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REMOVING SOLUBLE SALTS FROM PHOTOGRAPHIC EMULSIONS

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3 Claims. (Cl. 95—7)

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This invention relates to the production of photographic emulsions and particularly to a method for removing water-soluble salts from such emulsions.

It is well known that in the production of photographic emulsions made by precipitating a silver halide in the presence of a colloidal protein such as gelatin, the following steps are usually involved. (1) The emulsification and digestion or ripening of the silver halide, (2) the freeing of the emulsion from excess soluble salts usually by washing and (3) the second digestion or after-ripening to obtain sensitivity.

Photographic silver halide emulsions are made from solutions containing generally 10% or more of the soluble halide and a soluble silver salt such as silver nitrate. The addition of the silver nitrate to the solution of soluble halide is made under fixed conditions of temperature and rate of addition in the presence of a small amount of gelatin or other colloidal protein to produce the dispersion of silver halide required and the precipitated silver halide is then ripened by heat until the processes of growth and coalescence and crystallization of the silver halide are completed. When this stage is finished, sufficient gelatin is added so that on cooling, the emulsion sets to a firm jelly. It is then cut into small pieces and the soluble salts consisting of alkali nitrate produced from the reaction and excess of alkali halide are removed, generally by washing in chilled water until the proper concentration of soluble salts is achieved.

The washing operation removes the potassium nitrate and any excess of potassium bromide or other soluble salts present during the ripening operation. Careful maintenance of the conditions of the water used in washing and the time and temperature of washing are required to secure optimum results. This washing procedure is frequently long and tedious and involves an appreciable amount of the total time required for production of the emulsion.

It is, therefore, an object of the present invention to provide a novel method for removing the soluble salts from a silver halide emulsion. A further object of the invention is to provide a method for removing the soluble salts which does not require a long washing procedure. Other objects will appear from the following description of my invention.

These objects are accomplished by precipitating the gelatin or other colloidal protein of the emulsion together with the silver halide after the silver halide has been formed in the emulsion,

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with trichloroacetic acid, leaving the soluble salts in solution and then removing the precipitated gelatin or other protein together with the silver halide from the solution.

In the production of silver halide emulsions, a protective colloid such as gelatin or other colloidal protein, for example, glue, is used to retain the precipitated silver halide in suspension. As stated above, the silver halide is formed by mixing a soluble halide such as potassium bromide, with a soluble silver salt such as silver nitrate in the presence of a small amount of the gelatin or other colloidal protein. It is customary to use an excess of potassium bromide and this results in the formation of potassium nitrate and this salt together with the residual potassium bromide must be removed from the emulsion prior to the final digestion step. I propose to accomplish the removal of these soluble salts from the emulsion by precipitating the gelatin or other protein of the emulsion with trichloroacetic acid. When the gelatin or other protein is precipitated, it carries with it the silver halide and leaves the soluble salts in solution. The precipitated gelatin and silver halide can then be removed from the solution and treated further, for example, by a simple washing with water. The gelatin and silver halide can then be mixed with additional gelatin, followed by further digestion to form the finished emulsion.

The following example illustrates a method of removing soluble salts from an emulsion according to my invention.

A bromo-iodide emulsion was prepared employing the usual technique of emulsion making. To 2700 cc. of this emulsion containing 27 grams of gelatin were added 356 grams of trichloroacetic acid in 2200 cc. of water. The gelatin came down as a granular precipitate carrying with it the silver halide. In a very few minutes the supernatant liquid carrying a large portion of the salts with it could be decanted from the precipitate. The precipitate was then washed twice with approximately 2000 cc. portions of cold water, and after the precipitate had settled the water was then decanted from it. The pH was 2.5 after the second wash. The emulsion was then prepared for use by the addition of 15 cc. of 2.5 N sodium hydroxide, bringing the pH to a value of 6.3, stirring at 40° C. and melting in the additional gelatin needed.

The concentration of the solution of trichloroacetic acid used in my method is not critical. In general, trichloroacetic acid in amounts equal to at least 10 times the amount of gelatin or other

colloidal protein in the emulsion can be used. It is also possible to add the solid trichloroacetic acid directly to the emulsion.

In place of trichloroacetic acid, tannic acid may be used according to my invention.

My method of eliminating the soluble salts from a silver halide emulsion has a number of definite advantages. It provides for the elimination of the usual tedious washing procedure and permits the formation of a granular precipitate which is easily washed for a short time in water. It also permits the re-dispersion of the emulsion as a part of the final emulsion in a simple and convenient manner.

It will be understood that the modifications described herein are illustrative only and that my invention is to be taken as limited only by the scope of the appended claims.

I claim:

1. The method of removing water-soluble salts from a dispersion of silver halide in gelatin, said dispersion having been prepared by dissolving water-soluble salts of silver and of a halide in said gelatin and precipitating said silver halide therein which comprises precipitating said gelatin together with the silver halide, with trichloroacetic acid, leaving the soluble salts in solution, and removing the precipitated gelatin and silver halide from the solution.

2. The method of removing water-soluble nitrates and bromides from a gelatino-silver halide emulsion, said emulsion having been prepared by dissolving water-soluble silver salts and water-soluble bromides in the gelatin and precipitating said silver bromide therein, which comprises precipitating said gelatin together with the silver bromide with trichloroacetic acid, leaving the soluble nitrates and bromides in solution, and removing the precipitating gelatin and silver bromide from the solution.

3. The method of removing water-soluble salts from a gelatino-silver halide emulsion, said emulsion having been prepared by dissolving water-soluble salts of silver and of a halide in said gelatin and precipitating said silver halide therein, which comprises adding a solution of trichloroacetic acid in water to said emulsion containing soluble salts, precipitating the gelatin and silver halide from the solution with said trichloroacetic acid and removing the precipitated gelatin and silver halide from the solution, thereby leaving the soluble salts in the solution.

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