo compound is then stabilized by adding a solution containing 7.5 gr. of the diamyl ester of sodium sulfosuccinate in 20 cc. of water, followed by the addition of 7 gr. ZnCl₂ dissolved in a small amount of water. The stabilized diazo is then salted out with sodium chloride equivalent to 10% of the volume, then filtered, sucked dry, standardized and dry mixed with sodium sulfate, aluminum sulfate, and calcined magnesium sulfate.

The product obtained in accordance with this example has a coarser crystalline structure than the product obtained in a similar manner omitting the addition of diamyl ester sulfosuccinate. Because of this crystalline structure the filter cake has a higher degree of Initial dryness, thus requiring a smaller amount of the drying compounds used in dry mixing and lessening the tendency to dust of the particles.

Example 8
31.5 gr. of 5-chloro-o-anisidine are added to 115 cc. of water and the mixture heated together to 85° C. 27 cc. of hydrochloric acid (21° Bé.) are then added. The resulting solution is cooled to −5° C. and an additional 27 cc. of 21° Bé. hydrochloric acid are added. The solution is again cooled to −5° C. and 13.8 gr. sodium nitrite are added as a 33% solution. The resulting solution is purified by the addition of charcoal and filtered. To the filtered solution there is added a solution of 5 gr. of an acylamidoalkylsulfonate having the formula

\[ \text{CH}_3\text{CONHCH}_2\text{SO}_3\text{Na} \]

in 20 cc. of water. A concentrated aqueous solution of 7 gr. of ZnCl₂ is then added. The resulting stabilized diazo compound is then salted out very slowly with NaCl equivalent to 10% by volume. The precipitate is filtered, sucked dry, standardized and dry mixed with sodium sulfate, aluminum sulfate and calcined magnesium sulfate. A product is obtained which has a coarser crystalline structure than that obtained in a similar manner without the addition of the acylamidoalkylsulfonate. This coarser crystalline product has a higher degree of initial dryness, thus eliminating the necessity for special drying procedures and requiring the addition of smaller amounts of the drying compounds used in dry mixing. The tendency to dust of the dry powder is also considerably reduced.

Example 9
28.3 gr. of 4-chloro-o-toluidine are added to 115 cc. of water and the solution heated to 85° C. 27 cc. of 21° Bé. HCl are then added and the solution cooled to −5° C while adding an additional 27 cc. of 21° Bé. HCl. The solution is again cooled to −5° C, after which 13.8 gr. of sodium nitrite are added as a 33% solution. The resulting diazo compound is purified by the addition of charcoal and filtered. To the filtered diazo solution there are added 4 gr. of the sodium salt of oleyl sarcosine in 20 cc. of water. A concentrated aqueous solution of 7 gr. of ZnCl₂ are then added and the stabilized diazo compound, resulting from these additions, is salted out by adding slowly NaCl equivalent to 10% by volume. The precipitate is filtered, sucked dry, standardized and dry mixed with sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The product thus obtained has characteristics similar to the product of Example 10.

Example 10
31.5 gr. of 4-chloro-o-anisidine are added to 115 cc. of water and the solution heated at 85° C. 27 cc. of 21° Bé. HCl are then added. The resulting solution is cooled to −5° C, and an additional 27 cc. of 21° Bé. HCl are added. The solution is again cooled to −5° C, after which 13.8 gr. sodium nitrite are added as a 33% solution. The resulting diazo solution is purified by the addition of charcoal and filtered. To the filtered solution there are then added 5 gr. of a sodium salt of an alkyl sulfonamido alkyl sulfonate having the general formula

\[ \text{CH}_3\text{(CH}_2)_n\text{SO}_3\text{NHCH}_2\text{CONHCH}_2\text{SO}_3\text{Na} \]

in a solution of 20 cc. of water. A concentrated aqueous solution containing 7 gr. ZnCl₂ is then added. The resulting stabilized diazonium salt is salted out by slowly adding an amount of NaCl equivalent to 10% of the volume. The precipitate is filtered, sucked dry, standardized and dry mixed with sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The product obtained has a coarser crystalline structure than that obtained in a similar manner without the addition of the sodium alkyl sulfonamido alkyl sulfonate. This coarser crystalline product in the form of the filter cake has a higher degree of essential dryness, thus eliminating the necessity for special drying procedures and reducing the amount of the drying compounds required in the dry mixing. The tendency to dust of the dry particles is also reduced.

Example 11
33.6 gr. 5-nitro-o-anisidine are slurried with 100 cc. of water. To this slurry are added 65 cc. of 21° Bé. HCl. The mixture is cooled to −10° C. and there are then added 13.8 gr. sodium nitrite as a 33% solution. The resulting diazonitrosation solution is agitated for half an hour, clarified by the addition of charcoal and filtered. To the filtered diazo solution there are added 5 gr. of the sodium alkyl sulfonamido acetate having the formula

\[ \text{CH}_3\text{(CH}_2)_n\text{SO}_3\text{NHCH}_2\text{CONHCH}_2\text{SO}_3\text{Na} \]

dissolved in 15 cc. of water. A concentrated aqueous solution of ZnCl₂ is then added. The stabilized diazonium salt thus obtained is salted out by the addition of an amount of NaCl equivalent to 10% of the volume. The precipitate is filtered, sucked dry, standardized, and dry mixed with sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The crystalline product thus obtained has characteristics similar to those obtained as the product of Example 10.

Example 12
33.6 gr. 2-nitro-o-anisidine are slurried in 46 cc. of water, 53 cc. of 21° Bé. HCl are added to the slurry and the resulting solution is cooled to 5° C, after which 13.8 gr. sodium nitrite are added as a 33% solution. This diazotization solution is then salted to saturation, clarified by treatment with charcoal and filtered. To the filtered solution there are added 9 gr. of sulfate of...
mixed fatty acid monoglycerides known as Arctic Syntex M. (Young and Coon's Surface Active Agents, p. 31) is dissolved in 30 cc. of water. To this solution there are added 7 gr. of ZnCl₂ in a concentrated aqueous solution. The thus stabilized diazonium salt is then salted out, filtered, sucked dry, standardized and dry mixed with sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The product thus obtained has a coarser crystalline structure than that obtained in a similar manner but without the addition of the sulfated mixed fatty acid monoglyceride.

**Example 13**

33.6 gr. 2-nitro-p-aminodisine are diazotized in the same manner as in the preceding example. To the filtered diazo solution there are added 7 gr. of the sodium sulfate ester of isooctyl phenyl monoglycerol ether dissolved in 20 cc. of water. A concentrated aqueous solution of 9.5 gr. of cadmium chloride is then added. The resulting product is salted out and finished in the same manner as the product of the preceding example. It has similar characteristics to the product of that example.

**Example 14**

2-nitro-p-aminodisine is diazotized in the same manner as in Example 12. To the filtered diazo solution there are added 5 gr. of the steareic acid soap of 2-amino-butanol followed by the addition of 7 gr. ZnCl₂ in a concentrated aqueous solution. The resulting product is salted out and finished in the same manner as in the case of Example 12. Its properties are similar to those of the product of Example 12.

**Example 15**

The following mixture is slurried for 1 hr.: 24.4 gr. dianisidine 532 cc. water 63 cc. 21° Bé HCl The mixture is cooled to 5° C. and tetrazotized in 3-4 hr. with 13.8 gr. sodium nitrite as a 33% solution. The temperature should be held below 10° C. An excess nitrite test is maintained for 30 min. The diazonium solution is clarified by charcoal treatment followed by filtration. 9 gr. of sodium lauryl sulfate are added and a concentrated aqueous solution of 7 gr. of ZnCl₂. The stabilized diazo is salted out with an amount of NaCl equivalent to 10% of the volume, filtered, sucked dry, and dry mixed and standardized by the addition of sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The crystalline product thus obtained is similar in characteristics to the product of Example 10.

**Example 16**

The following mixture is slurried for 1 hr.: 42.8 gr. 4'-methoxy-4'-amino-phenylamine 555 cc. water 107.5 cc. 21° Bé HCl The mixture is cooled to 0° C. and 13.9 gr. sodium nitrite are added as a 15% solution. The temperature is held below 10° C. for several hours. An excess nitrite test is maintained for 30 minutes. The diazonium salt crystallizes out. This slurry is heated for 1 hr. at 50° C. to complete solution, then purified by charcoal treatment and filtered. 7.5 gr. diamyl ester of sodium sulfosuccinate in 20 cc. water are added while still warm. The stabilized diazonium salt thus obtained is salted out by the addition of an amount of NaCl equivalent to 15% of the volume while cooling to about 10° C. The precipitate is filtered, sucked dry, and standardized and dry mixed with sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The crystalline product thus obtained has characteristics similar to those obtained as the product of Example 10.

**Example 17**

The following mixture is slurried for 1 hr.: 51.2 gr. 4'-amino-6'-methyl-m-benzaniside 63 cc. 21° Bé HCl 160 cc. water The mixture is cooled to 5° C. and diazotized in 3-4 hr. with 13.8 gr. sodium nitrite as a 33% solution. The temperature should be held below 15° C. An excess nitrite test is maintained for 30 minutes. The diazonium solution is clarified by charcoal treatment followed by filtration. 18 gr. sulfonated castor oil are added to the diazo solution and a solution of 7 gr. ZnCl₂ in a small amount of water. The stabilized diazo is salted out with NaCl equivalent to 10% of the volume, filtered, sucked dry and dry mixed and standardized by the addition of sodium sulfate, aluminum sulfate and calcined magnesium sulfate. The product has characteristics similar to the product of Example 10.

We claim:

1. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, and zinc chloride, said complex diazonium salt combination having a crystalline form which retains less mother liquor in a filter cake obtained by filtration of an aqueous suspension thereof than a filter cake of the corresponding aromatic diazonium salt combined as a complex solely with zinc chloride.

2. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, and sodium chloride, said complex diazonium salt combination having a crystalline form which retains less mother liquor in a filter cake obtained by filtration of an aqueous suspension thereof than a filter cake of the corresponding aromatic diazonium salt combined as a complex solely with sodium chloride.

3. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, and 1,5-naphthalene sulfonic acid, said complex diazonium salt combination having a crystalline form which retains less mother liquor in a filter cake obtained by filtration of an aqueous suspension thereof than a filter cake of the corresponding aromatic diazonium salt combined as a complex solely with 1,5-naphthalene sulfonic acid.

4. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of sulfonated castor oil and zinc chloride.

5. A stable, dry, non-dusting diazonium com-
position comprising an aromatic diazonium salt in complex combination with a mixture of sodium oleate and 1,5-naphthalene disulfonic acid.

6. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of the sodium sulfate ester of isooctylphenyl glycol ether and cadmium chloride.

7. In a process for the preparation of a stable diazonium salt in solid form which involves precipitation of an aromatic diazonium salt from aqueous solution with a Fast Color Salt stabilizer, the improvement which comprises adding to the aqueous solution of said diazonium salt, prior to said precipitation, a substantial amount of an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, and effecting the aforesaid precipitation of the stable diazonium salt in solid form in the presence of said surface-active agent, to form a complex aromatic diazonium double salt partly with said anionic surface-active agent and partly with said Fast Color Salt stabilizer.

8. In a process for the preparation of a stable diazonium salt in solid form which involves precipitation of an aromatic diazonium salt from aqueous solution with a Fast Color Salt stabilizer, the improvement which comprises adding to the aqueous solution of said diazonium salt, prior to said precipitation, an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, the surface active agent being added in an amount equal to at least 5% of the amount of the diazonium salt, and effecting the aforesaid precipitation of the stable diazonium salt in solid form in the presence of said surface-active agent, to form a complex aromatic diazonium double salt partly with said anionic surface-active agent and partly with said Fast Color Salt stabilizer.

9. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of a Fast Color Salt stabilizer and an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, said complex diazonium salt composition having a crystalline form which retains less mother liquor in a filter cake obtained by filtration of an aqueous suspension thereof than a filter cake of the corresponding aromatic diazonium salt combined as a complex solely with said Fast Color Salt stabilizer.

10. A stable, dry, non-dusting diazonium composition comprising an aromatic diazonium salt in complex combination with a mixture of a Fast Color Salt stabilizer and an anionic surface-active agent containing at least one long chain alkyl group and having its hydrophilic group aliphatically linked, the surface-active agent being present in an amount equal to from 5 to 20% of the amount of the diazonium salt, said complex diazonium salt combination having a crystalline form which retains less mother liquor in a filter cake obtained by filtration of an aqueous suspension thereof than a filter cake of the corresponding aromatic diazonium salt combined as a complex solely with said Fast Color Salt stabilizer.

WILLIAM H. VON GLAHN.
HERMAN A. BERGSTROM.

REFERENCES CITED
The following references are of record in the file of this patent:

UNITED STATES PATENTS

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<tr>
<th>Number</th>
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<td>2,107,159</td>
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FOREIGN PATENTS

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