[72]] Inventors Hiroyuki Amano; Nobuo Tsuji; Kazuo Shirasu; Yoshinori						
			itiya, all of Kanagawa, Japa				
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[73]	Assigne		ji Photo Film Co., Ltd.				
			higara-Kamigun, Kanagaw	a. Japan			
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[54]	PHOTO	GRAP	HIC LIGHT-SENSITIVE!	MATERIAL			
e.	CONTA	INING	A POLYMERIC BRIGHT	ENING			
	AGENT						
	9 Claim:	s, No D	rawings				
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			96	/84, 117/33.5			
[51]	Int. Cl		***************************************				
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				117/33.5 T			
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Primary Examiner—Ronald H. Smith Attorney—Sughrue, Rothwell, Mion, Zinn & MacPeak

ABSTRACT: A photographic light-sensitive material containing, in at least one layer thereof, a polymer having the following recurring monomer units:

Where R_σ is hydrogen, alkyl, aryl, hydroxyalkyl, alkoxyalkyl or sulfoalkyl, R_1 and R_2 are halogen,

$$-OR$$
, $-SR$, $-N$, and $-N$,

M is alkali metal or ammonium, R and R' are hydrogen, alkyl, hydroxyalkyl, sulfoalkyl, carboxyalkyl, aralkyl or cycloalkyl, and A is $-(CH_2)_4-,-(CH_2)_5-,-(CH_2)_2-O-(CH_2)_2-O-(CH_2)_2-S-(CH_2)_2-R_1$ and R₂, and R and R' may be the same or different.

PHOTOGRAPHIC LIGHT-SENSITIVE MATERIAL CONTAINING A POLYMERIC BRIGHTENING AGENT

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a photographic light-sensitive material containing a high molecular weight compound having ultraviolet ray absorbing action and fluorescence brightening action.

2. Description of the Prior Art

In general, when a color image consisting of an azomethine or an indoaniline dye is obtained by color developing and then the color image is exposed to ultraviolet rays, it is faded or discolored depending on the strength and wave length of the rays. As is well known, unfavorable stains are formed in a finished photographic layer by attacking with ultraviolet rays when coexistent materials, besides forming a color image (for example a coupler) remains in the photographic emulsion layer after the color image is formed. Such phenomenon is 20 often caused by ultraviolet rays of 300 to 400 millimicrons in wavelength. For the purpose of preventing this, ultraviolet absorbers have often been incorporated in the photographic emulsion layer, intermediate layer, protective layer, subbing layer, or the back of the support. However, many of the 25 known ultraviolet absorbers have such disadvantages that the absorber itself tends to cause photolysis by ultraviolet rays, thus resulting in unfavorable stains, or a large part thereof diffuses out in the steps of photographic processing and washing with water so that its effect is weakened.

It has been customary, heretofore, as a means for brightening a photographic printing paper, to add a fluorescent brightening agent. The agents are capable of emitting violet to blue fluorescence, during ultraviolet radiation, to a photographic light-sensitive emulsion layer including a positive 35 layer of printing paper, by the diffusion transfer and reversal method, or to an auxiliary layer such as subbing layer, protective layer or baryta layer of baryta paper. However, many commercial fluorescent brightening agents encounter the disadvantage that most of the brightening agent diffuses out in 40 the steps of photographic processings and washing, as in the case of the ultraviolet absorber, thus losing the brightening effect, since it is hard to dye on a colloid of a photographic emulsion layer or auxiliary layer, for example, gelatin, due to its low molecular weight.

Since then, various efforts have been made in order to overcome the foregoing disadvantages, such as, by introducing a substituent excellent in dyeing affinity to gelatin, and by dispersion in a gelatin-polyvinylpyrrolidone (Japanese Pat. publication No. 7127/59) or poly-N-vinyl-5-methyl-2- oxazolidinone (Japanese Patent publication No. 22065/64). The substituent has excellent dyeing affinity to fluorescent brightening agents, but in these methods, diffusing-out of a fluorescent brightening agent cannot be prevented enough, and the brightening effect decreases considerably.

SUMMARY OF THE INVENTION

It is an object of the invention to provide a photographic light-sensitive material having a marked ultraviolet ray inhibiting effect as well as a stain preventing effect.

It is another object of the invention to provide a photographic light-sensitive material having a marked fluorescent brightening effect.

DESCRIPTION OF THE INVENTION

These objects can be achieved by incorporating a polymer in at least one layer of a photographic light-sensitive emulsion 70 layer and photographic auxiliary layer, such as intermediate layer, protective layer, subbing layer, including baryta layer of baryta-coated paper, back layer, etc. The polymers are homopolymers or copolymers having a recurring unit represented by the following formula:

wherein R_0 represents a hydrogen atom, an alkyl group having from one to eight carbon atoms such as methyl, ethyl, propyl, butyl, pentyl, hexyl, octyl, etc., an aryl group having from six to 12 carbon atoms, such as phenyl, tolyl, xylyl, naphthyl, etc., a hydroxyalkyl group having from two to four carbon atoms such as hydroxyethyl, hydroxypropyl, etc., an alkoxyalkyl group having from two to six carbon atoms such as methoxyethyl, ethoxyethyl, a sulfoalkyl group having from one to four carbon atoms such as sulfomethyl, sulfoethyl, sulfopropyl, sulfobutyl or an alkali metal, or ammonium salt thereof such as sodium, potassium, ammonium, etc. R_1 and R_2 may be the same or different and each represents a halogen atom, -OR, -SR,

wherein R and R' may be the same or different and represent a hydrogen atom, an alkyl group having from one to 12 carbon atoms, such as methyl, ethyl, propyl, butyl, hexyl, octyl, decyl, dodecyl, etc., a hydroxyalkyl group having from one to four carbon atoms such as hydroxyethyl, hydroxybutyl, etc., a sulfoalkyl group having from one to four carbon atoms, an alkali metal or ammonium salt thereof, a carboxyalkyl group having from one to four carbon atoms such as carboxymethyl, carboxyethyl, carboxybutyl, etc., an alkali metal or ammonium salt thereof, an aralkyl group having from seven to 14 carbon atoms such as benzyl, etc., an aryl group having from six to 18 carbon atoms such as phenyl, tolyl, hydroxyphenol, chlorophenyl, carboxyphenyl, sulfophenyl, naphthyl, carboxynaphthyl, sulfonaphthyl, etc., a cycloalkyl group having from six to 10 atoms such as cyclohexyl, etc., or a heterocyclic group such as pyridyl, benzothiazolyl, etc., and, A represents $-(CH_2)_4$, $-(CH_2)_5$, $-(CH_2)_2$, -0, $-(CH_2)_2$ or $-(CH_2)_2$ -S- $-(CH_2)_2$. M represents an alkali metal such as sodium, potassium etc., or an ammonium group.

A monomer unit which constructs the copolymer used in the present invention is represented by the following formula:

wherein X represents -OH, -OCH₃, -OCH₂CH₃,-OCO₂CH₂OH, -OCOCH₃, -OCOCH₂CH₃, -OCONH₂.

65 or

Although the preferred copolymers have a monomer unit as defined by formula II, the copolymer used in the invention may have any other monomer units that are copolymerizable

therewith. The polymers used in the present invention are characterized by having the recurring unit represented by the formula I

By incorporating the above-mentioned high molecular weight compound of the invention in any of the layers of a photographic light-sensitive material, a marked ultraviolet ray absorbing effect and stain preventing effect can be given, and there is no stain even under a strong ultraviolet radiation. The high molecular weight compounds used in the invention have remarkable brightening effects on photographic printing papers, being held even after the steps of photographic processings and washing.

Addition of an ultraviolet ray absorbing material, in general, is carried out by a direct addition method using water or a solvent as a dissolving medium or by an indirect addition method using a high boiling point solvent as a dispersing or emulsifying medium. It is generally desirable that the ultraviolet ray absorbing material be in the uppermost layer, but, in the case of a light-sensitive material produced by the latter method, this is not as necessary, since the added material tends to segregate by shock or friction. On the contrary, the compound of the invention may be incorporated in the uppermost layer by the direct addition method.

Synthesis of the high molecular weight compound of the invention is generally carried out by dissolving or dispersing a compound of the triazylaminostilbene type represented by following formula III in polyvinyl alcohol or a copolymer thereof with a solvent such as water or dimethylformamide, adding thereto an aqueous solution of caustic soda in an amount equivalent to, or somewhat in excess of, that of the compound 30 III and reacting them at a temperature of 50°-150° C.

 $\begin{array}{c|c} C1 & & R_2 \\ \hline N & N & & N \\ \hline R_1 & N & & N \\ \hline R_0 & SO_3M & SO_3M & (III) \\ \end{array}$

in which R_0 , R_1 , R_2 and M have the same meaning as in the formula I. Of course, the synthesis is not limited to the above-mentioned method.

The cyanuric chloride derivative represented by formula III can readily be obtained by the stepwise reaction of a derivative of 4,4'-diaminostilbene-2,2'-disulfonate with a suitable derivative of cyanuric chloride in a conventional manner, as is disclosed in "Yuki Gosei Kagaku Kyokaishi (Bulletin of Organic Synthetic Chemistry Association)" Vol. 20, page 64 (1962).

The ratio of reaction of compound (III) to polyvinyl alcohol or its copolymer is not particularly limited, but, considering the respects of economy and fluorescent strength, it is not always necessary to react it with all the vinyl alcohol units. For instance, it is desirable, in order to obtain good fluorescent strength, that compound (III) be linked with polyvinyl alcohol in such a manner that dispersion can be effected to an extent that no concentration quenching occurs. As a result of our experiments it was unexpectedly found that a polymer substituted with compound (III) in a proportion of 0.1 to 50 mole percent, particularly, 0.5 to 10 mole percent, based on the vinyl alcohol unit, is sufficiently effective.

Illustrative of the polymer used in the invention are compounds having the following recurring units:

Compound 2.
$$-M.W. = 150,000$$
 $-CH_3-CH OH$
 OH
 $CH_3HN NH SO_3Na$
 SO_3Na
 SO_3Na

Compound 4.—M.W.=300,000
$$-CH_{2}-CH-$$

$$OH$$

$$(HOCH_{2}CH_{2})_{2}N-NH-NH-CH=CH-NH-NN-N(CH_{2}CH_{2}OH)_{2}$$

$$SO_{3}Na$$

$$SO_{3}Na$$

Compound 7.—M.W.=300,000

Compound 8.—M.W.=400,000

Compound 9.—M.W.=350,000

Compound 10.-M.W.=150,000

Compound 11.-M.W.=350,000

Compound 12.-M.W.=200,000

$$-(CH_2-CH)_5-(CH_2-CH)_{95}-\\OOH\\OOH\\OOH\\NAO_3S--NH-NH--NH--NH--NH--NH--SO_2Na\\SO_3Na\\SO_3Na\\SO_3Na$$

Compound 13.-M.W.=100,000

$$-(CH_{2}-CH)_{5}-(CH_{2}CH)_{53}-(CH_{2}-CH)_{12}-\\OHOCOCH_{3}OH\\N_{8}O_{3}S-(CH_{2})_{2}-NH-\\N_{1}-NH-\\CH=CH-\\N_{1}-NH-\\CH=CH-\\N_{1}-NH-\\CH_{2}SO_{3}N_{8}$$

Compound 14.-M.W.=150,000

Compound 15.-M.W. = 200,000

Compound 16.-M.W. = 150,000

Although the larger the molecular weight of the high molecular weight compound used in the invention, the better the prevention of dissolving out in photographic processing, the need for a lack of solubility can be satisfied if polyvinyl al- 70 cohol or its copolymer, as a trunk polymer, has a degree of polymerization of more than about 50 or a molecular weight of more than about 20,000.

In the present invention, the high molecular weight com-

more particularly about 100,000-400,000 is preferably used.

The photographic emulsion used in this specification means the commonly used light-sensitive silver halide emulsion, but, as a colloidal agent for such emulsions, not only gelatin, but also synthetic resins such as polyvinyl alcohol and polyvinyl acetal can favorably be used.

The following examples are given in order to illustrate the results about the light fading absorbing effect of an image, difpound having a molecular weight of about 20,000-1,000,000, 75 fusion resistance and fluorescent brightening and a photo-

layer was prepared by gradually adding 1 kg. of an aqueous

solution of gelatin to 100 g. of a 4 percent solution of each of

the high molecular ultraviolet inhibiting materials of the invention, compound 1-compound 16, in dimethylformamide-

Then, the thus prepared color printing paper was subjected to exposing, developing with a developing solution containing

N-ethyl-N- β -hydroxyethyl-p-phenylenediamine, stop-fixing, washing, bleach-fixing, washing, hardening, washing and dry-

ing. The fading ratio at an image density of 1.0 is shown in the

following table, when the coated paper was exposed by means

water (1:1,) coating and drying.

graphic printing paper in instances where the high molecular weight compound of the invention is used, without limiting the scope of the invention.

EXAMPLE I

1 kg. of a4percent aqueous solution of gelatin was gradually added to 100 g. of a 4 percent solution of the high-molecular ultraviolet absorbers of the invention, compound 1-compound 16, and a known low molecular weight ultraviolet absorber, compound 17, in water-dimethylformamide (1:1).

Compound 17 (for comparison)

The emulsions were coated onto film supports and dried to prepare a filter layer having a thickness of 2.0 microns. The dissolving-out effect of the compounds measured on the thus resulting filters is tabulated below.

of a Xenon tester for 20 hours. The Xenon tester used in this fading test was a light source having a strength and spectrum distribution of energy very similar to the spectrum distribution of the daylight energy.

Additive	Decrease ratio of optical density			(350 m _μ)	
Process	Water washing	water washing	3% aqueous NA ₂ CO ₃ solution	overall photo- graphic pro- cessing	
Time	30 min.	2 hours	30 min.	25 min.	
Compound 1	8%	10%	9%	9%	
Compound 2	11%	13%	12%	12%	
Compound 3	10%	12%	11%	11%	
Compound 4	5%	6%	6%	6%	
Compound 5	0 .	2%	1%	0	
Compound 6	13%	15%	14%	14%	
Compound 7	3%	4%	4%	4%	
Compound 8	0	2%	0	0	
Compound 9	2%	3%	3%	2%	
Compound 10	12%	14%	13%	13%	
Compound 11	2%	3%	3%	3%	
Compound 12	10%	12%	11%	11%	
Compound 13	13%	15%	14%	14%	
Compound 14	12%	14%	13%	13%	
Compound 15	10%	12%	11%	11%	
Compound 16	11%	13%	12%	12%	
Compound 17	85%	95%	90%	90%	

It is apparent from these results that the high molecular weight compounds of the invention, compounds 1-16, are only slightly dissolved out and very diffusion resistant while a low molecular weight, diffusible substance, such as known compound 17, almost completely dissolves out from a gelatin so layer in the steps of photographic processing and washing. The overall photographic processings designated herein involve the steps of developing, stop-fixing, washing, bleach-fixing, washing, hardening, washing and drying, ordinarily used in color photography.

Each of the high molecular weight compounds used in the invention gave a similar effect to the above-mentioned when having a limit viscosity $[\eta]$ of 0.1 to 2.0 in a solvent of dimethylformamide-water (1:1) at 30° C.

EXAMPLE II

A blue-sensitive silver iodobromide emulsion containing 3,5-di-carboxy- α -(4-stearoylamidebenzoyl) acetanilide, yellow coupler was coated onto a baryta paper. The coupler was added as an alkaline aqueous solution. Then, a green-sensitive silver chlorobromide emulsion containing 1-(3-sulfo-4-phenoxyl)-3-stearoyl-5-pyrazolone, magenta coupler and further a red-sensitive silver chlorobromide emulsion containing N-n-octadecyl-1-hydroxy-4-sulfo-2-1-naphthamide, cyan coupler were coated thereon and further a protective layer

Fading ratio of color image after being exposed for 20 hours by Xenon tester. 30 Color image Additive Yellow Magenta Compound 1 109 2% 11% Compound 2 12% 2% 13% Compound 3 10% 35 Compound 4 10% 2% 11% Compound 5 6% Compound 6 12% 9% 7% 8% 1% Compound 7 Compound 8 6% 1% 8% 1% Compound 9 40 Compound 10 12% 2% 13% 1% 9% Compound 11 8% No addi-40% 20% 40%

As is evident from the test results, fading of a color image in a case where no ultraviolet absorber is added to a protective layer can be reduced by the use of the ultraviolet absorber of the invention. In instances where such ultraviolet absorbing layer is not used for a protective layer but is used for an intermediate layer between a first emulsion layer and second emulsion layer, as well as, between a second emulsion layer and third emulsion layer, the fade preventing effect on the color image appears in the layer under the intermediate layer containing the ultraviolet absorber, as expected.

In the case of using a film base as a support in place of the baryta paper, substantially similar results were obtained. The stain preventing effect of the thus provided and processed color printing paper is shown in the following table, in which the stain is represented by the increase of density of the yellow component in the nonexposed area when using a Xenon tester as an ultraviolet light source.

Density of yellow component before and after exposure by Xenon tester for 20 hours

65

	Additive	Before Exposure	After exposure	
70 —	Compound 1	. 0.11	0.12	
	Compound 2	0.11	0.12	
	Compound 3	0.11	0.12	
	Compound 4	0.11	0.12	
	Compound 5	0.11	0.12	
	Compound 6	0.11	0.12	
75	Compound 7	0.11	0.12	
	Compound 8	0.11	0.12	

	Table – Continue	ed
Compound 9	0.11	0.12
Compound 10	0.11	0.12
Compound 11	0.11	0.12
Compound 12	0.09	0.12
Compound 13	0.09	0.12
Compound 14	0.09	0.12
Compound 15	0.09	0.12
Compound	0.09	0.12
No additive	0.13	0.20

It will be apparent from these test results that the stain preventing effect can be given by adding the high molecular weight compound of the invention to a protective layer. The brightening degree of a color printing paper can be improved more markedly by adding the high molecular weight compound of the invention, as compared with the case of using no additive.

EXAMPLE III

The first emulsion layer of example II, i.e., 1 kg. of the redsensitive silver chlorobromide emulsion was gradually added to 50 g. of a 4 percent solution of each of the high-molecular weight ultraviolet preventing materials of the invention, compound 1-compound 11 in dimethylformamide-water (1:1) and mixed completely, to which the cyan coupler was added (thickness: 4.0 microns). The magenta layer as a second layer and the yellow layer as a third layer were the same as those of example II.

The thus prepared multiemulsion layer sensitive materials were subjected to exposing, developing, stop-fixing, washing, bleach-fixing, washing, hardening, washing and drying and the fading ratio at a color image density of 1.0 was measured after exposure for 20 hours by means of a Xenon tester to obtain the following results.

Fading ratio of color image after being exposed for 20 hours by Xenon tester			
Additive	Cyan	Magenta	Yellow
Compound 1	22%	2%	12%
Compound 2	25%	2%	14%
Compound 3	23%	2%	13%
Compound 4	22%	2%	12%
Compound 5	13%	. 0	8%
Compound 6	25%	2%	15%
Compound 7	17%	1%	10%
Compound 8	14%	1%	8%
Compound 9	18%	1%	10%
Compound 10	25%	2%	15%
Compound 11	16%	1%	11%
lo additive	40%	20%	40%

It will be apparent from the test results that a very large ultraviolet preventing effect can be given by adding the high molecular weight ultraviolet preventing material of the invention directly to an emulsion even though providing no particular ultraviolet preventing layer.

EXAMPLE IV

To a filter (subbing layer) containing each of the compounds 1-11 of the invention, prepared in the same manner as described in example 1 (thickness: 2.0 microns) was applied a blue-sensitive silver iodobromide emulsion containing the yellow coupler as in example II, a green-sensitive silver 70 chlorobromide emulsion containing the magenta coupler, a red-sensitive silver chlorobromide emulsion containing the cyan coupler, and then a protective layer.

The thus prepared multiemulsion layer sensitive materials were subjected to exposing, developing, stop-fixing, washing,

bleach-fixing, washing, hardening, washing and drying and the fading ratio at a color image density of 1.0 was measured after exposure for 20 hours from the back of the support by means of a Xenon tester to obtain the following results.

Fading ratio of color image after being exposed for 20 hours by Xenon tester

10	Additive	Cyan	Magenta	Yellow	
	Compound 1	10%	2%	11%	
	Compound 2	119	2%	13%	
	Compound 3	9%	2%	11%	
5	Compound 4	10%	2%	10%	
•	Compound 5	6%	0	7%	
	Compound 6	12%	2%	12%	
	Compound 7	7%	154	9%	
20	Compound 8	5%	1%	7%	
	Compound 9	8%	1%	8%	
	Compound 10	11%	29€	12%	
	Compound 11	7%	1%	9%	
	No additive	40%	20%	40%	

As is evident from the test results, fading of a color image in a case where no ultraviolet absorber is added to a subbing layer (lowermost filter layer) can be prevented or reduced by the use of the ultraviolet absorber of the invention. In instances where such an ultraviolet absorbing layer is provided not on a subbing layer but on the back of the support, a substantially similar fade preventing effect was obtained. Moreover, where the ultraviolet absorber of the invention is added to a protective layer and a subbing layer or on the back of the support, also, a substantially similar fade preventing effect to light from the emulsion surface, as well as light from the back of the support appears.

EXAMPLE V

40 10 g. of a 4 percent solution of each of the high molecular weight compounds of the invention, compound 1-compound 16, in dimethylformamide-water (1:1) was gradually added to 1 kg. of a chlorobromide emulsion for enlargement, mixed with a necessary hardener and wetting agent, and coated onto a baryta paper. The thus prepared printing paper held the same brightening degree as the nonprocessed paper, while another printing paper prepared by the use of the known low molecular material, compound 17 lost a substantial amount of brightness after the steps of developing, fixing and washing.

What is claimed is:

1. A photographic light-sensitive material containing a silver halide emulsion layer and in which a polymer having recurring units represented by the following formula is incorporated into at least one layer on said material.

wherein R_o represents a member selected from the class consisting of a hydrogen, an alkyl group having from one to eight carbon atoms, an aryl group having from six to 12 carbon atoms, a hydroxyalkyl group having from two to four carbon atoms, an alkoxyalkyl group having from two to six carbon atoms, a sulfoalkyl group having from one to four carbon atoms, and alkali metal salts and an ammonium salt of said sulfoalkyl group, R₁ and R₂ represent a member selected from the class consisting of, halogens,—OR,—SR,

$$-N$$
, and $-N$ A

wherein R and R' represent a member selected from the class consisting of a hydrogen, an alkyl group having from one to 12 carbon atoms, a hydroxyalkyl group having one to four carbon atoms, a sulfoalkyl group having from one to four carbon atoms, alkali metal salts and ammonium salts of said sulfoalkyl group, carboxy alkyl groups having from one to four carbon atoms, alkali metal salts and ammonium salts of said carboxyalkyl group, an aralkyl group having from seven to 14 carbon atoms, an aryl group having from six to 18 carbon atoms, and a cycloalkyl group having from six to 10 carbon atoms, said R and R' being the same or different; and A represents a member selected from the class consisting of $-(CH_2)_4-, (CH_2)_5$,-,- $(CH_2)_2$ -o- $(CH_2)_2$ -and- $(CH_2)_2$ -S- $(CH_2)_2$ -, said R₁ and R₂ being the same or different, and M represents a member selected from the class consisting of al- 20 kali metals and ammonium.

2. A photographic light-sensitive material as claimed in claim 1, wherein said polymer is a copolymer, and additionally contains at least one recurring unit represented by the following formula:

(HOCH2CH2)2N

-CH2

s OaNa

so₂Na

NaO₃S

wherein X represents a member selected from the class consisting of -OH, -OCH₃, -OCH₂CH₃, -OCH₂CH₂OH, -OCOCH₃, -OCOCH₂CH₃, -OCONH₂, -OCOHNCH₂OH, -CONH₂, -CONHCH₃, -CON(CH₃)₂,

3. A photographic light-sensitive material as claimed in claim 1, wherein said polymer has a recurring unit selected 25 from the class consisting of,

SO₃Na

SO₃Na

SO3Na

and

- 4. The photographic light-sensitive material as claimed in claim 2, wherein said recurring units of claim 1 are present in an amount of from 0.1 to 50 mole percent, based on the 35 polymer backbone units.
- 5. The photographic light-sensitive material as claimed in claim 4, wherein said recurring units of claim 1 are present in

claim 1, wherein said polymer has a molecular weight of from 20,000 to 1,000,000.

- 7. The photographic light-sensitive material as claimed in claim 6, wherein said molecular weight is from 100,000 to 400,000.
- 8. The photographic light-sensitive material as claimed in claim 1, wherein said recurring units have the formula:

(M.W.=400,000)

an amount of from 0.5 to 10 mole percent.

- n amount of from 0.5 to 10 mole percent.

 9. The photographic light-sensitive material as claimed in claim 1, wherein said recurring units have the formula:

(M.W.=400,000)

70