

Archival processing of black-and-white photographs as preventive conservation

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ABSTRACT

The need for producing large quantities of black-and-white photographs calls for automation of the processing. The need for producing long lasting images calls for continuous quality control of the processing. At the Department of Conservation at the National Museum of Denmark we have combined these often contradictory demands. Tests for residual silver and fixer are frequently used on film negatives as well as prints. A modified processor for prints is used which ensure the removal of all processing chemicals. Negatives for special purposes are stabilised after processing in polysulfide and all prints in selenium solution.

KEYWORDS

Black-and-white photographs, machine processing, quality control, archival standard, stabilisation.

1. INTRODUCTION

The responsibility for preservation of photographic images can be divided into several stages. First the manufacturer is responsible for the choice of materials and the manufacturing processes. Second the processor is responsible for the proper use of processing solutions and for washing out of the chemicals. Third the user has the responsibility for correct storage, handling and exhibition of the photographs.

At all stages the national and international standards guide how to make photographic images permanent. Today only cellulose acetate and polyester polymers are considered as base materials for films which have permanent value [E.g.; DS/ISO 543 or ANSI IT9.1]. Polyester is superior to acetate film. It is estimated that polyester film base has a useful life of 500 years compared to 100 years for acetate when stored at 20°C, 50% relative humidity (RH) [Adelstein et al., (1991) 353].

Several standards describe criteria for processing of films [ANSI IT9.1] and papers [ANSI PH4.32] for permanence. To estimate the stability of the processed photographs, other standards describe methods for measuring the amount of residual fixer in films and papers [ANSI/ASC PH4.8] and the conversion of image silver to more stable compounds such as silver selenide or silver sulfide [ANSI/NAPM IT9.15]. Fine grain black and white films should not contain more than 0,7µg/cm² of residual fixer after processing [ANSI IT9.1-1992, 5] and if a high percentage of the image silver is converted in a toning bath to silver sulfide or silver selenide the image should not discolour even in an atmosphere with a relatively high concentration of oxidising gases [ANSI/NAPM IT9.15-1993, 3].

After processing, the images which have permanent value should be stored, used and exhibited following recommendations on the storage environment and

materials used for storage and exhibition [e.g. DS/ISO 3897, DS/ISO 5466, DS/ISO 6051 or ANSI IT9.2, ANSI IT9.11]. In short, photographs should never be kept above 25°C and 60% RH but preferably as cold as possible, at 20-50% RH, in an atmosphere without oxidizing gases, enclosed by, or mounted in archival-quality materials.

In general if these recommendations are followed excellent stability of the material can be expected.

This paper describes the processes we have developed at the Department of Conservation at the National Museum of Denmark which enable us to follow the standards and guidelines for making photographs permanent and at the same time respond to the demand for a large production of pictures.

2. THE PHOTOGRAPHIC WORK AT THE DEPARTMENT OF CONSERVATION

Three photographers serve the approximately 80 conservators at the department with documentation and process photographs in both black-and-white and colour. Moreover the photographers work for other departments at the National Museum as well as other institutions and private persons. They, together with the photograph conservator, also make safety copies and duplicate negatives for clients both inside and outside the Museum. All black-and-white processing and printing is done in house while colour processing except prints from colour slides (Ilforchromes, formerly known as Cibachromes) is done by commercial processing labs.

The demand for archival processing of large numbers mainly of black-and-white negatives and prints needs a high level of automation of the processes as well as quality control of the processing. We now use a processing machine as well as tanks for processing of negatives. All prints are machine processed. Prints and duplicate negatives are stabilised with selenium and polysulfide respectively.

3. MACHINE PROCESSING OF NEGATIVES

A standard processing machine normally consists of three tanks for the developer, for the fixer and the rinse. Archival processing of black-and-white negatives needs more tanks for processing solutions and variable washing possibilities. We choose a Jobo AutoLab ALT-3 processor. This model contains 12 bottles for processing chemicals and the possibility of programming the length of each individual processing step. Moreover within each step it is possible to wash the film (or paper) for any length of time up to 90 minutes. This makes the model suitable for archival processing.

For making duplicate negatives of historic glass plate or cellulose nitrate negatives we programme the following processing steps: Prerinse, developer, stop bath, fixer 1, fixer 2, rinse, hypo clearing agent, wash,

wetting and then normal drying in a dry chamber. For duplicates which require special stabilising treatment the sequence after the hypo clearing agent is: rinse, polysulfide treatment, hypo clearing agent (again), wash, wetting and drying. All processing is done automatically which means that the operator can prepare another drum while the first is processing.

4. MACHINE PROCESSING OF PRINTS

In spite of much scepticism, resin-coated (RC) photographic paper is now commonly accepted for making permanent prints [Reilly, 1991, 19 & 30]. For many years we have used a standard tray processing procedure for RC-papers, followed by a stabilising selenium treatment. No detectable residual fixer is left, according to the measuring methods described in the ANSI PH4.8 standard. To respond to the demand for prints at the Conservation Department we have tested several commercial machine processors. No processor could make prints with the small amount of residual fixer left by the tray processing procedure.

Three variables are important when prints are processed in a machine. First, for making prints as quickly as possible the dry-to-dry time should be as short as possible. Normally prints are no more than 20 seconds in the wash water leaving a lot of residual fixer in the prints. Secondly, the replenish rate of developer and fixer might be low in order to save chemicals. This results in a high amount of complex silver thiosulfate compounds in the fixer solution. Thirdly to save energy and avoid washing out of the optical brightener in the print paper, it is recommended that the temperature of the wash water should not be too high. However, all three processes result in a high concentration of residual chemical compounds in the prints. Although these might not discolour immediately after processing, it might happen very quickly when later the prints are exposed to an oxidising atmosphere, for instance when displayed on a freshly painted wall.

Although no standard configurations of the tested processors could process prints to the same quality as tray processing, we decided for practical reasons to continue the tests on an Ilford 2240RC Processor. The wash water in this processor comes to the wash tank from an inlet in the right side of the processor, while the outlet is placed in the left side of the machine. Our initial tests showed that prints processed on the right side did not contain measurable amounts of thiosulfate while those processed on the left side contained measurable thiosulfate. Ilford Anitec A/S in Denmark agreed to build an extra spray bar into the washing tank. Now the whole processing band is provided with fresh wash water (see also figure 1). Moreover the dry-to-dry time was increased from 70 to 120 seconds, which means a little longer stay for the prints in the wash tank.

Next the best temperature for the wash water had to be decided. The results are shown in table 1 and show first that the residual fixer is removed at the same rate independently of where it has been placed on the processing band. The temperature of the wash water is recommended to be between 25°C and 30°C. Too high

a temperature can result in washing out of the optical brightener in the paper. Without optical brightener the print will look a little more yellow (or less white). This will happen anyway when the print is exhibited since the optical brightener will bleach.

Experience from three years of operation has shown that the quality of the prints is constant and comparable to the initial test results. As one might expect, a few problems with the machine have occurred. Once, failure to replenish fixer resulted in prints that immediately showed yellowed stains. In another case even selenium toned prints became yellow after a few weeks on a notice board. While the reason for this is not clear it might originate in the processing. Constant control of the operation of the machine is recommended. Even with stabilising aftertreatment frequent checking of the processing is essential for making long lasting RC prints.

5. STABILISING AFTERTREATMENT FOR NEGATIVES AND PRINTS

Photographic image silver is less stable to oxidation than silver salts such as silver sulfide and silver selenide. Treatment which change the image silver to the more stable compounds will protect the image silver against fading and discoloration.

In the mid 1980s selenium treatment was recommended for extending the stability of black and white films and prints in several publications [Lee et al., (1984) and Drago et al., (1986)]. On this background all RC-prints and duplicate negatives were selenium treated at our department. Further research around 1990 showed that the selenium treatment (Kodak Rapid Selenium Toner, (KRST)) did not effectively protect the image silver specially in the low density areas of photographs [Reilly et al., (1989) 124-125 and Reilly et al., (1991), 101-102]. On the other hand the effectiveness of a polysulfide treatment has been demonstrated recently [Reilly and Cupriks, 1991] and commercial solutions and recipes are available (Kodak Brown Toner, IPI SilverLock).

In 1993 a new ANSI standard [ANSI/NAPM IT9.15] was published recommending methods for measuring the effectiveness of stabilising treatments for silver gelatin images. We therefore decided to compare selenium and sulfur treatments for the different black-and-white films and print papers used at the Conservation Department.

5.1. The test methods

The ANSI/NAPM-standard describes two test methods for evaluation of stabilising treatments. The hydrogen peroxide test is an accelerated aging test that simulates the effect of oxidising gases. Oxidising gases can be released from envelopes, shelves, paints or from polluted air outside the archive. The blackness of each step on a step wedge (grey scale) is measured with a blue filter on a densitometer before incubation. After 18 hours at 50°C and 80% relative humidity (RH) in 1000 ppm hydrogen peroxide the step wedge is redeveloped to reduce ionised silver, dried and measured

again. The density change (change in the blackness of each step) is corrected for any discoloration in the base and reported as density change after incubation. In the graphs this is reported as sensitivity to peroxide attack. The standard suggests that less than 0,05 density change after incubation represents effective stabilisation against peroxides.

An indirect measure of the amount of silver altered from metallic image silver to silver sulfide is described in the standard. It is assumed that all metallic silver can be bleached away by an acid potassium dichromate solution, leaving compounds of silver selenide or silver sulfide untouched. The grey scale containing the unbleachable silver salts is measured with a blue filter in the densitometer. The result is expressed as the percentage of the blue density before bleaching. In the graphs this is reported as percent alteration of silver.

In annex A of the standard the correction of density values is described. The back correction of the reflection density gives unrealistic and wrong values when used on ordinary photographic print papers. This problem will be considered when the standard is revised according to correspondance with the chairman of the standard committee. The reflection densities presented in this paper (figure 6-7) are therefore calculated in the same way as described for transmission density in the standard.

5.2. Selenium and polysulfide treatment of prints

Recommendations from Kodak concerning the use of Kodak Rapid Selenium Toner to protect the prints has varied. On the bottle of KRST is printed: 'To enhance D-max areas and protect the image, dilute toner 1:20 or 1:40. To save time, mix toner with hypo clearing agent instead of water'. Although the treatment time is not clear, the treatment time for toning is stated as 2-8 minutes in a solution containing 1 part of toner to 3-19 parts of water. In a paper from 1984 there is a recommendation to dilute 1 part of KRST with 3 parts of water and treat for 4 minutes [Lee et al. (1984) 126]. In another publication from 1986 a solution of 1 part of KRST to 10 parts of water for 6-8 minutes is recommended for both FB and RC papers [Drago and Lee, (1986) 63-64]. From Kodak AB, Sweden comes a recommendation to mix 1 part of KRST with 20 or 40 parts of a working solution of KHCA. [SmÅ Korn Nr. 1 (1994) 43].

The Image Permanence Institute (IPI) at the Rochester Institute of Technology has developed a polysulfide treatment to protect microfilms from oxidative attack, which is called IPI SilverLock [Image Permanence Institute (1992)]. For prints we have tested a solution of one part of IPI SilverLock to 25 parts of water for 1-15 min.

In table 2 all test results on Ilford Multigrade RC and FB papers are represented. Figures 2-5 show images after treatment with selenium (figure 2) and an untreated print after the acid dichromate bleach test (figure 3). Figure 4-5 shows dichromate bleached prints after treatment in selenium and polysulfide respectively.

Figure 6 demonstrates that selenium and sulfur treated prints resist peroxide attack better than untreated prints. However, it is also clear that prints treated with a dilution of 1 part of KRST to 20 parts of water or more do not meet the test criteria formulated in the ANSI/NAPM IT9.15 standard. To protect the image against oxidation, a solution of one part of toner to 3 parts of water for 4 minutes is needed. In figure 7 we might find an explanation. This figure shows the percent alteration of silver to silver sulfide or silver selenide. Untreated Ilford Multigrade RC papers probably contains an antioxidant compound which results in an average of 20% alteration (curve no. 1) without any treatment at all. After treatment in 1 part of KRST to 20 parts of water, only the darkest steps on the grey scale are altered to silver selenide while there is almost no alteration in the highlights. The same pattern can be seen when prints are treated in a more concentrated solution (1 part of KRST to 3 parts of Water, curve nr. 3). However, this is enough to resist peroxide attack. For the polysulfide treated image a more consistent conversion of silver to silver sulfide in all grey patches can be seen, which results in a very resistant image. 3 minutes in the polysulfide solution is plenty.

The recommended procedure of mixing selenium with Kodak Hypo Clearing Agent (KHCA) seems to reduce the amount of silver converted to silver selenide. Table 2 show that the average amount of conversion is reduced from 75 to 70% when mixed with a working solution of fresh hypo clearing agent.

The test results shown in table 2 and figure 6-7 indicate problems, specially in the highlights when using selenium to stabilise silver images. The results suggests that polysulfide treatment should be preferred to selenium. On the other hand selenium treated prints are generally found more attractive than polysulfide treated prints. Selenium toning enhances the black image tones, while the polysulfide treated images appear more reddish black. This is true for Ilford Multigrade papers developed in Ilford 2000 RT developer but might be different with other combinations of developers and print papers.

Our tests have lead to the conclusion that we treat all RC and FB prints in a working solution of 1 part of Kodak Rapid Selenium Toner to 3 parts of water for 5 minutes. In this case one gallon of working solution can be used for 200 20x25 cm prints. Quality control of every 50th print has shown that they all pass the hydrogen peroxide test.

5.3. Selenium and polysulfide treatment of film negatives

1 part of KRST to 19 parts of water, with a treatment time of 3 minutes has been recommended for normal black-and-white films, such as Kodak Plus-X and Kodak Tri-X. Protection against formation of microspots on spectroscopic plates can be achieved by selenium treatment [Drago and Lee (1986) 63-64].

IPI SilverLock was originally developed for microfilm but we have used a dilution of 1 part of stock solution

to 50 parts of water for 1 and 3 minutes to test the effect on ordinary black-and-white films.

All our test results so far, with selenium diluted in both water and KHCA and polysulfide are presented in table 3. In figure 8-10 a comparison of the two treatment processes are shown.

Figure 8 demonstrates the effect of selenium and polysulfide treatment on a Kodak T-max 100 film after incubation in a hydrogen peroxide atmosphere. It shows that untreated metallic image silver is very sensitive to peroxide attack (curve no. 1). The protection is good for both films treated with selenium (curve no. 3-4) and polysulfide (curve no. 5). On the other hand an incomplete reaction has occurred with the normal recommended selenium solution (1+20 for 3 minutes, curve no. 2). It can be concluded that treated films are more resistant to peroxide attack, but complete resistance with selenium calls for a more concentrated solution or longer treatment time (25 minutes!) than normally stated.

As for prints, the mixing of KHCA and selenium generally reduces the reaction of image silver to silver selenide. This is specially true for Kodak T-max 100 treated in a 1+20 solution. When this solution is prepared with water, an average of 50% of silver is altered while only 20% is altered when mixed with a fresh working solution of hypo clearing agent. In this case the film treated in the water diluted solution passes the hydrogen peroxide test, while the result is negative when the solution is mixed with KHCA. When a more concentrated solution of selenium and hypo clearing agent (1+3) is made, the problem is much reduced.

In figure 9 the relation between percent alteration of silver and sensitivity to peroxide attack is presented for the standard recipes of selenium and polysulfide. The polysulfide treated film negatives had a consistent and high conversion of silver to silver sulfide, which results in full resistance to peroxide attack. This is not true for the selenium treated film negatives. Even though they have received exactly the same treatment (which includes processing) the results are very different. The only common result is that no film passes the peroxide test. The amount of silver converted to a more stable compound varies from 5 to 40%. One could expect that the higher degree of conversion results in higher resistance to peroxide attack, but that is not the case! Only 15% of image silver has converted in the Kodak Plus-X film but this gives almost full resistance to peroxide, while Kodak Tri-X and Kodak T-max 400 show a higher degree of sensitivity although 34% of image silver is altered.

The difference in reaction kinetics of selenium and polysulfide is illustrated in figure 10 where the percent alteration is plotted against increasing density on the grey scale. In all steps on the grey scale, around 70% of silver is converted when normal black-and-white film negatives are treated with IPI SilverLock 1+50 for 3 minutes. From table 3 it can be seen that all three film types pass the peroxide test. The selenium treated films demonstrates a low degree of conversion in the low density steps, with a correspondingly low resistance to peroxide attack. These findings correspond

with results from another study of selenium toning [Reilly et al. (1991) 101-102].

Two main problems in using selenium treatment to stabilise films against oxidation can be deduced from our test. First selenium react very differently with different film types treated in the same solution. It is interesting to note that faster films (400 ISO films such as Ilford HP5, Kodak Tri-X) react more readily with the selenium solution than the slower films (100-125 ISO films such as Kodak Plus-X, Kodak T-max 100 and Ilford FP4 Plus: see figure 9). Secondly, a very high concentration of selenium or a longer treatment time, are needed if film negatives should resist the peroxide tests.

Our conclusion so far is that all duplicate negatives made of historic negative collections should be treated with IPI SilverLock, 1+50 for 3 minutes. Since we keep all documentation negatives made at the conservation department in a climatized storage room, and therefore always can make new prints from the negatives, we have for the moment decided not to treat negatives in a stabilising solution. In general, if negatives are to be stored in places where it is not possible to ensure the absence of oxidising contaminants, we would recommend stabilising film negatives with a polysulfide treatment.

6. CONCLUSION

Traditionally, processing of black-and-white films and prints for archival purpose is considered a slow and manpower demanding. This reputation originates presumably from processing of fiber base prints which can take more than one hour for each print, if double-weight fiber base paper is processed without using a hypo decomposing bath. Moreover, the relatively many yellowed RC prints around have often been processed in machines. This has given automatic processing for archival pictures a bad reputation. In both cases I think the conclusions are right. However, the need for documentation and process photographs and working copies, combined with the economic aspect excludes more subtle processing and printing. Therefore we have tried to develop sound and automatic procedures with a high degree of quality control to respond to the demand for pictures and at the same time process the images for durability. The procedures presented here have resulted in a much more productive work with shorter delivery times. For the future, we plan to look into the processing of colour materials, where in some areas, such as microfilms and colour slides, there might be an advantage in controlling the processing quality to archival standards while still keeping the photographic quality at a professional level.

ACKNOWLEDGEMENTS

I would like to thank Ulla B̄gvad Kejser and Karen Brynjolf Pedersen who have done a lot of the practical work on selenium and polysulfide treatment. I would also like to thank John Lee and Gudrun Jacobsen for processing and printing most of the test materials and Tim Padfield for discussions and revision of the text.

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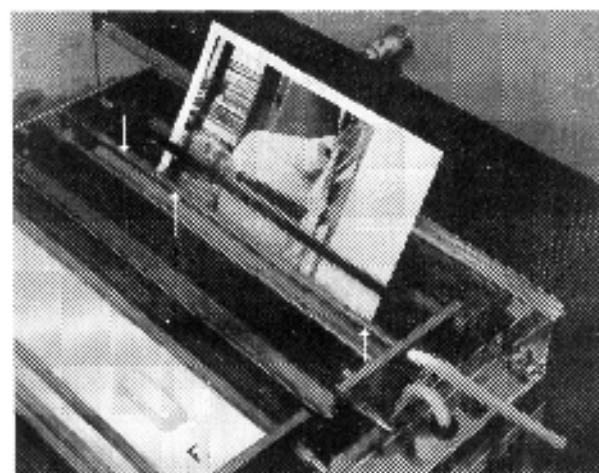


Figure 1: Hford 2240RC Processor rebuilt with an extra spray bar, which irrigates the whole surface of each print with fresh wash water.



Figure 2: Print on Ilford Multigrade RCIV de luxe, after stabilising treatment in Kodak Rapid Selenium Toner 143 for 4 minutes. This treatment prevents discoloration of the print in the ANSI/NAPM 9.16 hydrogen peroxide test.



Figure 4: Print on Ilford Multigrade RCIV treated in Kodak Rapid Selenium Toner 143 for 4 minutes, after bleaching, leaving the "insurance image" of stabilised silver.



Figure 3: Unstabilised print on Ilford Multigrade RCIV de luxe, after ANSI/NAPM 9.15 dichromate bleach test, which removes all reactive image silver, leaving only stable compounds of silver selenide or silver sulfide.



Figure 5: Print on Ilford Multigrade RCIV treated in 111 Silverlock for 3 minutes, after bleaching, leaving the "insurance image" of silver sulfide.

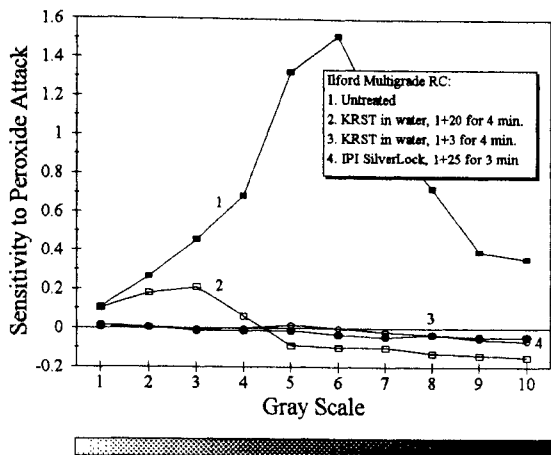


Figure 6: Sensitivity of image silver to hydrogen peroxide. Step 1 on the grey scale represents a low density of silver. The darkness of the patch is measured through a blue filter. High resistance to peroxide means low sensitivity, best close to zero. This means that curves no. 2, 3 and 4 show treatments which can resist the peroxide test.

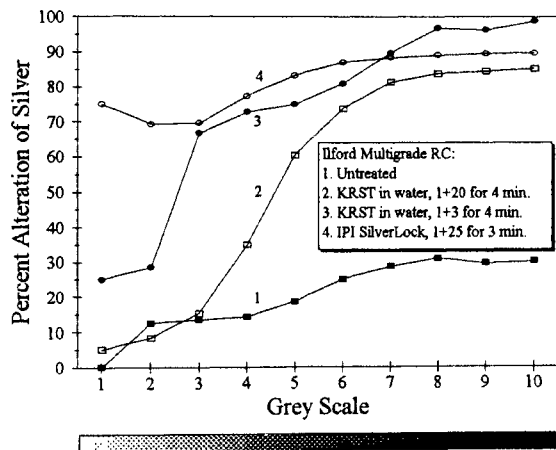


Figure 7: The alteration of silver to an unbleachable silver compound. If all image silver were converted, a horizontal line at 100% could be drawn. This figure shows the difficulty of altering the image silver with selenium toner, specially in the lightest part of the image.

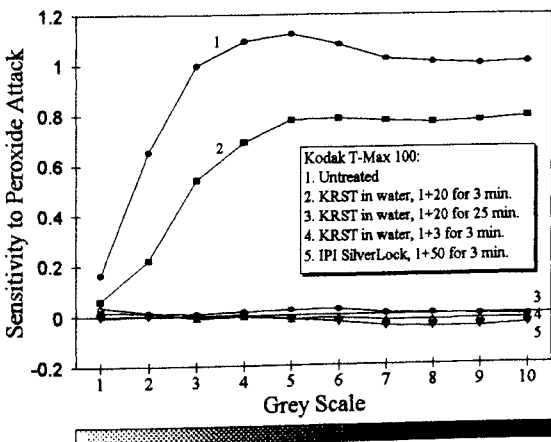


Figure 8: Sensitivity of image silver in Kodak T-max 100 to hydrogen peroxide in different situations. Films treated as shown in curves no. 3, 4 and 5 resist the peroxide test.

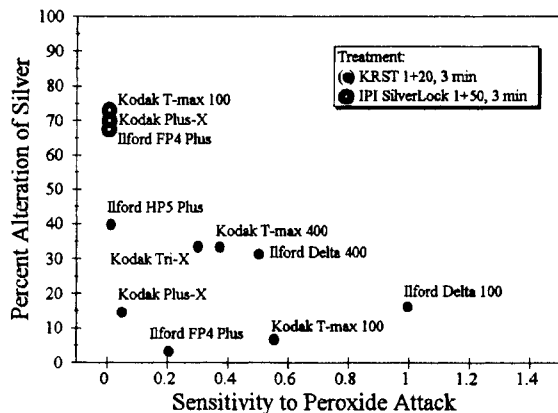


Figure 9: Percent alteration of image silver plotted against sensitivity to peroxide attack, measured in step 3 on the grey scale. Polysulfide treated negatives react consistently with a high percentage of alteration. This results in stable images even in peroxide atmospheres. Selenium treated negatives react very differently and very unpredictably.

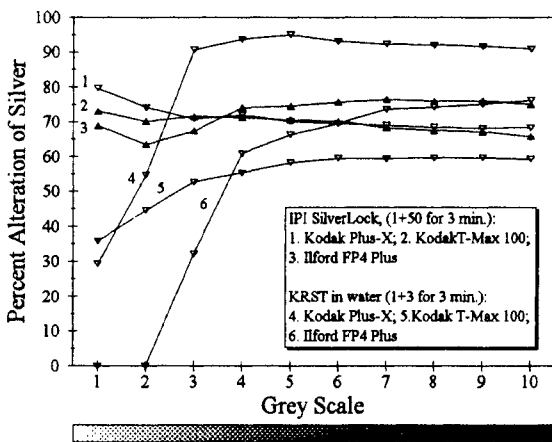


Figure 10: Comparison of the degree of alteration of silver in selenium and polysulfide treated film negatives. The different reaction kinetics is shown. The difficulty of altering the silver to silver selenide in the low density areas of the films is demonstrated very clearly.

Temperature of the wash water, °C	14,00	20,00	26,00	31,00	42,00
Fixer in print washed in the left side, $\mu\text{gS}_2\text{O}_3^-/\text{cm}^2$	0,26	0,16	0,10	nd	nd
Fixer in print washed in the middle, $\mu\text{gS}_2\text{O}_3^-/\text{cm}^2$	0,26	0,16	0,10	nd	nd
Fixer in print washed in the right side, $\mu\text{gS}_2\text{O}_3^-/\text{cm}^2$	0,26	0,16	0,10	nd	nd

Table 1: Residual fixer in prints measured as $\mu\text{gS}_2\text{O}_3^-/\text{cm}^2$ with the Methylene Blue method (ANSI/ASC PH4.8). The prints are processed in either the right or left side, or in the middle. The amount of residual fixer decreases with increasing temperature. A wash water temperature between 25-30°C is recommended (nd = not detectable).

Print paper	Stabilising bath	Dilution with water	Dilution with KHCA	Time, min.	% stabilised silver	Hydrogen Peroxide Test Pass/Fail
Ilford Multigrade RC	Unprotected				20	fail
-	Kodak Selenium	1+20		4	52	fail
-	-	1+3		4	70	PASS
-	IPI SilverLock	1+25		1	80	fail
-	-	1+25		2	82	fail
-	-	1+25		3	85	PASS
Ilford Multigrade FB	Unprotected				47	not tested
-	Kodak Selenium	1+20		5	75	-
-	-		1+20	5	70	-
-	IPI SilverLock	1+25		3	93	-

Table 2: Test results of Ilford Multigrade resin coated (RC) and fiber based (FB) paper according to ANSI/NAPM IT9.15. In column no. 2 Kodak Selenium is identical with Kodak Rapid Selenium Toner. In column no. 4 KHCA is identical with Kodak Hypo Clearing Agent. Column no. 6 reports the average percentage of metallic silver converted to silver salts as measured with the acid dichromate test, while the pass/fail criteria in column no. 7 is less than 0,05 density change after incubation. Testing of fiber base paper with hydrogen peroxide gives unreliable results. The peroxide is probably absorbed in the paper fibres and does not react much with the image silver.

Film	Stabilising bath	Dilution with water	Dilution with KHCA	Time, min.	% stabilised silver	Hydrogen Peroxide Test Pass/Fail
Kodak T-max 100	Unprotected				8	fail
-	Kodak Selenium	1+20		3	7	fail
-	-	1+20		25	50	PASS
-	-	1+3		3	54	PASS
-	-		1+20	3	2	fail
-	-		1+20	25	20	fail
-	-		1+3	3	50	fail
-	IPI SilverLock	1+50		1	50	PASS
-	-	1+50		3	70	PASS
Kodak T-max 400	Kodak Selenium	1+20		3	34	fail
-	-		1+20	3	34	fail
Kodak Plus-X	Unprotected				4	fail
-	Kodak Selenium	1+20		3	15	fail
-	-	1+3		3	82	fail
-	-		1+20	3	11	fail
-	IPI SilverLock	1+50		1	44	fail
-	-	1+50		3	71	PASS
Kodak Tri-X	Kodak Selenium	1+20		3	34	fail
-	-		1+20	3	32	fail
Ilford FP4 Plus	Unprotected				0	fail
-	Kodak Selenium	1+20		3	5	fail
-	-	1+3		3	53	fail
-	-		1+20	3	8	fail
-	IPI SilverLock	1+50		1	40	fail
-	-	1+50		3	73	PASS
Ilford HP5 Plus	Kodak Selenium	1+20		3	40	fail
-	-		1+20	3	42	fail
Ilford Delta 100	Kodak Selenium	1+20		3	17	fail
-	-		1+20	3	20	fail
Ilford Delta 400	Kodak Selenium	1+20		3	32	fail
-	-		1+20	3	32	fail
Agfa Gevatone N33p	Kodak Selenium	1+20		3	50	PASS
-	-		1+20	3	43	PASS

Table 3: Results of testing films according to ANSI/NAPM IT9.15. In column no. 2 Kodak Selenium is identical with Kodak Rapid Selenium Toner. In column no. 4 KHCA is identical with Kodak Hypo Clearing Agent. Column no. 6 reports the average percentage of metallic silver converted to silver salts, as measured with the acid dichromate test, while the pass/fail criteria in column no. 7 is less than 0,05 density change after incubation.