Photographic emulsions by Michael Ridgway

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The Book Barn, Westbourne, Hants. PO10 8RS. UK
Tel: +44 (0)1243-377977 Fax: +44 (0)1243-379136
e-mail : info@ResearchDisclosure.com

PHOTOGRAPHIC EMULSIONS by Michael Ridgway

The present disclosure relates to light-sensitive silver halide photographic emulsions.

The present disclosure provides a photographic emulsion in which the migration of substances can be minimised.

The present disclosure provides a method of preparing a light-sensitive silver halide photographic emulsion, which comprises the steps of adding in liquid-form a mixture of a silver complex and a photographically useful substance to an aqueous solution of a hydrophilic colloid and then adding an aqueous solution of a water-soluble halide to the mixture so as to precipitate silver halide grains having occluded therein the photographically useful substance.

The silver complex used in

the present invention should either be normally liquid or should have a low melting point. A preferred silver complex is silver benzylamine but silver cyclohexylamine or di-n-butylamine silver complexes are also useful.

The photographically useful substance may be any substance normally incorporated in photographic emulsions such as sensitizing dyes or colour forming materials.

The photographically useful substance may be dissolved directly in the silver complex. Alternatively, it may be dissolved in a water-insoluble solvent which is soluble in the liquid complex to assist formation of a homogeneous mixture of the complex and the photographically useful substance.

The hydrophilic colloid may be any of those commonly used in photographic emulsions but gelatin is preferred. The hydrophilic colloid solution is of a concentration commonly employed in the making of photographic emulsions. An approximately 10% by weight gelatin solution is particularly useful.

Afterwards, the silver halide emulsion containing the occluded substance may be washed by noodling or precipitation as is well known in the art.

The colour forming materials can be colour forming couplers or dyes which may be bleachable or stable. The dyes may also be transferable to a receiving sheet. When a colour forming coupler is occluded it becomes located very close to the development centre and the risk of migration of oxidised developer is thereby reduced.

To assist in the dispersion of the silver complex in the aqueous hydrophilic colloid a conventional emulsifying agent may be present.

The water soluble halide is preferably an alkali metal halide such as potassium bromide.

There is also provided by the present disclosure, a light-sensitive silver halide photographic emulsion comprising a hydrophilic colloid binder having dispersed therein silver halide grains, wherein the silver halide grains have occluded therein a photographically useful substance.

The disclosure also provides the photographic emulsions whenever prepared by the method of the present invention.

The photographic emulsions of the disclosure can also contain conventional addenda such as plasticizers, antifoggants, hardeners, speed-increasing agents and wetting agents.

The emulsions of the disclosure are generally coated onto supports to form photographic materials comprising a support bearing a layer of photographic emulsion according to the disclosure.

The choice of support is not critical and it can be any of those commonly employed in photography.

The disclosure will now be illustrated by the following examples.

Example 1 A complex of silver was produced by dissolving 40g. $AgNO_3$ in 100cc. benzylamine at 60°C. The complex precipitated out on cooling. The melting point of the complex is approximately 70°C when dry, but somewhat lower as used. Partial analysis of the crude precipitate showed the most likely composition to be

$$AgNO_3\ (C_6H_5CH_2NH_2)_2$$

20 gm. of the silver benzylamine complex prepared above together with 5gm. of phenolphthelein in 5ml. benzylamine was added to 25 ml. of 10% by weight aqueous gelatine solution. The mixture was homogenized for 5 minutes. 125 ml. of 5% KBr were then run in over 7½ minutes. The precipitate was washed by acid flocculation. The gelatin used was an inert bone (alkali processed) gelatin.

It was shown that some phenolphthalein was substantially occluded in the grains or in small clusters of grains by adding buffer at pH 9 to 10. Only negligible colour developed unless a silver halide solvent such as cyanide was present or the emulsion was coated onto a support and the layer was exposed and developed in

D163 Developer.

Example 2 Four grams of the crude silver benzylamine complex of Example 1 were melted and added to 1 gram of 1 - (2,4,6 - Trichlorophenyl) -3- 3' - -(2",4" - di-tert-amyl-phenoxy) acetamido benzamido -5-pyrazolone (see UK Patent 680,488) dissolved in 1 ml. benzylamine or 1 ml. ethylacetate. This mixture was added at 60°C to 10 ml. 2% by weight aqueous gelatin same as in Example 1 with 5 ml. of a solution of a spreading agent while homogenizing. After homogenizing the mixture was added to 25 ml. of a 25% by weight aqueous solution of KBr under No 3 safelight. Adding approx 7 ml. 2N H_2SO_4 was sufficient to flocculate this precipitate which was then washed in the normal way. The washing removes sufficient free amine to allow the gelatin to set when the coagulum is made up in the normal way.

Example 3 As Example 2 but cyclohexylamine was used instead of benzylamine.

Example 4 As Example 2 but di-N-butylamine was used instead of benzylamine.

For Examples 2 to 4 it can be shown that the coupler is substantially occluded within the silver halide grains by coating the emulsion onto a support, exposing the layer, partially developing it in a colour developer and 1) Examining the partially colour developed emulsion under the microscope, using refected light. Magenta coloured particles were clearly seen with silver filaments developing out of them. (Grain size was very large, 20µ for the largest grains). Developing with colour developer gives the magenta colouration while oxidised developer alone gives a very much less intense colouration. 2) The layer was then fixed and approximately as many particles remained after fixation as before.

A dry image was transferred to a receiving layer in the following way: A coated sample from Example 1 was reduction sensitized by bathing for 2 minutes in D163, then exposed to an image and developed in HC110 (1+1) for 2 minutes. Filter paper wet with potassium cyanide was placed in contact with the layer and a positive image of phenolphthalein was obtained on the filter paper.

Disclosed by: MICHAEL RIDGWAY, KODAK LIMITED, Kodak House, Kingsway, London WC2

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