Patented June 12, 1945 2,378,203

UNITED STATES PATENT OFFICE

2,378,203

ETHYL TRICHLOROACETATE ANTIFOGGANT

George E. Fallesen, Rochester, N. Y., assignor to Eastman Kodak Company, Rochester, N. Y., a corporation of New Jersey

No Drawing. Application January 13, 1944, Serial No. 518,121

3 Claims. (Cl. 95—88)

This invention relates to fog inhibitors for photographic emulsions and to photographic emulsions containing them.

It is known that photographic emulsions, particularly ultra-sensitive emulsions, exhibit a tendency to form a deposit of silver in the emulsion. This deposit extends more or less uniformly over the entire emulsion and is known as fog. Photographic fog is of two types, local and general. Local fog is formed by exposure of the film or plate at undesired points, as by a light leak in the camera, or by local action of chemicals. General fog or, as it is sometimes known, chemical fog, is formed in a number of ways. It may be caused by the conditions under which the film or plate is stored, such as conditions of high temperature or unusually long time of storage. The nature of the emulsion may also produce chemical fog, as well as the conditions of development of the emulsion, as by development for protracted periods of time or at temperatures above normal. Highly sensitive emulsions are more likely to fog than those of lower sensitivity. I am concerned primarily with general or chemical fog.

It is an object of the present invention to provide a fog inhibiting agent for ultra-sensitive photographic emulsions and thereby to preserve the emulsions against deterioration causing fog. A further object is to provide a novel antifoggant which does not markedly lower the sensitivity of the emulsion.

These objects are accomplished by developing an exposed silver halide emulsion in the presence of ethyl trichloroacetate, preferably by incorporating it in the emulsion.

Ethyl trichloroacetate, $Cl_3CCOOC_2H_5$, may be incorporated in an emulsion which is intended to be used directly after emulsification or one which is subjected to ripening.

The antifoggant properties of ethyl trichloroacetate were tested as follows:

Silver iodobromide was precipitated in 0.8% gelatin solution. After precipitation was complete, this entire volume of emulsion was cooled and divided into portions weighing 1643 grams each. To each portion was added enough gelatin to increase the gelatin concentration to approximately 6.6%. Each portion was then heated to the required digestion temperature, that is, about 60° C. and as soon as this temperature was reached a certain amount of the compound to be tested, e. g., 25 grams of ethyl trichloroacetate, dissolved in 300 cc. of methyl alcohol, was added. Digestion was then continued for 30 minutes at the specified temperature and the emulsion was then chilled and stored in preparation for shredding and washing. One of these portions received no additions other than the required gelatin and it served as the control for all the portions originating from the same precipitation of silver halide.

After washing, each emulsion was heated to the optimum point for maximum speed and gamma with acceptable fog. Unsensitized and panchromatized samples of each emulsion so finished were coated by machine on glass plates. After the plates were dry, a series of sensitometric tests were made on them. The data were then compared with those of the check emulsion containing no addendum. These tests include initial tests for speed, gamma and fog, made on the day after coating, similar tests on a second set of plates incubated at 120° F., dry heat, for 6 days and a third set of tests made on a third set of plates after having been kept under tropical conditions for 14 days. The "tropical conditions" are simulated by varying the temperature and humidity through a cycle to reproduce day and night conditions in tropical climates. Plates for each of these tests were developed for 3 minutes at 65° C. in Kodak D–19 developer which had the following composition:

Monomethyl-p-aminophenol sulfate, grams... 2.2
Sodium sulfite (desiccated) ...do... 96
Hydroquinone ...do... 8.8
Sodium carbonate (desiccated) ...do... 48
Potassium bromide ...do... 5
Water to ...liter... 1

The results of these tests are tabulated below, in each case the ethyl trichloroacetate being added to an unsensitized emulsion:

Amount	Initial fog tests		Tropical condition 14 day incubation fog tests		6 day 120° F. incubation fog tests	
	Emulsion with compound	Emulsion without compound	Emulsion with compound	Emulsion without compound	Emulsion with compound	Emulsion without compound
1.5 grams per liter	.06	.05	.07	.17	.12	.23
6.3 grams per liter	.04	.05	.05	.17	.10	.23
12.5 grams per liter	.05	.05	.06	.17	.10	.23

The amount of fog was determined by reading the density at the toe of the curve after exposure and development of the emulsion, in the usual manner.

The amount of the fog inhibiting agent used in the emulsion will depend upon the composition of the agent and upon the type of emulsion. In general, the amount of fog inhibiting agent used will vary from 1 gram to 15 grams per 1000 cc. of wet emulsion. It will be understood that more or less than this amount may be used. Generally, however, if a greater amount is used in the emulsion, either no increased fog inhibiting effect is obtained or there is a decrease in the sensitivity of the emulsion. The antifoggant may be incorporated by mixing it with any suitable solvent which is inert with respect to the emulsion such as water, methyl alcohol, ethyl alcohol, acetone and others.

The fog inhibiting agent which I have described may be used in various kinds of photographic emulsions. In addition to being useful in ordinary non-sensitized emulsions, it may be used in orthochromatic, panchromatic and X-ray emulsions. Various silver salts may be used as the sensitive salt such as silver chlorobromide. The dispersing agent may be gelatin or other suitable colloid such as collodion, albumen, cellulose organic derivatives or synthetic resins.

In addition to its use in the sensitive emulsion itself, my fog inhibiting agent may also be incorporated in an overcoating or intermediate layer in the photographic element containing the sensitive emulsion layer.

It is to be understood that I contemplate as included within my invention all modifications and equivalents falling within the scope of the appended claims.

I claim:

1. A gelatino-silver halide emulsion containing a fog inhibiting amount of ethyl trichloroacetate.

2. A gelatino-silver halide emulsion containing from 1 to 15 grams per liter of wet emulsion of ethyl trichloroacetate.

3. The method of reducing the amount of fog produced upon development of a silver halide emulsion which comprises developing said emulsion in the presence of a fog-inhibiting amount of ethyl trichloroacetate.

GEORGE E. FALLESEN.