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SINGLE POWDER DEVELOPERS HAVING IMPROVED PHYSICAL CHARACTERISTICS AND METHOD OF FORMING THE SAME

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This invention relates to single powder photographic developers, and more particularly to single powder developer compositions, containing stabilizing materials, which compositions have been treated by a novel method to impart improved stability to the single powder developers over a wide range of elevated temperatures.

Single powder developer mixtures may contain an organic developer component or mixtures of such components, including monomethyl paraminophenol sulfate (elon or metol), hydroquinone, p-aminophenol hydrochloride, p-aminophenol sulfate, pyrogallol, parahydroxyphenyl glycine, catechol, diaminophenol hydrochloride, and others, with an alkaline component, a stabilizing component, an oxidizing preventer and an antifogging component. The alkali component may be the stable hydrate of an alkali carbonate, a desiccated alkali carbonate, or an alkali sulfite. Generally the stable alkali carbonate monohydrate is preferred. The oxidation preventing component may be an alkali sulfite or bisulfite and the anti-fogging component may be potassium bromide or iodide. Stabilizing components for such a single powder developer are disclosed in Patent 2,384,592 of September 11, 1945, and include maleic anhydride, salicylic acid, alkali metabisulfite, benzoic anhydride, orthobenzoic sulfimide, phatalimide, and phthalic anhydride. Of these stabilizers phthalic anhydride has outstanding stabilizing properties in the presence of either sodium carbonate or borax and has been employed considerably more than the other stabilizers mentioned in that patent.

Phthalic anhydride has been employed most successfully in developer compositions having as the alkali component either sodium carbonate or sodium sulfite, or mixtures thereof.

Possibly the greatest disadvantage of employing phthalic anhydride in single powder developer mixtures, which contain alkali carbonates and sulfite, is that phthalic anhydride, when used alone in sufficient amounts for stabilizing purposes, may react due in part to temperature effects with these basic substances and form gas under sufficient pressure to distend the hermetically sealed can in which the developer powder is packaged, and often cause a portion of the powder to be ejected with some violence as the package is opened by removing the rip strip from the can.

A second objection to using phthalic anhydride as a stabilizer in such single powder developers in the manner described in Patent 2,384,592 is that it has a relatively slow rate of solution as

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compared to the solution rate of the other ingredients of the composition. This rate is improved somewhat by employing phthalic anhydride which has been finely ground so as to pass a 100 mesh screen size. This fine phthalic anhydride is found to dissolve approximately as rapidly as do the residual components, but it has a tendency to float to the top of the liquid and create an unsightly scum while being dissolved.

An object, therefore, of the present invention is to provide an improved method for treating stabilized single powder photographic developers to improve their physical properties.

Another object of this invention is to provide an improved method for treating single powder developers which contain phthalic anhydride as the stabilizing agent whereby the tendency of this anhydride to form gas pressure when the single powder is enclosed in sealed containers is substantially diminished.

A further object of this invention is a single powder photographic developer which is stabilized with phthalic anhydride and which has on heating up to 130° F. a gas pressure of not more than 3 cm. of mercury.

In accordance with the present invention we have discovered that the objections to employing phthalic anhydride in single powder photographic developers may be overcome by preheating the single powder developer composition before it is packaged in the hermetically sealed containers in which it is sold. We have found it is only necessary to bring the single powder mixture to a temperature approximately within the range of 130 to 140° F. for a few minutes to accomplish the desired results.

When a single powder mixture is heat treated in this manner, is packaged or canned, it has been found that negligible pressures, of the order of 1 to 3 cm. Hg, exist even after prolonged storage at 120° F. Indeed if the mixture is canned while still warm a slight vacuum is encountered. The difficulty in preparing solutions from these single powder developer mixtures has been found to be virtually eliminated by this novel treatment.

A further advantage of this preheating method has been found in the case of such single powder developers which are to be packaged in packets formed from laminated foils on automatic machines. A significant increase in keeping qualities has been discovered in the case of materials so packaged when the preheating has been employed.

This advantageous result may possibly be due to a mild agglomerating action taking place due to moisture released during the heating from hydrated components of the mixture. This would result in less dust in the mixture and would improve the quality of the packet seal since it is known that in the case of a dusty product it is practically impossible to obtain a perfect seal.

The following examples will further serve to illustrate developer mixtures which have been treated in accordance with our invention and which therefore possess novel properties not present in similar mixtures known heretofore.

Single powder preparations were made of these three formulas using phthalic anhydride as the protectant. Portions of these preparations were then heated to 130–140° F., allowed to cool somewhat and then packaged in sanitary cans. The unheated single powder preparations were packaged as checks on the effectiveness of the heating operation to eliminate pressure buildup.

Example 1

Elon	115 grains
Sodium sulfite	12 ozs.
Hydroquinone	1 oz., 30 grains
Sod. carbonate monohydrate	7 ozs.
Potassium bromide	290 grains
Phthalic anhydride	46 grains

One sample was heated to approximately 140° F., cooled and packaged. After 72 hours at 120° F., the sample, cooled to room temperature, showed a pressure of 2.5 cm. mercury. The check sample which was not heated before packaging showed a pressure of 23.0 cm. mercury after the same keeping time.

Example 2

Elon	175 grains
Sodium sulfite	6 ozs.
Hydroquinone	1 oz., 260 grains
Sod. carbonate monohydrate	10½ ozs.
Potassium bromide	115 grains
Phthalic anhydride	41 grains

A sample in a hermetically sealed can, after 24 hours keeping at 120° F., showed a pressure of 30 cm. mercury. A sample which had been heated to 130° F. and cooled prior to packaging showed a slight vacuum after a corresponding incubation.

Example 3

Elon	90 grains
Sodium sulfite	3 ozs.
Hydroquinone	350 grains
Sod. carbonate monohydrate	2¼ ozs.
Potassium bromide	90 grains
Phthalic anhydride	14 grains

In this case the untreated check sample developed a pressure of 6 cm. mercury whereas the sample which had been heated to 135° F. had a slight vacuum.

The packaging of the treated developer may be conducted at approximately room temperature i. e. 20° C. or at any temperature up to that of the heat treatment.

The heating of the single powder developer composition can be accomplished in a suitable manner. For example the powder can be conveyed through a screw feeding device having heated walls and an internally heated screw. A heated rotating cylinder may also be employed for continuous operation.

The heated jacketed screw has been employed successfully. Steam at approximately atmospheric pressure may be employed as the heating medium. Suitable temperature controls are employed in accordance with well known practices to maintain the temperature of the single powder developers in the range of 130–140° F. The apparatus is operated to heat a given portion of the powder for one to two minutes. Heating for one minute gives a satisfactory product. Longer periods of heating may be employed but appear to offer no particular advantage.

In every instance the pressure in the can of the preheated material was much lower than the pressure in the unheated can. The dissolving characteristics of the preheated powder were also much better than those of the unheated powder.

We claim:

1. A single powder photographic developer composition which has been heat treated for 1 to 2 minutes at a temperature within the range of 120–130° F. containing an organic silver halide developer, a preservative selected from the group consisting of alkali sulfite and alkali bisulfite, a buffer compound selected from the group consisting of the alkali salts of carbonic acid and phosphoric acid, an anti-fogging agent and a stabilizing compound comprising phthalic anhydride and having a vapor pressure in a sealed container of less than 3 cm. of mercury.

2. A single powder photographic developer composition which has been heat treated for 1 to 2 minutes at a temperature within the range of 120–130° F. containing an organic silver halide developer, a preservative selected from the group consisting of alkali sulfite and alkali bisulfite, a buffer compound selected from the group consisting of the alkali salts of carbonic acid and phosphoric acid, an anti-fogging agent selected from the group consisting of potassium bromide and potassium iodide and a stabilizing compound comprising phthalic anhydride and having a vapor pressure in a sealed container of less than 3 cm. of mercury if heated to 130° F.

3. A single powder photographic developer composition which has been heat treated for 1 to 2 minutes at a temperature within the range of 120–130° F. containing monomethyl p-aminophenol sulfate, hydroquinone, sodium sulfite, sodium carbonate monohydrate, potassium bromide and phthalic anhydride and having a vapor pressure in a sealed container of less than 3 cm. of mercury.

4. The method of improving the stability of single powder photographic developers containing a mixture of monomethyl p-aminophenol sulfate, hydroquinone, sodium sulfite, sodium carbonate monohydrate, potassium bromide and phthalic anhydride which comprises heat treating said mixture for one minute at a temperature within the range of 120–130° F.

No references cited.