## PATENT SPECIFICATION



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No. 18936 / 34.

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## COMPLETE SPECIFICATION

## Improvements in Colour Photography

We, Kodak Limited, a British Company, of Kodak House, Kingsway, London, W.C. 2, (Assignees of Leopold Damrosch Mannes and Leopold 5 Godowsky, Junior, both Citizens of the United States of America, both of Kodak Park, Rochester, New York, United States of America,), do hereby declare the nature of this invention and in what 10 manner the same is to be performed, to be particularly described and ascertained in and by the following statement:-

This invention relates to photographic processes and particularly those adapted to colour motion pictures.

More specifically the invention relates to new or improved methods of colour processing photographic elements having a plurality of layers containing different 20 colour sensation records on a single sup-

Specification 376,838 describes and claims the method of producing a colour photograph comprising forming in super-25 imposed layers of emulsion sensitized respectively to record different color values, superimposed latent images of different color sensations, simultaneously developing and then fixing said images, 30 then successively forming color images in the different layers by bleaching and redeveloping with developers containing respectively different color formers. It is stated that the number of layers of 35 differently sensitized layers to be treated is not limited to two or three, though three layers is generally the maximum number desired. A way of carrying out that invention is to separate the treatment of the developed, fixed and washed film or plate for subsequent coloring by methods

tions. The coatings may be simultaneously bleached in potassium ferricyanide 45 and the upper one (or two in the case of triple coating) alone redeveloped by controlled diffusion of a concentrated developer. The undeveloped lower layer, still containing an image-record in silver ferro-

of controlled diffusion of chemical solu-

50 cyanide, may be developed by immersion in a color-forming developer as described above which will not affect the redeveloped silver of the upper layer or layers. The

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plate, or film, may be fixed in sodium thiosulphate, washed, and then dried, and the remaining layer or layers bleached to silver ferrocyanide for redevelopment in another color forming developer, and so on to the third coating in the case of

three color photography.
Instead of this method of separation by controlled diffusion of concentrated redeveloper the negative may, according to this prior patent, be treated as follows. The plate or film is developed, fixed and dried. The top layer is converted to silver ferrocyanide by controlled diffusion of potassium ferricyanide in solution. Treatment with a colour developer will then affect this bleached layer only. The silver may be removed from this top image record by controlled diffusion of any suitable silver solvent. The lower layers are successively treated in this manner using a different colour developer for each until only pure colour remains. Obviously this method depends on controlled diffusion of the potassium ferricyanide employed.

The process of the present invention resembles the process described in the above specification in so far as it results in the formation of colour images in the different layers by bleaching and redevelopment with developers containing respectively different colour formers but differs from it in features which will be apparent from the following description and the appended claims and which are of substantial importance in the practical pro-

duction of colour photographs.

According to the present invention the method of colour processing a photo-graphic element having a plurality of superimposed layers containing different colour sensation silver salt records on the same side of a single support includes colour developing all the silver salt re-cords in the layers to one colour, fixing if necessary, and effecting bleaching of the 100 colour developed record in an upper layer by subjecting the element to the action of a bleaching agent which will remove and/or destroy the colour and will transform the silver which was developed there- 105 with into a developable silver salt and

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which also contains a high concentration of a water soluble loading agent to retard the diffusion of said bleaching agent therethrough and arresting such diffusion 5 before the bleaching agent substantially penetrates into a lower layer and materially affects the image in such lower layer. The expression "silver salt records" is intended to include latent or bleached 10 silver images.

There is preferably provided, immediately above the lower layer referred to, a layer of gelatine and diffusion of the bleaching agent is arrested before it has 15 penetrated through this clear gelatine layer. The bleached image in the upper layer may be colour developed to a

different colour. In order to enable the action of the 20 bleaching bath to be restricted the bleaching operation is subjected to time control and stop baths may be used to arrest the

diffusion of the bleaching agent.

Such methods of control of the penetra-25 tion of a bleaching bath by time control and stop baths are utilised in carrying out the method of processing described and claimed in our co-pending application No. 18932/34 (Serial No. 427,516) but the present invention is characterised by the employment of loading agents.

The control of the diffusion of a bleaching bath by the aid of water-soluble loading agents and the employment of a clear 35 gelatine layer to facilitate the control are claimed otherwise generally in co-pending application No. 18934/34 (Serial No. 427,518).

The use of loading agents in photo-40 graphic developers is known. developers for use in tropical countries have been suggested in which quantities up to about 20% of a salt such as crystalline sodium sulphate or potassium sul-45 phate or ammonium sulphate are included for the purpose of retarding the swelling of the gelatine. Chrome alum has also been added to amidol developers for the same purpose. The development is also 50 slowed down by the presence of such salts but for the purposes to which such developers were put, this was a disadvantage because at high temperatures it is important to work quickly to avoid trouble 55 due to the softening of the gelatine. Other developers have also been proposed for use in tropical countries such as a metol-hydroquinone developer containing besides the usual ingredients, 8% of 60 acetone and 1.5% of formalin. Also the use of 50% alcohol has been suggested for the same purpose. The use of sugar for this purpose has been suggested. It is also known to add to developing solutions 65 such substances as glycerine and treacle

which, by increasing the viscosity confine the development to the surface of ordinary black and white photographic prints the purpose being usually to produce "soft" or other "artistic" effects. So far as we are aware it has not hitherto been known, in colour photographic processes, to load processing baths, still less bleaching baths, with loading agents for the purpose of assisting in confining the action of the processing reagent to the upper of a plurality of layers on a single

support.

The invention further includes bleaching bath comprising a solution of a reagent adapted to bleach a colour developed silver image by removal and/or destruction of the colour and by transformation of the silver which was developed therewith into a developable silver salt, such as a solution of chromic acid and hydrobromic acid, and which also contains a large quantity of a watersoluble loading agent to retard the diffusion of the bath, for carrying out the improved method.

In carrying out the present invention suitable water-soluble loading agents are water-soluble substances such as sodium sulphate or organic liquids such as

methanol.

The expression "colour development", when used herein is intended to designate a process effected by developing a silver image with a developer containing a 100 colour former, as described in patent specification No. 376,838. Such colour formers are organic compounds acting as couplers in connection with certain developers to form coloured compounds, 105 usually insoluble in water, in the presence of the finely divided silver which is being formed by reaction. The colour com-pound formed may belong, for example, to the class of indophenols, indoanilines 110 and indamines and remains colloidally dispersed in the gelatine layer even when the silver has been removed. It is thus possible to produce by this means a substantially transparent coloured image in 115 proportion to the extent and depth of the original silver image. The expression "colour development" when used herein does not therefore include the known process in which colour formers are incor- 120 porated in the layers themselves.

If the colour sensation records are in the form of latent images or if the colour development is not carried to completion, it is necessary to employ a fixing step 125

before the bleaching operation. The developable silver salt produced by the bleaching is afterwards colour developed to a colour different from that to

which it was previously colour developed. 130

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Preferably all the silver salt records in the layers are first colour developed to a red.

The invention may be applied to the 5 processing of negative or positive photographic elements.

The invention will be illustrated by the following description of a preferred manner of processing a film which is

10 given by way of example. The film consists of a support carrying superposed layers of green-sensitized and red-sensitized emulsions respectively, the green-sensitized emulsion being nearest the support. A thin intermediate layer of unsensitized gelatine is provided between these differentially sensitized layers. Such a film may be exposed in a split-beam two-colour camera of the type 20 shown and described in our copending application No. 26084/33 (Serial No. 427,472). The exposed film is then processed to produce an image in the lower layer colour developed to a minus green 25 colour and an image in the upper layer colour developed to a minus red colour.

In practice the following material has been found to give the best results.

The film base is first coated with a layer 30 of a thickness of the order of .0002 inches of a very rapid emulsion sensitized to the green region of the spectrum between  $510-590 m\mu$ , with a maximum at about 550 mμ. A fast emulsion sensitized with 35 erythrosin has been found satisfactory. Over this emulsion is coated a very thin layer, say between .0001 and .0003 inches, of clear gelatine, clear enough to permit adequate exposure of the green-sensitized The final top coating is of the same order of thickness as the greensensitized layers and is a red-sensitized rapid emulsion which has been diluted with an equal weight of gelatine 45 before coating to give greater transparency, less density, less tendency to exhaust the developer diffusing through it, and finally, less tendency to harden the

gelatine where the image develops. For 50 this top emulsion layer a fast emulsion has been used, sensitized with a red sensitizer conferring sensitivity primarily in the region between 600 and 700  $m\mu$  with a maximum near 650 mµ. Such a sensi-55 tizer is naphthocyanol. Both layers are, of course, sensitive to blue. The red sensitive emulsion is placed above the green sensitive emulsion so that when

carrying out the subsequent processing as 60 hereinafter described, it is the red dye which is subjected to the bleaching step such as step 4.

It is important for this red-sensitized layer to use an emulsion that is relatively 65 insensitive to light of wave-lengths

between 510 and 590  $m\mu$ . For the lower green-sensitized emulsion a corresponding restriction is not necessary, as will be seen. The total thickness of the coatings of this material altogether should not substantially exceed the thickness of a single normal coating on negative motion picture film as ordinarily supplied.

It is naturally important that these two emulsions, as finally coated, have very similar essential characteristics of latitude, speed, contrast, and maximum

density.

The unsensitized clear gelatine intermediate layer affords protection against possible wandering of the sensitizing dyes from one emulsion to the other and also gives latitude in the differential treatment of the layers described hereinafter.

COLOUR PROCESSING.

STEP 1. After the film has been exposed, it is developed and fixed as in ordinary photographic procedure. A developing formula is chosen which produces the minimum of selective hardening of the gelatine where the image develops.

Diamino-phenol-sodium sulphite containing a small quantity of sodium bisulphite has been found very satisfactory.

Such a formula is:

FORMULA I. Diamino phenol -5 grm. Sodium sulphite -10 grm. Sodium bisulphite 5 grm. 100 Potassium bromide  $4~\mathrm{grm}$ . Water to 1 litre. When it is desired to reduce the number of steps in processing one may develop the latent images in the film directly in a red 105 colour-forming developer (for example Formula III), fix, and then proceed to step 4 below. However, increased exposure seems to be necessary when develop ment is to be carried out by this method 110 and it is therefore preferred to perform the operation of colour development upon actual silver salt images.

STEP 2. The film is now immersed in dilute 115 potassium ferricyanide to convert the silver images to silver ferrocyanide without local hardening of the gelatine. Silver ferrocyanide produced is very easily reducible back to metallic silver. 120 A suitable bleach is:

FORMULA II. Potassium ferricyanide -- 10 grm. Ammonia 28% 10 cc. Water to -1 litre. 125 STEP 3.

The next step is the exposure and re-development of the bleached images (actual silver salt images) in a colourforming developer yielding insoluble 130

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for this bleach:

FORMULA IV.

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	monochrome dye images, together with re-developed silver. A red or magenta dye (minus green) is required and has been satisfactorily obtained by using
- 5	diethyl para-phenylene diamine in alka-
10	the subsequent stop bath used in connection with that bleach. In the example described a chromic acid-bromide-alcohol
15	bleach bath is employed. If this image is required to print on a green-sensitized layer of a printing element the dye must in addition have as sharp an absorption
20	printing image. The dye in this printing image should absorb as efficiently as possible the light in the sensitivity range
25	printing element, and at the same time transmit as efficiently as possible all the light in the sensitivity range of the red-sensitive emulsion of the printing element. This red dye may, and
30	A suitable minus green colour developer
	is: FORMULA III.
35	Sodium carbonate 10 grm. Sodium sulphite 0.5 grm. Diethyl $p$ -phenylenediamine HCl 0.5 grm.
40	In 100 c.c. of this solution is dissolved
	That a colour, have infinitely of point and
•	must either be carried to completion or,
	if stopped before completion, must be followed by a hypo bath to remove any
	ionowed by a mypo bath to remove the state of the state o
45	undeveloped silver ferro-cyanide. After washing, the film is dried in order to
	onsure the nest result from the arrest
	treatment which follows. STEP 4.
50	The next step is the critical one in the
90	' 1 Jifferentiation between the two
	lorrong and denerities of legitivities
	penetration of a bleaching solution to the depth of one layer only. An ordinary

depth of one layer only.

55 dilute water solution would penetrate the surface layer in 1 to 2 seconds. By using

60 water solution containing chromic acid, hydrobromic acid and potassium bromide

a high concentration of a loading agent

such as alcohol, the time may be greatly extended. A 75% methanol and 25%

bleaches the surface layer in 15 to 30

seconds, depending upon temperature and the thickness of the emulsion coating.

The following is a satisfactory formula

An ordinary

Chromic acid, 10% solution - Hydro-bromic acid 41% solution - 10 cc. 3 cc. Potassium bromide -2 grm. 70 - 300 cc. Methanol Water --90 cc. WaterThe time of immersion in this bleach is carefully measured by trial, which is easily done, and after the bleaching bath the film is immediately immersed in a stop bath of this formula: Ammonia 28% -- 100 cc. WaterThe action of this bleach bath is to convert to silver bromide the silver in the top emulsion layer only and to bleach the dye formed with the silver image to a colourless compound which is very soluble in the alcohol of the bleach bath. bleaching action would continue undesirably to the lower layer if not checked. Also, some of the undissolved bleached dye would be restored to colour in the image again as the acid was gradually washed out of the film. To check the bleaching action quickly and prevent restoration of dye, the stop bath given is used with very satisfactory effect. An alternative formula for a stop bath is the following: Methanol Sodium sulphite - - -20 grm. 10 cc. Ammonia 28% Water to make 1 litre. The sodium sulphite inhibits restoration of the dye, while the methanol dissolves out the bleached dye compound. The step involving bleach and stop bath is most practicable in a processing 105 machine where time and temperature control may be maintained within reasonable limits of variation. To allow for some error, the thin gelatine layer between the two emulsions has been found to afford 110 sufficient tolerance. That layer may be about one-half the thickness of the emulsion coatings. STEP 5. The above differential treatment leaves 115 the film with silver plus dye in the lower layer, and silver bromide in the upper Therefore, subsequent immersion in a blue-green (minus red) colour-forming developer, such as dichloro- or tri- 120 chlor-alpha-naphthol with diethyl-paraphenylene-diamine, causes development only in the top layer where the image is formed of silver bromide. Of course, the film must be exposed to white light at this 125 point to render the silver bromide developable.

A suitable blue-green colour developer

is the following:

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FORMULA V.	to rondon 41
SOLUTION A	to render the appearance of the double-
M/ oton	oodica mini oniv taintiv onegrio Ac
Sodium carbonate - 10 crm	stated, each of these layers may be colour-sensitized with the same sensitizing
o boarding suiparte 5 arm	dve used for the nemetic sensitizing
p-pnenylene diamine	of these emulsions are prodominantly of
HCI 2 grm	silver bromide.
SOLUTION B.	The printing material has a layor on
2:3:4 trichlor-alpha naphthol - 1 grm	one leverse side of the film support com
10 Methanol 150 cc.	Prising a unit coating of slow emplaion 75
Solution A 100 cc. Solution B 15 cc.	not necessarily transparent sensitized to
Solution B 15 cc	oso mira-red region from about 750 to
Arter development in this bath the film	coo non and covered with a water proof
15 is then washed.	stripping varnish layer which may contain dve or lamp block to
STEP 6.	tain dye or lamp-black to serve also as 80 backing necessary to avoid halation, due
The final step in processing the negative	to reflection of red, green, and infra-red
is to remove the silver image remaining	rays from the rear emulsion-air interface
in both emulsion layers, preferably by	Denzyl cellulose has been found adapt
20 potassium ferricyanide and sodium thio- sulphate in combination	able to this purpose, as it may be easily or
sulphate, in combination or as separate baths, leaving a pure minus green dye	builded minimize emiliation surface when
image in the lower layer and a pure minus	it is desired to process the infra-rad-gangi
ted dye image in the unner lawer Term	dized coating as given below. A suitable
20 tills bleach the Well-known Harmer's re-	variable for this purpose is:
ducer is satisfactory.	FORMULA VI.  Benzyl cellulose 150
The film is then washed and dried.	Benzyl cellulose 150 grm. Benzene 1550 cc.
II a negative, processed as described	Toluene 100 cc.  Xylene 400 cc.
above, is printed in a contact printer by	Xylene 400 cc.
30 yellow light on printing material double- coated (i.e. without the layer on the re-	(Ine manufacture of a low viscosity or
verse side indicated below) and if the	beilzyl cellulose suitable for this nurnose
printed material be then processed as do.	18 described in British Patent Specifica
scribed a master positive will regult	orons Nos. 321,714, 339,902 and 356,308).
of the petition of this process will result in a	Although this infra-red-sensitive emul-
master negative.	sion may be of the type described for the 100 other two coatings, it is advisable to have
To avoid excessive re-duplication and	it in the form of a pure silver chloride
consequent loss in quality the double	emuision of whatever grain size may be
coated record element may be put through	necessary to secure sufficient latitude An
40 an ordinary reversal treatment after exposure and thereafter processed as de-	Illra-red sensitizer which is suitable for to
scribed.	viiis circuision is neocvanine (thio tri
In making master positive or master	car bucyanine).
negative prints, it will be of advantage to	The infra-red sensitizing of such a
40 use as a source of printing light not	chloride emulsion has been found to con- fer sufficient speed to infra mallilable
yellow light but additive red plus green	fer sufficient speed to infra-red light for 110 practical purposes.
11914 11014 two nearly monochrometic	Such a film, in which one of the layers
light sources which will minimize the loss	is sensitized to inira-red, and features of
of colour intensity due to excessive re- 50 duplication.	The processing from the subject of our
For printing purposes the following	co-benuing, application No 18032/3/44*
material is suitable.	(Derival IVO.  42(.019).
On one side of a film base two emulsions	The reason for having this single emul-
are coated with an intermediate very	chloride is composed of pure silver
of thin getatine layer between them. These	THE TOTAL TO USE DELINITY OF OTTERPORTS OF FIRE
emulsions are coated respectively in the	tion in ammonia which will dissolve the 120 silver chloride of this single layer with-
same order to approximately the same	out appreciably effecting the silver
thickness and with substantially the same	browning formed in a subsequent bleaching
colour-sensitizing specified for the nega-	biep of the positive processing which
30 tive material. Accordingly, there is a	be described later. This feature of the for
green-sensitive layer next to the support, then the intermediate gelatine layer, and	
then the red-sensitive layer. These	publication of our co-nending application
printing emulsions, however, are chosen	100 1000 04 (Derial No. 497 517)
35 for exceedingly fine grain, fine enough	A method of printing on this material is
	described in our copending application 130

No. 26084/33 (Serial No. 427,472) already mentioned. COLOUR PROCESSING THE PRINTED MATERIAL. The printed material has a waterproof varnish layer on one side so that the double layer side only is treated at the outset. The first steps of the processing are development of the images in a red 10 (minus green) colour developer and treatment of the film in a bleach which is restricted to the depth of one layer only. For the red (minus green) colour developer, we prefer the following solution 15 for the printed red images: FORMULA VII. 1 litre. Water Diethyl para-phenylene diamine 10 grm. HCL  $5 \mathrm{\ grm}.$ 20 Sodium sulphite 20 grm. Sodium carbonate Potassium bromide (Molar solution) To 100 cc. of this solution add brom-thio- $25~{\rm indoxyl}~0.05~{\rm grm}.$ The bleaching of the upper layer only is then accomplished by the method given in Step 4 of colour processing. At this stage where the top layer only 30 of the double coating has been bleached to silver bromide and the dye in that layer removed coincidentally, the protective varnish is removed from the infrared-layer at the back of the film. varnish layer is easily stripped when dry. The film is now immersed in a pure yellow (minus blue) colour-forming developer. A suitable formula for the yellow colour developer is: FORMULA VIII. 40 1 litre. Diethyl-p-phenylene diamine 10 grm. HCl 5 grm. Sodium sulphite 20 grm. Sodium earbonate Potassium bromide (Molar solu-To 10 cc. of this solution are added benzoyl acetone 0.1 grm. dissolved in 50 ethyl alcohol 5 cc. Other yellow couplers are possible and available. Besides benzoyl acetones, aceto-acetic esters have been used as couplers with 55 diethyl-paraphenylene diamine as developer. After the yellow development, the film is immersed in dilute ammonia, approximately 4%, to fix out the undeveloped silver chloride in the infra-red-sensitive layer without affecting the silver bromide

formed by the bleach bath in the top

bromide is therefore the only developable

65 deposit in the film and is developed after

layer of the double coating.

exposure to white light in a minus red colour-forming developer which may be of the same type as that given in Step 5 of Colour Processing. After thorough washing, the residual silver images remaining in all three emulsions are simultaneously removed as described in Step 6 of Colour Processing. This step involves a simple reduction by 75Farmer's reducer. The film is now washed and dried and is a complete-three colour subtractive picture in which the dye image in the three layers respectively, successively absorb red, green 80 and blue, and by the subtractive process transmit a properly coloured three-colour image. If differential fixation in ammonia is inconvenient or impossible (for example when the infra-red sensitized layer contains silver bromide and the image in the upper of the two layers on the other side of the support is bleached to silver bromide) it is necessary to employ methods of flotation or other mechanical means in 90 differentially processing opposite sides of the film. These are practicable and may be used to avoid the necessity of waterproofing one layer with varnish but appear less useful than the one fully described 95 above. The colour sensitizing of the layers of negative material may be changed altogether from that described. The sensitivity range may be quite different from 100 that mentioned. The two layers of negative material may be coated in the reverse The intervening thin gelatine layer between the two layers of negative material may contain a dye to act as a 105 screen for the layer underneath. The colours chosen for processing negative material may be changed, as green and red need not necessarily be used. A blue and a yellow, or a blue green and 110 orange yellow, may be used to form efficient printing images, in which case the double coating of the printing material to be used therewith will have two emulsion layers sensitized to blue and to orange res- 115 pectively, in either order. While we have described this process and designed it particularly for a colour motion picture, we do not wish to be limited to the motion picture field. 120 Similar methods could be applied to the making of still pictures on either plates or films. Having now particularly described and ascertained the nature of our said inven- 125

tion and in what manner the same is to be performed, we declare that what we

1. A method of colour processing a

photographic element having a plurality 130

claim is:

This silver

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of superimposed layers containing different colour sensation silver salt records on the same side of a single support which includes colour developing all the silver 5 salt records in the layers to one colour, fixing if necessary, and then bleaching the colour developed record in an upper layer in which such bleaching is effected by subjecting the element to the action of a 10 bleaching agent which will remove and/or destroy the colour and will transform the silver which was developed therewith into a developable silver salt and which also contains a high concentration of a water 15 soluble loading agent to retard the diffusion of said bleaching agent therethrough and such diffusion is arrested before the bleaching agent substantially penetrates into a lower layer and materi-20 ally affects the image in such lower layer. 2. A method as claimed in Claim I in which there is provided immediately above the lower layer referred to a layer of clear gelatine and diffusion of the 25 bleaching agent is arrested before it has penetrated through this clear gelatine

3. In a method as claimed in claim 1 or claim 2 colour developing the bleached 30 image in the upper layer to a different colour.

4. A method of processing a photographic element as claimed in any of the preceding claims in which diffusion is 35 arrested by the aid of a stop bath.

5. A method of processing a photographic element as claimed in any of the preceding claims in which a chromium bleach bath is employed.

0 6. A method of processing a photographic element as claimed in claim 5 in which diffusion is arrested by the aid of

a stop bath containing alkali with or without a reducing agent.

7. A method of processing a photographic element as claimed in any of claims 1 to 6 in which the loading agent is a water-soluble organic liquid.

8. A method of processing a photographic element as claimed in claim 7 in which the loading agent is methanol.

9. A method of processing a photographic element as claimed in any of the preceding claims in which the photographic element is substantially dried before the step of bleaching the image in an upper layer only.

10. Photographic elements whenever processed by the methods claimed in any of the preceding claims.

11. A bleaching bath for the restricted bleaching of a photographic element having contiguous layers containing different colour sensation records in the form of colour developed images by the method claimed in claim 1 comprising a solution of a reagent adapted to bleach a colour developed silver image by removal and/or destruction of the colour and by transformation of the silver which was developed therewith into a developable silver salt and which also contains a large quantity of a water-soluble loading agent to retard the diffusion of the bath.

12. A bleaching bath for carrying out the process of claim 7 comprising a solution of chromic acid and hydrobromic acid which also contains a high concentration of methanol.

Dated this 26th day of June, 1934. W. P. THOMPSON & CO., 12, Church Street, Liverpool, 1, Chartered Patent Agents.

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