

PATENT SPECIFICATION

NO DRAWINGS

1.056.455

1.056.455



Date of Application and filing Complete Specification: July 10, 1963.

No. 33018/66.

Application made in United States of America (No. 209263) on July 11, 1962.

(Divided out of No. 1056454).

Complete Specification Published: Jan. 25, 1967.

© Crown Copyright 1967.

Index at acceptance: —G2 C(C10A3, C10D)

Int. Cl.: —G 03 c

COMPLETE SPECIFICATION

Photographic Processing Compositions Containing Boron Compounds

We, EASTMAN KODAK COMPANY, a Company organized under the Laws of the State of New Jersey, United States of America, of 343 State Street, Rochester, New York 14650, United States of America, (Assignees of WESLEY TURNELL HANSON JR. and HOWARD WALTER VOGT), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to compositions capable of rendering a photographic silver halide layer developable.

In our parent application 27275/63 Serial No. 1,056,454 there is described and claimed a process of rendering developable a photographic layer having an image-wise distribution of silver halide which comprises subjecting said layer to treatment with an aqueous solution of a boron compound which has at least one and not more than three hydrogen to boron bonds or which contains two or more amine groups each bonded to a BH_2 .

Embodiments of the method of the invention of application 27275/63 Serial No. 1,056,454 include treating with said aqueous solutions of boron compounds exposed black and white photographic materials in the production of black and white reversal images and exposed colour photographic materials in the production of colour reversal images.

According to the present invention there is provided an aqueous solution containing a boron compound having at least one and not more than three boron to hydrogen bonds or a boron compound containing two or more amine groups each bonded to a BH_2 group, said aqueous solution containing an added acid or base and the pH thereof being not lower than 3 nor higher than 13.

Examples of the boron compounds which

are used in this invention are given in application 27275/63 Serial No. 1,056,454. These compounds include monoalkyl-amine boranes, dialkyl-amine boranes trialkyl-amine boranes, monoaryl-amine boranes and heterocyclic amine boranes.

The composition comprising boron compounds provided by this invention may be used in aqueous solution in the manner described in application 27275/63 Serial No. 1,056,454.

To make the solution alkaline sodium hydroxide or sodium carbonate may be added and to make the solution acid acetic acid may be added.

The boron compounds defined herein are sufficiently stable in aqueous solutions at pHs as low as 3 and are thus useful in the range from pH3 to pH13, the particular level of pH depending on the sensitometric results desired. The concentration of the boron compound may be from 1 to 1000 milligrams per litre.

The aqueous solutions of this invention are particularly valuable for processing colour photographic materials which contain colour couplers in the silver halide emulsion layers since the boron compounds in these solutions are sufficiently stable at the low pHs used in the processing of these materials.

As described in our application 27275/63 Serial No. 1,056,454 in the processing of photographic materials containing colour couplers the colour developing solution may contain the boron compound whereby the reversal silver halide images in the material (which has been exposed and developed in a black and white developer) are rendered developable and are colour developed by treatment with a single solution.

Accordingly, this invention also provides a photographic colour developer comprising a

[Price 4s. 6d.]

colour developing agent and a boron compound as herein defined.

- 5 The preferred concentration of boron compound in the colour developer is from 10 to 100 milligrams per litre. The pH of the solution may be acid or alkaline, e.g. up to a pH of 11.6.

- 10 Preferred boron compounds for use in the colour developer are dimethylamine borane, trimethylamine borane, diethylamine borane, triethylamine borane, *t*-butylamine borane, pyridine borane and 2,6-lutidine borane.

- 15 As described in application 27275/63 Serial No. 1,056,454 the aqueous solutions of boron compounds provided by this invention may also be used in processing colour photographic materials not containing colour couplers. As described in application 27275/63 Serial No. 1,056,454 the colour development of the last
20 reversal silver halide image of the material (the other layers already having been reversally colour developed) may take place simultaneously with or subsequently to the treatment of the layer with the aqueous solution of the boron compound.
25

- 30 An aqueous solution containing a boron compound provided by this invention used to render the last reversal silver halide image in such a material developable as a separate step from subsequent colour development may contain from 100 to 400 milligrams of the boron compound per litre. The pH of the solution may be, for example, 12.3.

- 35 Preferred boron compounds include *t*-butylamine borane, dimethylamine borane, trimethylamine borane, diethylamine borane, triethylamine borane, pyridine borane and 2,6-lutidine borane.

- 40 The aqueous solution may also comprise a sequestering agent such as sodium hexametaphosphate or may contain citrazinic acid.

Examples of solutions of this invention are as follows:

EXAMPLE 1

	grams
sodium hexametaphosphate	1
sodium hydroxide	1.5
<i>t</i> -butylamine borane	0.4
water to make	1000 ccs

EXAMPLE 2

	cc	grams
benzyl alcohol	6.0	
sodium hexametaphosphate	2.0	
sodium sulphite, anhydrous	5.0	
trisodium phosphate	40.0	

potassium bromide	0.25 grams	55
0.1% by weight solution		
potassium iodide	10.0 cc	
sodium hydroxide	6.5 grams	
colour developer*	11.33 grams	
ethylenediamine sulphate	7.8 grams	60
citrazinic acid	1.5 grams	
<i>t</i> -butylamine borane	0.1 grams	
water to make	1000 cc	

* 4 - amino - N - ethyl - N - (β - methane-sulphonamidoethyl) - *m* - toluidine sesquisulphate monohydrate.

Further examples of the present invention were produced by substituting for the *t*-butylamine borane in the above Examples 1 and 2, equal weights of trimethylamine borane, diethylamine borane, triethylamine borane, pyridine borane and 2,6-lutidine borane.

WHAT WE CLAIM IS:—

1. An aqueous solution containing a boron compound having at least one and not more than three boron to hydrogen bonds or a boron compound containing two or more amine groups each bonded to a BH₃ group, said aqueous solution containing an added acid or base and the pH thereof being not lower than 3 nor higher than 13.
2. A solution according to claim 1 containing from 1 to 1000 milligrams of said boron compound per litre.
3. A solution according to claim 2 containing from 100 to 400 milligrams of said boron compound per litre.
4. A solution according to claim 1 or 2 which is a colour developer containing a colour developing agent and said boron compound.
5. A solution according to claim 4, the concentration of boron compound being from 10 to 100 milligrams per litre.
6. A solution according to any of the preceding claims, the boron compound being a monoalkyl-amine borane, a dialkyl-amine borane, a trialkyl-amine borane, a monoaryl-amine borane or a heterocyclic-amine borane.
7. A solution according to claim 6, the boron compound being *t*-butylamine borane, dimethyl-amine borane, trimethyl-amine borane, diethyl-amine borane, pyridine borane or 2,6-lutidine borane.
8. An aqueous solution containing a boron compound as defined in claim 1, substantially as described in the Examples herein.

L. E. T. BRANCH, B.Sc., F.R.I.C.
Patent Agent

B.K.

colour developing agent and a boron compound as herein defined.

- 5 The preferred concentration of boron compound in the colour developer is from 10 to 100 milligrams per litre. The pH of the solution may be acid or alkaline, e.g. up to a pH of 11.6.

- 10 Preferred boron compounds for use in the colour developer are dimethylamine borane, trimethylamine borane, diethylamine borane, triethylamine borane, *t*-butylamine borane, pyridine borane and 2,6-lutidine borane.

- 15 As described in application 27275/63 Serial No. 1,056,454 the aqueous solutions of boron compounds provided by this invention may also be used in processing colour photographic materials not containing colour couplers. As described in application 27275/63 Serial No. 1,056,454 the colour development of the last
- 20 reversal silver halide image of the material (the other layers already having been reversally colour developed) may take place simultaneously with or subsequently to the treatment of the layer with the aqueous solution of the
- 25 boron compound.

- 30 An aqueous solution containing a boron compound provided by this invention used to render the last reversal silver halide image in such a material developable as a separate step from subsequent colour development may contain from 100 to 400 milligrams of the boron compound per litre. The pH of the solution may be, for example, 12.3.

- 35 Preferred boron compounds include *t*-butylamine borane, dimethylamine borane, trimethylamine borane, diethylamine borane, triethylamine borane, pyridine borane and 2,6-lutidine borane.

- 40 The aqueous solution may also comprise a sequestering agent such as sodium hexametaphosphate or may contain citrazinic acid.

Examples of solutions of this invention are as follows:

EXAMPLE 1

- 45
- | | grams |
|-----------------------------|----------|
| sodium hexametaphosphate | 1 |
| sodium hydroxide | 1.5 |
| <i>t</i> -butylamine borane | 0.4 |
| water to make | 1000 ccs |

EXAMPLE 2

- 50
- | benzyl alcohol | 6.0 cc |
|----------------------------|------------|
| sodium hexametaphosphate | 2.0 grams |
| sodium sulphite, anhydrous | 5.0 grams |
| trisodium phosphate | 40.0 grams |

potassium bromide	0.25 grams	55
0.1% by weight solution		
potassium iodide	10.0 cc	
sodium hydroxide	6.5 grams	
colour developer*	11.33 grams	
ethylenediamine sulphate	7.8 grams	60
citrazinic acid	1.5 grams	
<i>t</i> -butylamine borane	0.1 grams	
water to make	1000 cc	

* 4 - amino - N - ethyl - N - (β - methane-sulphonamidoethyl) - *m* - toluidine sesquisulphate monohydrate.

65 Further examples of the present invention were produced by substituting for the *t*-butylamine borane in the above Examples 1 and 2, equal weights of trimethylamine borane, diethylamine borane, triethylamine borane, pyridine borane and 2,6-lutidine borane.

70

WHAT WE CLAIM IS:—

1. An aqueous solution containing a boron compound having at least one and not more than three boron to hydrogen bonds or a boron compound containing two or more amine groups each bonded to a BH₂ group, said aqueous solution containing an added acid or base and the pH thereof being not lower than 3 nor higher than 13.

75

2. A solution according to claim 1 containing from 1 to 1000 milligrams of said boron compound per litre.

3. A solution according to claim 2 containing from 100 to 400 milligrams of said boron compound per litre.

80

4. A solution according to claim 1 or 2 which is a colour developer containing a colour developing agent and said boron compound.

5. A solution according to claim 4, the concentration of boron compound being from 10 to 100 milligrams per litre.

85

6. A solution according to any of the preceding claims, the boron compound being a monoalkyl-amine borane, a dialkyl-amine borane, a trialkyl-amine borane, a monoaryl-amine borane or a heterocyclic-amine borane.

90

7. A solution according to claim 6, the boron compound being *t*-butylamine borane, dimethyl-amine borane, trimethyl-amine borane, diethyl-amine borane, pyridine borane or 2,6-lutidine borane.

95

8. An aqueous solution containing a boron compound as defined in claim 1, substantially as described in the Examples herein.

100

L. E. T. BRANCH, B.Sc., F.R.I.C.
Patent Agent

B.K.

