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PROCESS FOR FORMING SILVER IMAGES

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This invention relates to photography. More particularly, it relates to a process for forming directly, by silver transfer during development, a silver image in a water-permeable colloid layer containing a latent image of exposed silver proteinate and, more particularly, silver gelatinate.

An object of this invention is to provide a new process for forming silver images. Another object is to provide such a process which is simple and dependable. Yet another object is to provide such a process which utilizes simple and relatively inexpensive photographic materials. A further object is to provide a new transfer process of forming a silver image in a photographic image-receptive layer containing silver nitrate and a water-permeable protein. Still other objects will be apparent from the following description of the invention.

It has been discovered that good silver images can be prepared by exposing to actinic light selected portions of a layer of a water-permeable macromolecular organic colloid containing silver proteinate and preferably silver gelatinate on a suitable support, and placing the exposed layer in contact with a layer of a water-permeable macromolecular organic colloid containing silver halide on a suitable support, at least said latter layer, before contact, being subjected to the action of an aqueous solution having reducing properties and containing a silver halide solvent and a silver halide developing agent until a silver image is developed in situ with the exposed silver proteinate, e.g., silver gelatinate (obtained by treating a layer of the protein with aqueous silver nitrate) and separating said layers. In general, good results are obtained when such developing solutions have a pH of at least 10.0. Washing the silver proteinate layer to remove residual salts may be carried out if desired. Suitable silver halide solvents include sodium, potassium and ammonium thiosulfates and thiocyanates or mixtures of these compounds.

In carrying out the process, the exposed layer of silver proteinate (e.g., gelatinate) and silver halide layer are preferably impregnated with or immersed in an aqueous photographic developer solution simultaneously and then pressed into intimate contact. The latter layer may, however, if desired, be treated, either prior to or after the silver proteinate layer, with the aqueous developer solution before the two layers are pressed into intimate surface contact. The period the layers are maintained in contact can vary, depending upon, for example, the silver-gelatin ratio in the silver proteinate layer but, in general, will be from 5 to 60 seconds.

When the layers are separated after development, a completely grainless, visible silver image will be found in the original silver proteinate layer. This image-bearing layer may be washed to remove the developer constituents and dried. No other treatment with photographic processing solutions is required.

The contact development can be carried out by the use of conventional apparatus used in inverse-transfer processes. There are various machines available for this purpose and they admit of impregnation of the two layers with the developing solution just prior to or simultaneously with the pressing of the surfaces of the layers into contact. The layer containing the exposed silver proteinate should not be subjected to the developer solution for any appreciable length of time prior to contact with the silver halide layer.

In making the exposure, any image-bearing transparency or cut-out stencil can be used. The source of actinic light must be sufficiently intense in radiation of wavelengths below 4500 A. to provide suitable exposure. Ultraviolet lamps and photoflood lamps are useful and practical sources of the actinic radiations. The exposure periods may vary over a wide range but, in general, an exposure of about 6 to about 60 seconds has been found to be satisfactory.

As an exemplary procedure, the image-receptive elements containing a light-sensitive silver gelatinate layer used in accordance with the invention can be made by coating onto a suitable support, e.g., paper, film base, glass, etc., an aqueous solution of gelatin containing 10 to 50 micromoles of silver nitrate per gram of gelatin. The pH of the coating may vary over a fairly wide range. However, a pH of 4–7 provides the most acceptable photographic characteristics of gamma, density, fog, etc. A low or high pH tends to give a faster toe speed as measured on the characteristic H. and D. curve but a lower overall density.

In place of gelatin other protein colloids, e.g., albumin, zein and casein, may be used with similar amounts of silver nitrate whereby silver proteinates are formed. These silver proteinates are silver nitrate/protein complexes. The gelatin used may be photographically active or photographically inert gelatin.

The layers containing the silver proteinate need not be made from layers composed wholly of water-permeable proteins having protective colloid properties. These protein colloids can have admixed therewith any compatible non-protein macromolecular organic colloids having hydrophilic or protective colloid properties, e.g., sodium o-sulfobenzaldehyde polyvinyl acetal and those in assignee's Shacklett applications Ser. No. 415,161 and Ser. No. 415,163, filed March 9, 1954 (U.S. Patent 2,846,417, August 5, 1958, and 2,834,758, May 13, 1958, respectively). These colloids can be present in various amounts, for example, up to 50% or more by weight of the protein colloid.

Various types of supports may be used for the image-receptive layers containing the silver proteinate. Suitable supports include films and plates composed of glass, cellulose derivatives, e.g., cellulose acetate, propionate, butyrate, acetate-butyrate, and nitrate; superpolymers, e.g., nylon, polyvinyl chloride, poly(vinyl chloride co vinyl acetate), polystyrene, polyethylene terephthalates, e.g., polyethylene terephthalate; thin aluminum sheets; paper and cardboard, etc. Of course, various sublayers may be present to anchor the layers to the base, as is common in photographic film and plate manufacture.

Any photographic reducing solution having a silver compound reducing agent and a silver halide solvent can be used in carrying out the process. The reducing agent can be present in the amounts used in conventional photographic developers for developing motion picture negatives and positives, X-ray film and other exposed silver halide films. In fact, the reducing agents may be present in amounts from that sufficient to silver transfer develop the exposed silver proteinate up to that sufficient to make saturated solutions. Suitable reducing agents are given in Mees, "The Theory of the Photographic Process," published by The Macmillan Company, New

York (1946), page 352. As will be apparent from the above, the pH of the solutions can be adjusted, if desirable, to at least 10.0, in the usual manners, e.g., by the addition of aqueous NaOH. The silver halide solvent may be present in amounts from about 3 to about 15 grams per liter.

The elements containing silver halide in a water-permeable colloid layer may use as the binding agent any of the colloids set forth above as well as any of the supports set forth above. These supports may, of course, have various resin or polymer sublayers and gelatin sublayers. The silver halide may be simple or mixed halides or may be coated from mixed emulsions. Thus, the silver halide may be silver chloride, silver bromide, silver chlorobromide or silver chloride/bromide/iodide. Such silver halide layers may or may not be light-sensitive. Other silver salt compounds which are soluble in the usual silver halide solvents may be used as donors.

The invention is further illustrated in but is not intended to be limited by the following examples wherein all parts and percentages are by weight, unless otherwise indicated.

Example 1

A 5% gelatin solution comprising 250 grams of deionized photographically inert gelatin in distilled water was made and to this there were added 75 ml. of an approximately 5% solution of saponin, 10 ml. of an approximately 16% alcoholic solution of thymol and 30 ml. of an approximately 10% solution of chrome alum. After the above additions, 48 micromoles of silver nitrate from 10% aqueous solution per gram of dry gelatin were added at 95° F. After these additions, the pH of the resulting solution was approximately 4.1. This solution was coated on the gelatin sublayer of the vinylidene chloride copolymer-coated polyethylene terephthalate photographic film support described in Alles et al U.S. 2,627,088, patented February 3, 1953, to a coating weight of 0.3 mg./dm.^{2} of silver or 60 mg./dm.^{2} of gelatin. The resulting gelatin silver nitrate coating was optically clear and colorless. All of the above operations were carried out under a safe-light equivalent to Eastman Kodak No. 613 amber. The freshly coated film was exposed for 30 seconds through an Eastman Kodak No. 3 $\sqrt{2}$ step tablet to a General Electric Photoflood lamp (PHRFL 2) placed at a distance of one foot from the surface of the film. The exposed film together with photographic film bearing a gelatin-silver chlorobromide emulsion layer as a silver donor were immersed in a developer of the following composition:

	Gm.
Hydroquinone	24
Sodium thiosulfate (anhydrous)	3
Sodium sulfite (anhydrous)	60
Sodium hydroxide	36
Water to make 1 liter.	

The developing operation was carried out in an "Autostat" developing machine manufactured by the American Photocopy Equipment Company. The exposed silver nitrate-coated film and the unexposed silver halide-coated film were introduced into the machine with their water-permeable colloid layers in face-to-face relationship. About 10 seconds were required for them to pass through the machine. The two films emerged from the machine through rollers which pressed them into intimate surface contact. After the elements remained in surface contact for 30 seconds, the two elements were separated, washed for 2 minutes in water and allowed to dry in air at room temperature. A grainless warm-tone image of the step tablet was formed in the exposed silver nitrate element by such treatment. This image had a gamma of 1.05 and a density fog 0.2 above fog at 16.2 steps, fog 0.07, and a maximum density of 1.07. The element was also exposed through Wratten filters Nos. 18A, 2, 2A, and 9. The greatest density 0.79 was obtained through Wratten No. 18A which has a maximum transmission at 3550 A. The sensitivity to radiation through the No. 9 filter was very low and the density was 0.01. This filter has a cut-off at 4650 A. No. 2 filter gave a density of 0.62 and 2A a density of 0.16 above fog.

Example 2

A 5% gelatin solution was made up as in Example 1 except that 40 micromoles of silver nitrate per gram of gelatin were added after the pH of the solution had been adjusted to 1.8 with 1.0 N nitric acid. The coating weight was 0.23 mg./dm.^{2} of silver and 53 mg./dm.^{2} of gelatin.

The coated element was exposed and developed in the manner described in Example 1. Sensitometric data were as follows: Gamma 0.90, density 0.2 above fog at 17.6 steps, fog 0.06, maximum density 0.57. Exposures through Wratten filters 18A, 2, 2A and 9 gave densities above fog of 0.31, 0.14, 0.03 and 0.00 respectively.

Example 3

An element similar to Example 2 was made, exposed and processed as described there except that the 24 grams of hydroquinone in the developer were replaced by 16 grams of metol (N-methyl-p-amino phenol hydrosulfate). The exposure results were as follows: Gamma 0.45, a density 0.2 above fog at step 15, fog 0.07 and maximum density 0.78. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.56, 0.32, 0.14, 0.00 respectively.

Example 4

The element of Example 2 was made with the pH adjusted to 2.6. Under the conditions of processing described therein there was obtained: Gamma 0.92, a density 0.2 above fog at 16.4 steps, fog 0.23, and maximum density 1.03. Exposure through Wratten filters Nos. 18A, 2, 2A and 9 gave densities of 0.62, 0.36, 0.10 and 0.00 respectively.

Example 5

An element of Example 4 having a coating pH of 2.6 was processed with the metol developer of Example 3. The results were as follows: Gamma 0.75, a density of 0.2 above fog at 15 steps, fog 0.34, and maximum density 1.26. Exposure through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.73, 0.56, 0.26 and 0.00 respectively.

Example 6

An element was coated, exposed and processed as in Example 2 except that the coating pH was adjusted to 3.7 and the developer of Example 1 was used with the following results: Gamma 0.75, a density of 0.2 above fog at 13.2 steps, fog 0.19 and a maximum density 1.17. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.86, 0.83, 0.56 and 0.12 respectively.

Example 7

An element was coated, exposed and processed as in Example 3 except that the coating pH was adjusted to 3.7. The results were as follows: Gamma 0.80, a density of 0.2 above fog at 13 steps, fog 0.27 and maximum density 1.51. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.97, 0.86, 0.58 and 0.02 respectively.

Example 8

An element was coated, exposed and processed as in Example 2 except that the coating pH was adjusted to 5 with 1 N sodium hydroxide. The sensitometric results were as follows: Gamma 0.77, a density 0.2 above fog at 12 steps, fog 0.13 and maximum density 1.46. Exposures through Wratten filters Nos. 18A, 2, 2A and 9

gave densities above fog of 1.02, 0.92, 0.54 and 0.04 respectively.

Example 9

An element was coated, exposed and processed as in Example 3 except that the pH was adjusted to 5.0 with 1 N sodium hydroxide. The results obtained were as follows: Gamma 0.95, density of 0.2 above fog at 12.6 steps, fog 0.15 and maximum density 1.65. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 1.16, 1.02, 0.62 and 0.04 respectively.

Example 10

An element was made, exposed and processed as in Example 2 except that the pH of the coating solution was adjusted to 6 with 1 N sodium hydroxide. The results obtained were as follows: Gamma 0.80, a density 0.2 above fog at 13 steps, fog 0.10 and maximum density 1.30. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.90, 0.88, 0.53 and 0.06 respectively.

Example 11

An element having the pH adjustment of Example 10 was processed in the metol developer of Example 3. The results obtained were as follows: Gamma 0.86, a density 0.2 above fog at 12.2 steps, fog 0.15 and maximum density 1.52. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities of above fog 1.06, 1.05, 0.64 and 0.04 respectively.

Example 12

An element was made, exposed and processed as in Example 3 except that the pH was adjusted to 6.5 with 1 N sodium hydroxide. The results obtained were as follows: Gamma 0.95, a density 0.2 above fog at 12.4 steps, fog 0.13 and maximum density 1.55. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities of 1.10, 1.07, 0.67 and 0.07 respectively.

Example 13

An element was made, exposed and processed as in Example 2 except that the coating pH was adjusted to 8.7 with 1 N sodium hydroxide. The results obtained were as follows: Gamma 0.44, a density of 0.2 above fog at 13.6 steps, fog 0.22 and maximum density 0.85. Exposures through Wratten filters Nos. 18A, 2, 2A, and 9 gave densities above fog of 0.48, 0.52, 0.39 and 0.05 respectively.

Example 14

The element of Example 13 was processed in the metol developer of Example 3 and the results were as follows: Gamma 0.15, a density 0.2 above fog at 14.2 steps, fog 0.12 and maximum density 0.87. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.59, 0.58, 0.37 and 0.05 respectively.

Example 15

An element was made, exposed and processed as in Example 3 except that the pH of the coating solution was adjusted to 7.0 with 1 N sodium hydroxide. The sensitometric results obtained were as follows: Gamma 0.70, a density 0.2 above fog at 11.2 steps, fog 0.14 and maximum density 1.30. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.94, 0.91, 0.59 and 0.09 respectively.

Example 16

An element was made, exposed and processed as in Example 1 except that the silver halide gelatin mixture was digested at 120° F. for 10 minutes before coating. Sensitometric results were as follows: Gamma 1.1, a density of 0.2 above fog at 16.2 steps, fog 0.07 and maximum density 1.10. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.70, 0.63, 0.23 and 0.01 respectively.

Example 17

An element was prepared, exposed and processed as in Example 1 except that the silver nitrate-gelatin mixture was digested at 120° F. for 60 minutes before coating. Sensitometric results were as follows: Gamma 1.2, a density of 0.2 above fog at 15.6 steps, fog 0.07 and maximum density 1.05. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities of 0.77, 0.80, 0.38 and 0.03 respectively.

Example 18

An element was prepared, exposed, and processed as in Example 1 except that the silver nitrate gelatin mixture was digested at 140° F. for 10 minutes before coating. Sensitometric results were as follows: Gamma 1.0, a density 0.2 above fog at 15.6 steps, fog 0.08 and maximum density 1.02. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.77, 0.73, 0.40 and 0.10 respectively.

Example 19

An element was prepared, exposed and processed as in Example 1 except that the silver nitrate-gelatin mixture was digested at 140° F. for 60 minutes before coating. The coating pH was 4.23. Sensitometric results were as follows: Gamma 1.0, a density 0.2 above fog at 15 steps, fog 0.10 and maximum density 0.99. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.68, 0.73, 0.51 and 0.04 respectively.

It will be seen that the digestion operations of Examples 16–19 increased the sensitivity of the respective layers to longer wavelengths as indicated by the increased densities through the Wratten 2 and 2A filters.

Example 20

An element was prepared, exposed and processed as in Example 1 except that the silver nitrate-gelatin solution was digested for 10 minutes at 140° F. before coating and the developer solution contained p-phenylenediamine instead of hydroquinone as the developing agent. The sensitometric tests were run on the element three days after coating which differs from previous examples in that those tests were made on unaged samples. Sensitometric results were as follows: Gamma 1.1, a density of 0.2 above fog at 12.8 steps, fog 0.25 and maximum density 1.16. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.70, 0.83, 0.62 and 0.07 respectively.

Example 21

An element was prepared, exposed and processed as in Example 20 except that the digestion time was extended to 60 minutes instead of 10 minutes. Sensitometric results after three days aging were as follows: Gamma 0.78, a density 0.2 above fog at 12 steps, fog 0.28 and maximum density 1.12. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 0.71, 0.84, 0.74, 0.10 respectively.

Example 22

An element was made according to Example 1 except that the silver nitrate-gelatin composition was digested at 150° F. for 20 minutes before coating. The coated element was exposed and processed in the manner described in Example 1 substituting a developer having the following composition:

	Gm.
Metol	12.0
Sodium sulfite (anhydrous)	30.0
Sodium thiosulfate (anhydrous)	7.5
Sodium hydroxide	18.0

Water to make 1.0 liter.

for the one used in that example.

Sensitometric exposure tests gave the following results: Gamma 1.03, a density at 0.2 above fog at 11 steps, fog 0.48 and maximum density 2.10. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 1.29, 1.37, 1.27 and 0.39 respectively.

Example 23

An element was made, exposed and processed as described in Example 22 except that a silver iodobromide emulsion coating was used as the silver halide donor layer and the time the two elements were in intimate surface contact was extended to 60 seconds instead of 30 seconds. Sensitometric exposure tests gave the following results: Gamma (not given), a density of 0.2 above fog at 11 steps, fog 0.16 and maximum density 1.06. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities of 0.50, 0.73, 0.62 and 0.25 respectively.

Example 24

An element was made, exposed and processed as described in Example 13 except that a gelatino-silver chloride emulsion was used as the silver halide donor layer. Sensitometric exposure tests gave the following results: Gamma 0.75, a density 0.2 above fog at 11.6 steps, fog 0.22 and maximum density 1.57. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 1.10, 1.05, .78 and 0.00 respectively.

Example 25

An element was made, exposed and processed as described in Example 10 except that the following developer was used:

	Gm.
Hydroquinone	16
Sodium sulfite (anhydrous)	45
Sodium thiosulfate (anhydrous)	5
Sodium hydroxide	24
Water to make 1.0 liter.	

The exposed element and the gelatino-silver chlorobromide donor layer were left in intimate surface contact for 15 seconds after emerging from the developer. The element was left unwashed after processing and the image took on a frosty appearance on drying.

Sensitometric exposure tests gave the following results: Gamma 0.90, a density of 0.2 above fog at 11.8 steps, fog 0.21 and maximum density 1.54. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities of 0.91, 0.80, 0.46 and 0.02 respectively.

Example 26

An element was made, exposed and processed as described in Example 8 except that the developer had the following composition:

	Gm.
Metol	16
Sodium sulfite (anhydrous)	45
Sodium thiosulfate (anhydrous)	15
Sodium hydroxide	24
Water to make 1 liter.	

Tests indicated a fog level of 0.25 and a maximum density of 2.26. Exposures through Wratten filters Nos. 18A, 2, 2A and 9 gave densities above fog of 1.23, 1.01, 0.53 and 0.08 respectively. During processing the exposed element and donor element were left in contact for 15 seconds after emerging from the developing solution.

The transparencies, e.g., negatives used for printing on the elements containing a silver proteinate, e.g., gelatinate, image must transmit sufficient actinic radiation to form a latent image in the silver proteinate layer. Negatives having a glass support, for example, require more exposure than a cellulose acetate support which is not as highly absorptive of ultraviolet radiation.

The exposed silver gelatinate forms a latent image. The nuclei composing this image apparently act as sites for reduction of a silver complex diffusing into this layer during the contact development. The final silver image is formed in the original layer containing the latent image.

The process of this invention can be applied in various types of photographic reproduction. Thus, it is useful in industrial X-ray, photocopy work, recording work and other types of industrial photographic applications where ultraviolet light can be used.

An advantage of the invention is that it provides a simple and dependable process for producing grainless images which are sharp and are of high resolution. The process of the invention is economical since only small amounts of silver nitrate are required in the formation of the silver gelatinate, etc. layers as compared to the conventional silver halide emulsion layers known in the art. Further, only unsensitized and unfinished silver halide emulsion layers having low quantities of silver halide need be used as the image donor layer.

Another advantage is that the process is rapid and no extensive processing steps are required. The image-forming step is rapid and after the washing step, no further chemical processing is required. For work not requiring the highest quality the washing step may be omitted. Omitting the washing step results in a rapid semi-dry process.

Another important advantage of the invention resides in the fact that the exposure and processing steps can be carried out under normal room conditions, i.e., illuminated by tungsten filament and other lamps.

A further advantage of the invention is that it eliminates the expensive fixing apparatus and time required for fixing a developed silver halide film.

Still further advantages will be apparent from the foregoing description.

What is claimed is:

1. A process for forming a photographic image which comprises bringing into surface contact (1) a water-permeable, organic colloid layer containing as the sole type of silver compound a silver nitrate/protein complex obtained by treating a water-permeable protein colloid with aqueous silver nitrate in the amount of 10 to 50 micromoles per mole of protein at a pH of not more than 7, said layer having been exposed imagewise to actinic light containing wavelengths below 4500 A., with (2) a separately supported unexposed layer of water-permeable macromolecular organic colloid containing silver halide while subjecting said contacting layers to the action of an aqueous photographic developing solution containing a silver halide developing agent and a silver halide solvent, until a silver image is formed in the first layer, and separating said layers.

2. A process as set forth in claim 1 wherein the silver halide layer is impregnated with the developer solution prior to contact.

3. A process as set forth in claim 1 wherein both layers are impregnated with the developer solution immediately prior to contact.

4. A process as set forth in claim 1 wherein said complex is a silver nitrate/gelatin complex.

5. A process as set forth in claim 1 wherein said colloid layer is composed of gelatin and said complex is a silver nitrate/gelatin complex.

6. A process as set forth in claim 1 wherein said colloid in each layer is gelatin.

7. A process as set forth in claim 1 wherein said silver halide is silver chlorobromide.

8. A process as set forth in claim 1 wherein each said layer is on a separate flexible support.

9. A process as set forth in claim 8 wherein said support is a hydrophobic film.

10. A process for forming a photographic image which comprises exposing to actinic light of wavelength below 4500 A. selected areas of a supported layer of a water-

permeable protein colloid containing as the sole type of silver compound a silver nitrate/protein complex obtained by treating a water-permeable protein colloid with aqueous silver nitrate in the amount of 10 to 50 micromoles of silver nitrate per mole of protein at a pH not more than 7, to form a latent image in said layer, bringing said exposed layer into surface contact with a separately supported unexposed layer of a water-permeable, macromolecular organic colloid containing silver halide while subjecting said contacting layers to the action of an aqueous photographic developing solution containing a silver halide developing agent and a silver halide solvent, until an image is formed in the first layer, and separating said layers.

11. A process as set forth in claim 10 wherein said exposing step is through an image-bearing negative.

12. A process as set forth in claim 10 wherein said colloid is gelatin.

13. A process as set forth in claim 10 wherein said halide is silver-chlorobromide.

14. A process as set forth in claim 10 wherein the developer has a pH of at least 10.0.

15. A process as set forth in claim 10 wherein each said layer has a separate adherent flexible support.

16. A process as set forth in claim 15 wherein said support is a hydrophobic film.

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