

UNITED STATES PATENT OFFICE

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METHOD OF PREPARING DUSTLESS
PHOTOGRAPHIC FIXING POWDERSEdwin S. Wiitala, Rochester, N. Y., assignor to
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corporation of New JerseyNo Drawing. Application November 22, 1947,
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2 Claims. (Cl. 95—88)

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This invention relates to photographic fixing powders and more particularly to dustless substantially anhydrous fixing powders and the method for their preparation.

At the present time there is a considerable demand in the photographic trade for anhydrous fixing powders. These fixing powders are made up of a mixture of several chemicals, both inorganic and organic, many of which comprise such fine particles that they are inherently dusty and cause difficulty in handling during the preparation of the fixing powder as well as when the photographer employs them. In the latter case, unless special care is employed, the dust may fall upon other photographic elements in the vicinity and cause damage.

One method of preparing photographic fixing powders comprises thoroughly mixing several anhydrous chemicals in a conical blender. Among the chemicals usually found in the acid fixer the most prominent dust contributor is the anhydrous sodium thiosulfate. Other chemicals that produce dust particles include sodium acetate, sodium bisulfite, and boric acid. This dust condition presents a definite hazard to the photographic trade due to potential contamination of sensitive emulsions and darkroom areas, and consequently the photographer must exercise particular care when opening the package and employing the powder so as not to stir up dust.

The principal object of this invention is to obviate the above-mentioned dust defects of acid fixing powders and to provide a method of preparing these acid fixing powders which eliminates such dust from occurring in the fixing powder.

Another object is to provide an acid fixing powder containing substantially no dust-like particles.

A further object is to prepare a substantially anhydrous fixing powder which has improved solubility characteristics.

In accordance with the invention, these and other objects are attained by adding to the fixing powder mixture one or more components which contain water of hydration. The hydrated component is introduced into the mixture in a dry state and consequently can be dispersed uniformly throughout the fixing powder mixture. The mixture is then heated to a temperature suf-

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ficient to release water of hydration which is thus uniformly distributed throughout the fixing powder. If desired, only the particularly active dust-forming components need be thus treated, and other substantially dust-free components can be subsequently added to the composition.

This internal release of moisture apparently bonds the fine dust-like particles of the chemical to other particles of the mixture which are relatively larger. While the exact theory of the bonding action has not been established, possibly the bonding includes two different operations, which result in the granulated product. In one, the released water of hydration actually dissolves the small dust particles whose solution is deposited on other large crystals, and in this manner crystal growth is obtained and the dust particles eliminated. By the other operation, the hydrate crystals may form a nucleus of moisture on which the fine particles adhere. The product at any rate is agglomerated, whatever the mechanism, and may then be dried further to prevent caking and a possible sulfurization reaction on prolonged standing. The degree of drying is dependent upon the amount of moisture added as hydrate and that already present in the product.

The resulting fixing powder is about 99 percent dust free, whereas the same mixture without the moisture-releasing step is only 88 percent dust free. Since the moisture is uniformly distributed, there is less possibility of localized sulfurization reactions taking place as could be the case were the moisture added by other means. Drying is also more easily accomplished since there are no localized high concentrations of moisture present which may cause lumping and a non-uniformly dried product. My process has the advantages in that it is possible to control accurately the amount of moisture addition; a very uniform distribution of available moisture is had; the resulting product does not form a hard cake when put in solution; an increased rate of solution is experienced; the uniform moisture distribution affords a more uniform and complete drying, and a considerably more complete elimination of dust than results by other methods; on prolonged standing or aging in containers the granular product does not cake or

lump, as may be the case in wet binders; and the product has the appearance of a single powder chemical.

The following examples illustrate my novel process for preparing anhydrous acid fixing powders.

Example 1.—A substantially anhydrous dust-free fixing powder containing:

	Parts
Boric acid	5
Anhydrous sodium bisulfite	10
Anhydrous sodium thiosulfate	147
Anhydrous sodium acetate	22.5
Potassium alum	22.5
Sodium thiosulfate pentahydrate	20

is prepared in accordance with my invention as follows: A mixture containing the anhydrous sodium thiosulfate, the sodium thiosulfate pentahydrate, anhydrous sodium bisulfite, and sodium acetate is tumbled in a closed mixer which is adapted to be heated in 15 minutes to 120° F. to release the water of hydration of the sodium thiosulfate. This heating and tumbling eliminates dust particles and the remaining components of the formula are then thoroughly mixed into the composition. The resulting granular product is found to be 99.9 percent free of dust. A mixed sample of this formula without the dust-eliminating or heating step is only 88 percent free from dust. The treated composition was easily soluble in water, whereas the untreated composition tended to cake and dissolved slowly in water.

Example 2.—The formula shown in Example 1 is added together to a closed mixer and then mixed and heated in 20 minutes to 110° F. The mixture is then cooled and was found to be 99.9 percent free of dust. The resulting fixing powder was easily soluble in water.

Example 3.—The formula shown in Example 1 is mixed as follows in order to prepare a dustless fixing powder. The anhydrous sodium thiosulfate is preheated to 135° F. in the mixer and then the remaining components at a temperature of about 70° F. are added to the mixer and mixed for 15 minutes with the thiosulfate. The resulting temperature is about 110° F. which is sufficient to liberate some residual moisture in the chemicals as well as some water of hydration in the alum. The mix is not sulfurized and on cooling was found to be 99.0 percent free of dust.

Example 4.—To preheated anhydrous sodium thiosulfate, which has been heated in the mixer to 135° F., are added the other components in the fixing formula of Example 1 (excluding the sodium thiosulfate pentahydrate), except that 25 percent of the sodium acetate is in the form of sodium acetate trihydrate. Mixing was conducted at 110° F. for 20 minutes, and on cooling to room temperature the resulting fixing powder was found to be 99.9 percent dust free.

Example 5.—25 pounds of the acid fixing powder mixture containing:

	Parts
Boric acid	5
Anhydrous sodium bisulfite	12.5
Anhydrous sodium thiosulfate	290.0
Anhydrous sodium acetate	22.5
Sodium thiosulfate pentahydrate	9

were tumbled in a closed mixer for one hour at 130° F. The resulting tacky product was then dried for 14 hours in a tray at 125° F. under

vacuum. To the dried product was then added, with thorough mixing 22.5 parts of ammonium alum.

Dust measurements on this material showed it to be 99 percent free of dust.

Example 6.—25 pounds of an acid fixing powder mixture containing:

	Parts
Boric acid	5
Anhydrous sodium bisulfite	12.5
Anhydrous sodium thiosulfate	282
Anhydrous sodium acetate	30
Sodium thiosulfate pentahydrate	27

were tumbled in a closed mixer for one hour at 130° F. The resulting tacky product was then dried for 14 hours in a tray at 125° F. under vacuum. This mixture was uniformly blended with 20 parts of ammonium alum.

Dust measurement on this material showed it to be 99.9 percent free of dust.

The dustiness of the various fixing powders was determined in an air elutriation unit. This unit involves a slow flow of air at 18 ft. per minute through a 10-inch square duct into which is fed the material to be dust tested from a vibrating feed trough at the rate of 0.5 gm. per minute. The dustiest particles falling from the top of the duct will travel farther down the horizontal duct than the dustless particles before settling to its bottom. The rate of fall is dependent upon particle density and shape. The bottom of the duct is covered with aluminum trough-like plates to facilitate removal of the obtained fractions. The plates are graduated from the point of feed as follows: 1, 5, 14, 30, and 52 inches. This graduation results in plates of 1, 4, 9, 16, and 22 inches in length, respectively. It was found that particles falling on the last two plates are particularly objectionable. The particles have a flat cylindrical shape and have diameters of less than 1 to 80 microns.

It will be understood that in referring to dust particles I mean particles of one or more components of the fixing powder.

The fixing powder resulting from my invention may be termed substantially anhydrous and by that term I mean that the product contains only moisture resulting from the release of water of hydration from those components containing water of hydration. This amount of water may be as high as 10–15% of the composition, although generally only 5% is present. This water content compares favorably with the moisture in fixing powders made by former methods which moisture may be as high as 40%.

I claim:

1. The method of forming a substantially anhydrous dust-free photographic fixing powder which comprises thoroughly mixing 5 parts boric acid, 10 parts anhydrous sodium bisulfite, 147 parts anhydrous sodium thiosulfate, 22.5 parts anhydrous sodium acetate, 22.5 parts potassium alum, 20 parts sodium thiosulfate pentahydrate, said ingredients being in powdered form and comprising relatively large particles and relatively minute dust-like particles and then heating the mixture to release the water of hydration whereby the dust-like particles in the composition are bound to the larger particles of the powder.

2. The method of forming a substantially anhydrous, dust-free photographic fixing powder which comprises thoroughly mixing together in a closed mixer while heating to 110° F. in 20

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minutes, 5 parts boric acid, 10 parts anhydrous sodium bisulfite, 147 parts anhydrous sodium thiosulfate, 22.5 parts anhydrous sodium acetate, 22.5 parts potassium alum, 20 parts sodium thiosulfate pentahydrate, said ingredients being in a powder form and comprising relatively large particles and relatively minute dust-like particles, said heating causing the release of water of hydration from said pentahydrate, and resulting in binding said minute dust-like particles to the larger particles thereby substantially reducing the dustiness of the fixing powder.

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