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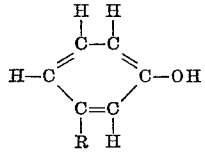
DIAZOTYPE MATERIALS AND METHODS FOR PRODUCING DIAZOTYPE IMAGES

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ABSTRACT OF THE DISCLOSURE

A method for producing high opacity diazotype images whereby a material containing a light-sensitive diazonium compound is selectively exposed to light so as to decompose said compound in the portion of said material where the light impinges and whereby substantially anhydrous ammonia is applied at a pressure of about 35 p.s.i.g. to 985 p.s.i.g. so as to couple said undecomposed diazonium compound with a coupler having the formula



wherein, in a specific compound, R is other than hydrogen only at the 2,3-, 2,5- or 2,6-positions and R is a radical selected from the group consisting of hydrogen, aryl, alkyl, alkene and aralkyl radicals, and said radicals unsubstituted or substituted by a radical selected from the group consisting of carboxy, hydroxy, carbonyl and amino, thereby producing a high opacity image.

This invention relates to diazotype materials and to methods for producing diazotype images. In particular, the invention is concerned with diazo materials capable of yielding high ultraviolet opacity images upon development by the procedure described in United States patent application Ser. No. 369,861, filed May 25, 1964, by Boone et al., for Diazo Development Process. This patent application is directed to the development of diazo images by the application of high pressure ammonia, especially high pressure anhydrous ammonia.

An object of the present invention is to provide diazotype compositions, papers, films and the like which yield high opacity images upon development by high pressure ammonia.

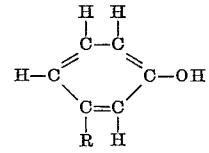
A further object of the invention is to provide diazo materials which exhibit good stability and shelf life, but which are capable of very rapid development by high pressure ammonia.

In general, the invention comprises diazo materials incorporating a meta-monosubstituted phenol coupler. The invention further comprises the production of high opacity images by high pressure ammonia development of diazo materials incorporating a meta-monosubstituted phenol coupler.

While the invention is generally applicable to one or two component systems, it is particularly valuable in two component systems. Such systems generally comprise a substrate provided with a coating containing, as the essential ingredients, a light-sensitive diazonium compound and a coupler, in this instance a meta-monosubstituted phenol. The coating may also contain an amount of an acid to stabilize the composition against premature coupling. Other coupling compounds may also be added to give a dye image of the desired color upon development.

The film or paper element is selectively exposed to light and is then developed by being contacted with high pressure ammonia. The developing agent is preferably anhydrous ammonia.

The couplers incorporated in the diazo materials of this invention have the following general formula:



where R is a radical selected from the group consisting of aryl, alkyl, alkene or aralkyl radicals. The radicals themselves may be unsubstituted or substituted by a carboxy, hydroxy, carbonyl or amino group and derivatives of any of these groups.

Specific compounds of the foregoing general formula, illustrative of those useful in this invention, include, for example, those in Table I.

TABLE I

m-Cresol
m-Ethylphenol
m-Isopropylphenol
m-Tertiarybutylphenol
m-Pentadecylphenol
m-Cyanophenol
m-Cresylglutaric acid
m-Hydroxy phenethyl alcohol
3,3'-dihydroxydibenzylamine
3-(N-methylaminomethyl)phenol
3-(β-hydroxyethyl)phenol
Methyl (m-hydroxyphenyl)carbamate
m-Hydroxyphenylpropionic acid
N-(β-hydroxyethyl)-m-hydroxyphenylpropionamide
2-hydroxy-m-hydroxyphenylacetic acid
N-(β-hydroxyethyl)-3-hydroxycinnamide
m-Hydroxy-N-proplybenzylamine
m-Hydroxy-N-butylbenzylamine
m-Hydroxy-N-phenylbenzylamine
m-Hydroxyphenylsulfonamide
m-Hydroxyphenylsulfonic acid

In a preferred embodiment of the invention, an organic solution is prepared containing an appropriate light-sensitive diazonium salt and a meta-monosubstituted phenol of the type described. A coating of the solution is deposited onto a suitable substrate and is dried. The substrate may comprise a transparent film support of cellulose acetate, a paper web or any other suitable support material.

Any of the conventional, well known, light-sensitive diazonium compounds may be employed in preparing the diazo materials of the invention.

The resulting diazo material is then selectively exposed in light to decompose the light-sensitive diazonium compound in those areas where no image is desired.

The exposed diazo element is then developed by being subjected to high pressure ammonia, preferably anhydrous ammonia. Application of the ammonia brings about very rapid coupling of the undecomposed portion of the diazonium compound and the coupler compound. The anhydrous ammonia is applied at a pressure significantly in excess of atmospheric pressure. For example, a pressure in the range of from about 35 to 985 p.s.i.g. absolute is satisfactory. With diazo materials of the type described, development is substantially complete in about 1 second or less upon exposure to high pressure ammonia, preferably anhydrous ammonia.

The following detailed examples of preferred embodiments of the invention will assist toward a fuller understanding of the invention.

EXAMPLE 1

As a specific example of the diazo materials and images forming process of the invention, diazo elements are produced and images formed in the following manner.

A solvent solution is prepared by mixing 30 parts by volume of methyl ethyl ketone with 40 parts by volume of methyl Cellosolve. The following ingredients are then dissolved in the solvent solution in the indicated percentages by weight:

	Percent
Citric acid	2
Sulfosalicylic acid	1
p-Dimethylaminobenzene fluoborate	1.5
3-(β-hydroxyethyl)phenol	2.0

A thin coating of the resulting solution is deposited onto a transparent cellulose actate substrate and the coating is allowed to dry. The element is then exposed to light through a negative for from about 3 to 5 seconds and is developed by contact with anhydrous ammonia at a pressure of 80 p.s.i.g. for 1.0 second. This produces a fully developed, high opacity, image.

EXAMPLE 2

The invention may also be practiced using diazo formulations comprising a resinous carrier or vehicle. Materials of this type are prepared and images are produced from such materials in the following manner:

A solvent solution is prepared by mixing 33 parts by volume of methyl ethyl ketone with 44 parts by volume of methyl Cellosolve. The following ingredients are then dissolved in the solvent solution in the indicated percentages by weight:

	Percent
Bakelite AYAT	1.12
Rohm & Haas B–82 Acryloid	0.75
Sulfosalicylic acid	1.0
Citric acid	2.0
p-Dimethylaminobenzene fluoborate	1.5
3-(β-hydroxyethyl)phenol	2.06

A thin coating of the resulting solution is deposited onto a transparent cellulose acetate substrate and is dried, exposed and developed as in Example 1. A fully developed, high opacity image is produced.

Measurements were made on representative formulations to demonstrate clearly the impracticability of attempting to use these couplers in conventional aqua ammonia development systems and their eminent suitability when developed under high pressure anhydrous ammonia. The "Ozamatic" of the General Aniline & Film Corporation was used for aqua ammonia development. Repeated passes were made in an attempt to obtain complete development. Anhydrous ammonia development was made at 80 p.s.i.g. for 1.0 second in all cases. Comparative data are tabulated in Table II.

TABLE II

Coupler	Anhydrous ammonia, 1.0 sec. at 80 p.s.i.g.	Ozamatic		
		No. of passes	Total dwell time	D_{7-51}
3-(β-hydroxyethyl)phenol	D_{7-51}=0.40	40	160	0.37
m-Hydroxyphenylpropionic acid	D_{7-51}=0.60	10	40	0.58
3-(m-hydroxyphenyl) propylbromide	D_{7-51}=0.47	10	40	0.41

It will be noted that in neither of the cases of the representative examples cited did aqua ammonia development produce an optical density equivalent to that produced by anhydrous ammonia at 80 p.s.i.g. for one second.

Although this invention has been generally described in terms of developing two component elements, that is, elements containing both a diazonium compound and a coupling compound in the same layer, it should be readily understood, that this invention has general applicability to one component system as well. In developing the one component system, the coupler is contacted with said undecomposed diazonium compound by application to the diazonium-containing element after exposure.

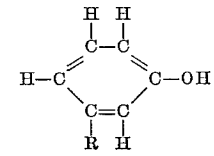
It will be obvious to those skilled in the art that various modifications may be made in the compositions and method illustratively described herein without departing from the spirit and scope of the invention as expressed in the following claims.

What we claim is:

1. A method for producing high opacity diazotype images comprising

selectively exposing to light a layer of material containing a light-sensitive diazonium compound so as to decompose said compound in the portion of said layer where the light impinges and where no azo dye formation is desired, and

applying substantially anhydrous ammonia at a pressure of 35 p.s.i.g. to 985 p.s.i.g. to said undecomposed diazonium compound, so as to couple said diazonium compound with a coupler compound having the formula



where R is a radical selected from the group consisting of aryl, alkyl, alkene and aralkyl radicals and said radical is unsubstituted or substituted by a radical selected from the group consisting of carboxy, hydroxy, carbonyl and amino, thereby producing a high opacity image.

2. The method of claim 1 wherein said undecomposed diazonium compound is contacted with said coupler by mixing said coupler compound with said light-sensitive diazonium compound in said layer prior to exposure.

3. The method of claim 1 wherein said coupler is contacted with said undecomposed diazonium compound by application to said layer after exposure.

4. The method of claim 1 wherein R is an aryl radical.

5. The method of claim 1 wherein R is an alkyl radical.

6. The method of claim 1 wherein R is an alkene radical.

7. The method of claim 1 wherein R is an aralkyl radical.

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