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2,949,359

PROCESS FOR DEVELOPING SILVER HALIDE EMULSIONS

Elkan R. Blout, Belmont, Milton Green, Newton Highlands, and Myron S. Simon, Newton Center, Mass., assignors to Polaroid Corporation, Cambridge, Mass., a corporation of Delaware

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The present invention is related to chemistry and more particularly to novel chemical compounds and to novel photographic processes utilizing said compounds as developing agents.

One object of this invention is to provide novel chemical compounds and suitable syntheses for their preparation.

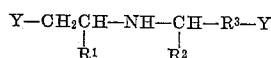
Another object of this invention is to provide novel chemical compounds useful as photographic developing agents and as intermediates.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the several steps and the relation and order of one or more of such steps with respect to each of the others, and the product possessing the features, properties and the relation of elements which are exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description.

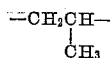
The novel compounds of this invention may be represented by the formula:



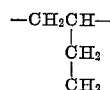
wherein each Y may be an unsubstituted, an alkyl-substituted or a halogen-substituted ortho or para dihydroxyphenyl group; R¹ is a hydrogen, methyl or ethyl group; R² is a hydrogen or alkyl group and preferably an alkyl group containing no more than 5 carbon atoms such as a methyl, ethyl, propyl, etc. group; and R³ is an alkylene group and preferably an alkylene group containing no more than 5 carbon atoms, such as

methylene —CH₂—, ethylene —CH₂CH₂—,

propylene —CH₂CH₂CH₂—, isopropylene

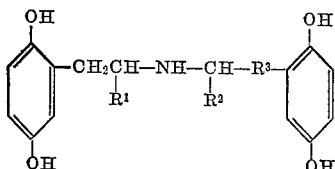


1,4-butylene —CH₂CH₂CH₂CH₂—, 1,2-butylene



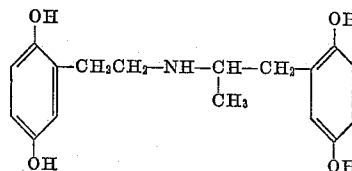
etc.; and the acid addition salts thereof, such as the hydrohalides, e.g., hydrochloride or hydrobromide, or organic acid salts, such as the oxalates.

In a preferred embodiment, each Y is a p-dihydroxyphenyl group. Such compounds may be represented by the formula:



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The preferred compound of this invention is:



α -Methyl- β,β' -bis-(2,5-dihydroxyphenyl)-diethylamine

The novel compounds of this invention may be prepared by condensing an amine of the structure:



wherein R¹ is the same as above and Y¹ is a dialkoxy derivative of Y, with an appropriate aldehyde or ketone of the structures:

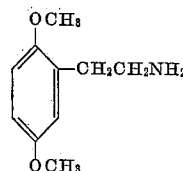


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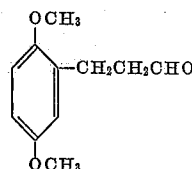


wherein R², R³, and Y¹ are the same as above, hydrogenating the resulting unsaturated compound and removing the alkoxy groups by hydrolysis.

As an example of an amine compound within Formula A, mention may be made of β -(2,5-dimethoxyphenyl)-ethylamine of the structure

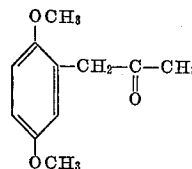


Dialkoxyphenyl-acetones coming within Formula C may be prepared by condensing a suitable dialkoxybenzaldehyde with nitroethane and converting the resulting product to the ketone by means of iron and hydrochloric acid. Other ketones and aldehydes within Formulae B and C may be prepared by reacting a suitable dialkoxybenzaldehyde with an aliphatic aldehyde such as acetaldehyde, propionaldehyde, etc. or with an aliphatic ketone such as acetone, ethyl-methyl ketone, etc. and hydrogenating the resulting product. As examples of useful aldehydes and ketones within Formulae B and C, mention may be made of:



β -2,5-dimethoxyphenylpropionaldehyde

and



2,5-dimethoxyphenylacetone

The following nonlimiting example illustrates the preparation of a novel compound within the scope of this invention.

Example 1

19 gms. of 2,5-dimethoxyphenethylamine and 23 gms.

of 2,5-dimethoxyphenylacetone in 15 gms. of ethanol are hydrogenated at 75° C. and 900 p.s.i. pressure for two hours in the presence of a platinum dioxide catalyst. The catalyst is removed by filtration, the alcohol is driven off, and the residue is distilled to yield 20.5 gms. of a viscous colorless liquid having a boiling point at 197° C. at 0.2 mm. pressure. Nitrogen analysis of this product gives a value of 5.5% as compared with the theoretical value of 5.2%.

20.5 gms. of the above liquid is refluxed for four hours, under nitrogen, with 250 cc. of hydrogen bromide. The excess acid is removed in vacuo, water is added and the evaporation is repeated. The product is dried in a desiccator over potassium hydroxide yielding 19 gms. of α -methyl- β,β' -bis-(2,5-dihydroxyphenyl)-diethylamine hydrobromide, melting at 152 to 154° C.

As pointed out previously, the novel compounds of this invention are useful as photographic developing agents. As an example of a suitable developing composition utilizing one of the novel compounds of this invention, mention may be made of the following composition:

	Gm.
Potassium bromide	0.2
Sodium hydroxide	3.0
α -Methyl- β,β' -bis-(2,5-dihydroxyphenyl)-diethyl- amine hydrobromide	1.0
Water to make 100 cc.	

It will be understood that it is within the scope of this invention to vary the above composition to suit particular needs. The percentage of materials may be varied and additional materials useful in photographic processes may be added. If the composition is to be spread on a silver halide emulsion, a film-forming thickening agent such, for example, as sodium carboxymethyl cellulose, may be added.

The novel compounds of this invention are useful as developing agents in diffusion transfer processes, both dye and silver transfer, and are especially useful in such photographic processes wherein it is desired to eliminate or minimize the need for washing or stabilizing operations in liquid baths subsequent to the formation of the silver print. Examples of such processes are disclosed in U.S. Patent No. 2,647,056 to Edwin H. Land. The utility of the developers of this invention is by no means limited to diffusion transfer reversal processes, for they may be satisfactorily employed in conventional multi-stage multibath photographic processes in black-and-white or color photography. Where the herein disclosed compounds are employed in diffusion transfer processes of the type disclosed in the said patent to Edwin H. Land, the novel developer composition may have incorporated therein as a further ingredient a silver halide solvent such as sodium thiosulfate. Thus, by adding sodium thiosulfate to the composition illustrated above and using

an image-receiving element in conjunction with the photosensitive element, a transfer image may be obtained.

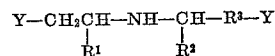
The novel compounds of this invention are again useful as intermediates. As an example of such use, mention may be made of the dye developers claimed and disclosed in the copending application of Elkan R. Blout and Myron S. Simon, Serial No. 680,437, filed August 26, 1957, wherein the compounds of this invention are used in the syntheses of said dye developers.

The novel compounds of this invention are further useful as antioxidants in petroleum products, etc.

Since certain changes may be made in the above product and process without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. A process for developing a photosensitive silver halide emulsion which comprises treating an exposed silver halide emulsion with an aqueous alkaline solution of a compound of the formula:



wherein Y is selected from the group consisting of unsubstituted, alkyl-substituted and halogen-substituted ortho and para dihydroxyphenyl groups; R¹ is selected from the group consisting of hydrogen, methyl and ethyl groups; R² is selected from the group consisting of hydrogen and alkyl groups; and R³ is an alkylene group, for a sufficient time to develop the latent image to silver.

2. A process as defined in claim 1 wherein each Y is a p-dihydroxyphenyl group.

3. A process as defined in claim 1 wherein R² is a methyl group, and R³ is a methylene group.

4. A process as defined in claim 1 wherein R¹ is hydrogen.

5. A process as defined in claim 1 wherein said compound is α -methyl- β,β' -bis-(2,5-dihydroxyphenyl)-diethylamine.

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