

UNITED STATES PATENT OFFICE.

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SENSITIVE LAYER ON A SUITABLE BASE AND PROCESS OF MAKING SAME.

No Drawing. Application filed June 19, 1925, Serial No. 38,288, and in Germany May 28, 1924.

In U. S. Patent No. 1,444,469 of Gustav Kogel, filed September 12, 1922, a process is described for the production of layers sensitive to light and of great technical value, particularly for making light copy paper, by means of the so-called diazo-anhydrides. By the application of these layers it is possible to obtain directly positive pictures from positives.

10 In U. S. Patent No. 1,444,469 it is set out that diazo compounds can be applied on the base simultaneously with the azo-dye-stuff components, without impairing thereby the effect of the picture to be produced.

15 Further it has been shown that the coupling of the diazo- and azo-components can be effected by means of a gaseous alkali, for instance ammonia, which permits the development of the picture without having to use any liquid.

20 It has been proposed to use an acid for preventing a premature coupling of the components during their storage. Thus it is possible to apply diazo compounds of different kind together with azo components. If diazo compounds other than diazo-anhydrides are chosen, it is necessary to add thereto a considerable quantity of an acid, for instance sulphuric acid, in order to prevent a premature coupling of the components, but this addition impairs considerably the durability of the paper-fibre. Moreover the simple diazo compounds involve the disadvantage that they are scarcely colored so that it is very difficult to see when the bleaching is finished.

30 Now we have found that there can be obtained papers which are equally durable or nearly as durable and capable of being stored as those described in U. S. Patent No. 1,444,469 without having to add a considerable quantity of an acid, by using as diazo components compounds which may be regarded as diazo anhydrides in a broad sense and which may be presumed to have been produced from diazonium compounds with elimination of water. We prefer to use diazo compounds formed from amino compounds containing at least one other amino group substituted or not.

These diazo compounds possess, like the diazo anhydrides already mentioned, the

necessary stability and they are also more or less colored.

As such diazo components there may be used amino-diazo compounds, for instance the diazo compounds from p-diamines and derivatives thereof and also aminodiphenylamines such, for instance as phenyliminoquinonediazide (comp. Hantzsch & Reddelin, "Die Diazoverbindungen" Berlin 1921, page 60 ff.), dialkylamino-o-aniline and its sulphonic acid.

For our new process there may also be used the diazo compounds obtained from aminocarbazoles, being also amino compounds, in which one of the amino groups is substituted by carbon atoms standing in a closed nucleus, aminofluorene, aminodiphenyleneoxide and similar stable diazo bodies, which are also colored and behave in a like manner.

As described in the above mentioned U. S. Patent No. 1,444,469 metallic salts may simultaneously be added to the layer sensitive to light, and the new process may be carried out according to any of the various methods described in the previous patent. Instead of incorporating the metallic salts into the light-sensitive layer there may also be used the metal compounds of the diazo- or azo-components in question or of both components in case they contain salt-forming groups or, when combined with metal salts, form double salts. In this case it is advantageous to develop the picture after exposure to light by means of a gaseous alkali, which latter may be produced according to any known method. The development of the picture may however also be effected in an aqueous alkaline bath and then the metal salts added to the alkali bath.

The other amino groups of the amino compounds, from which the diazo compounds are formed, can be substituted or not.

The diazo anhydrides already claimed in U. S. Patent No. 1,444,469 are excluded from what we claim in our present application.

The following examples illustrate our invention:

1. 13.6 parts by weight of o-aminodimethylaniline are diazotized in the usual manner and in this diazo solution, which

has a deep yellow color, 8 parts of resorcylic acid and 15 parts of nickel sulfate are dissolved. The solution is made up to one liter and used for being spread on the desired base (paper).

The layers obtained after drying yield, when exposed to light and treated with an alkali, brownish-orange copies.

2. 10 parts by weight of phenyliminoquinonediazidesulfate, 9 parts by weight of resorcylic acid and 30 parts by weight of nickel sulfate ($\text{NiSO}_4 + 6\text{H}_2\text{O}$) are dissolved in 1000 parts by volume of water and the base is painted over with this solution.

15 The layers so obtained are exposed to light in the usual manner and by then treating them with an alkaline agent, such as gaseous ammonia or sodium carbonate, immediate brown positive copies are produced.

20 3. 10 parts by weight of p-iminoquinonediazidesulfonic acid, which is obtained by diazotizing p-phenylenediaminesulfonic acid, are dissolved in 1000 parts by volume of water together with 20 parts of 1-amino-8-oxynaphthalene-3,6-disulfonic acid (H -acid) and the base is painted over with this solution.

The layers so obtained after being exposed to light and subsequently developed with an alkaline agent, for instance gaseous ammonia, yield dark bluish-violet positive copies.

30 4. 12 parts by weight of dimethyliminoquinonediazidesulfonic acid, which may be obtained by diazotizing p-dimethylaminoaniline sulfonic acid, and 20 parts by weight of β -naphthol-3,6-disulfonic acid are dissolved in 1000 parts by volume of water and the base is painted over with this solution.

In the same way as described in Example 2, brown positive copies are obtained.

45 5. 10 parts by weight of phenyliminoquinonediazidesulfate, obtained by diazotizing p-aminodiphenylamine, are dissolved in 1000 parts by volume of water together with 20 parts by weight of β -naphthol-3,6-disulfonic acid and applied upon a suitable base for instance paper. The layers so obtained when treated as indicated in Examples 3 and 4 yield dark violet positive copies.

50 6. 9 parts by weight of 3-aminocarbazole are diazotized in the usual manner and in this diazo solution are dissolved: 8 parts by weight of resorcylic acid and 15 parts of nickel sulfate. The resulting solution is

made up to one liter and spread on a base. The layers so obtained, after being dried, exposed to light under a positive or negative and developed with an alkali, yield brown copies.

We do not claim any diazo compounds described and claimed in U. S. patent of Kogel No. 1,444,469, filed Sept. 12, 1922.

We claim:

1. Process for producing sensitive layers on a suitable base, consisting in covering the base with a diazo compound being formed from amino compounds containing at least one other amino group and an azo dyestuff component.

2. Process for producing sensitive layers on a suitable base, consisting in covering the base with a diazo compound being formed from amino compounds containing at least one other amino group, an azo-dyestuff component and a metal salt.

3. Process for producing sensitive layers on a suitable base, consisting in covering the base with a diazo compound being formed from amino compounds containing at least one other amino group substituted or not, an alkali and a metal salt.

4. As new products, sensitive layers on a suitable base, consisting of diazo compounds being formed from amino compounds containing at least one other amino group, and an azo-dyestuff component.

5. As new products, sensitive layers on a suitable base, consisting of diazo compounds being formed from amino compounds containing at least one other amino group, and a metal salt.

6. As new products, sensitive layers on a suitable base, consisting of diazo compounds being formed from amino compounds containing at least one other amino group, and an alkali.

7. As new products, sensitive layers on a suitable base, consisting of diazo compounds being formed from amino compounds containing at least one other amino group, an azo dyestuff component and a metal salt.

8. As new products, sensitive layers on a suitable base, consisting of diazo compounds being formed from amino compounds containing at least one other amino group, an alkali and a metal salt.

In testimony whereof we hereunto affix our signatures.

Dr. MAXIMILIAN PAUL SCHMIDT.
Dr. WILHELM KRIEGER.

DISCLAIMER

1,628,279.—*Maximilian Paul Schmidt* and *Wilhelm Krieger*, Biebrich-on-the-Rhine, Germany. SENSITIVE LAYER ON A SUITABLE BASE AND PROCESS OF MAKING SAME. Patent dated May 10, 1927. Disclaimer filed February 3, 1940, by the assignee, *Kalle & Co. Aktiengesellschaft*.

Hereby disclaims claims 3, 5, 6, and 8 of said Letters Patent,

The comma and words “, aminofluorene, aminodiphenyleneoxide”, appearing on page 1, lines 69 and 70 of said Letters Patent, and

From the scope of claims 1, 2, 4, and 7 of said Letters Patent, those diazo compounds being formed from amino compounds containing at least one other amino group which compounds may not be regarded as diazo anhydrides in a broad sense and which compounds may not be presumed to have been produced from diazonium compounds with elimination of water.

[*Official Gazette February 27, 1940.*]