## Kallitype

### Lesson 1: Introduction

Kallitype is a silver-iron process. The light-sensitive substance is - as in platinotype - iron III oxalate. Platinum and palladium are not necessarily suitable for entry into the alternative processes because of the constantly rising costs.

The silver processes such as salt printing, Vandyke and Kallitypie are less cost-intensive and are recommended at least for initial experience.

Kallitype is a development process, so a special developer is needed.

Toning with precious metals such as gold, platinum or palladium is possible. Especially with gold toning, the maximum blackening increases significantly. The toning possibilities are manifold and will be presented in detail later.

### Safety advice

Iron II oxalate is toxic!
Silver nitrate is corrosive!
Skin contact with both solutions should be avoided!

### C**oating**

Silver nitrate 10% and ferric oxalate 20% are mixed in equal parts and applied to a suitable paper with a brush or glass rod in dim artificial light. After coating, the paper should be left to rest for a few minutes so that the solution can be fully absorbed. Only when the coating appears matt can it be dried with warm air (drying cabinet or hair dryer) at a moderate temperature (30-40°C). At room temperature, drying takes about 20 minutes.

For more heavily sized papers, Tween can be added to the sensitising solution to ensure an even, stain-free application. The amount needed should be determined by trial and error, usually one drop (10%) of Tween 20TM to 2-4ml of sensitising solution is sufficient. With the papers I prefer to use ( Arches Platinum, Bergger COT, Hahnemühle Platinum Rag) it is not necessary to add a wetting agent.

Only as much emulsion should be prepared as can be processed within 10-15 minutes, because silver can precipitate if left to stand for longer. Silver precipitations in scales and cups can be dissolved with a fixing bath, but this should not be allowed to happen. Immediately after use, wipe out vessels with paper towels and clean brushes with demineralised water, squeezing the brushes several times in paper towels (e.g. Zewa or Kokett). To avoid skin contact with the chemicals, laboratory gloves should be worn. The use of tap water must be avoided under any circumstances!

The coated papers do not have an unlimited shelf life. Only as much should be coated as can be used within two to three days.

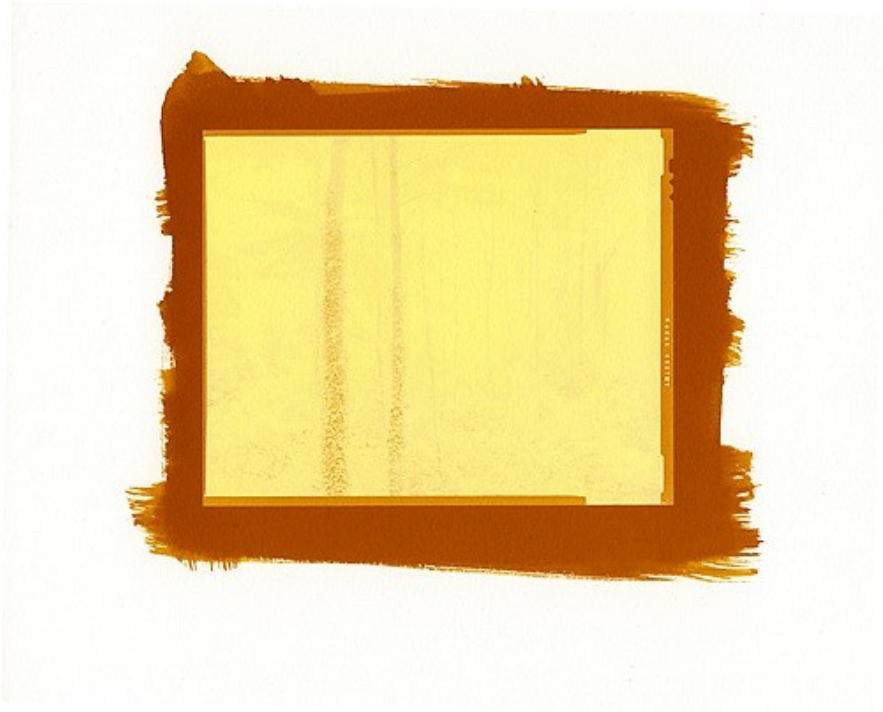
### The print negative

Original negatives developed in "staining" developers can be used because the stain acts as an additional density when exposed to UV light. All other negatives must be recopied to a contrast range of 1.2 to 1.5 logD. Because the Kallitype is a contact copying process, the negative must be produced in the desired print format. In the analogue chemical process, a slide is taken from the original, which is then enlarged onto a film with the required contrast range. Digitally produced negatives are suitable if the printing film and ink allow the required high coverage.

### E**xposure**

Exposure is done with daylight or, for better reproducibility, with UV light. Facial tanners or, in the case of larger formats, décolleté tanners are perfectly adequate. Exposure times are between three and twenty minutes, depending on the distance between light source and negative paper sandwich and the density of the negative.

When using a contact copy frame with a split pressure plate, the progress of the exposure can be checked. Correct exposure is when the deep shadows have taken on a delicate reddish tone. The mid-tones must not yet be visible!



**Development**

I recommend a 20% solution of sodium citrate or sodium acetate 10% as developer. There are many different developer substances mentioned in the literature, and they all work, sometimes more, sometimes less satisfactorily. A problem for the user of the technique often seems to be the fading of the copies before completion, at the latest during fixing. The processing chain described here should therefore be strictly adhered to.

The print is dipped quickly into the developer with the layer side up.

It is extremely important that the entire image is immediately and evenly covered with developer, otherwise streaks may form. The image appears abrupt, very dark and somewhat mulish. The print should be left in the developer for three to four minutes, it can be turned on its face to facilitate washing off the dissolved substances after some time; the tray should be tipped occasionally for this purpose. After the development time has elapsed, the print appears much brighter and richer in contrast than at the beginning.

**Clearing bath**

After development, the print is immediately clarified in a citric acid bath without a rinse in tap water (!). Before fixing, any contact with tap water should be avoided, otherwise the pH-value and the salts contained in the water could cause the formation of hardly soluble iron compounds (yellowing). For this reason, I strongly recommend preparing the clearing bath with demineralised water. A citric acid solution of 2 - 4% is best suited to dissolve the iron salts from the paper. The clearing time is three to four minutes, and the tray should be moved constantly, at least at the beginning. The image will now become brighter, but this will also change again in the course of the process.

This is how the print (developed in sodium citrate) must look before toning or fixing: Too bright, with yellow or orange highlights and reddish shadows. When developed in sodium acetate, the image appears darker and more neutral in tone.

**Toning**

Gold, platinum or palladium toning, or even combinations of these precious metal tonings are done before fixing!
Only the gold toner can also be used after the fixer.

Toning is done for the following reasons:
Increased durability - gold and platinum are more stable than silver.
Increasing the contrast range - the shadow densities increase.
Change in image tone - e.g. platinum neutral, or gold magenta to blue.

Less loss of density due to fading in the fixing bath is often cited as a reason for precious metal toning. In my opinion, this argument is only valid if unsuitable fixing baths are used.

**Fixing bath**

In the literature, weak sodium thiosulphate baths are recommended, sometimes even combined with extremely short times of about 15 seconds. Only silver salts that are not involved in image formation should be dissolved during fixation. Metallic silver must not be so aggressively attacked by thiosulphate that it is simultaneously dissolved with excess silver salt. However, this seems to happen even in some special fixing baths for Kallitype when the untoned print is "fixed". So if this fading were in the nature of the process, a more stable image body would have to be created by replacing the metal before fixation. This is not necessarily the case, even though it is repeatedly described in publications.

Quick fix baths based on ammonium thiosulphate should therefore theoretically be ruled out for this application from the outset. In my opinion, however, a slightly acidic ammonium thiosulphate bath is the best choice for Kallitypie. I use my ATS fixer in dilution 1+10 and cannot detect any fading with three to four minutes fixing time. On the contrary, there is a strong increase in density as soon as the untoned print is introduced into the fixing bath.

**Wash aid**

A wash aid is not absolutely necessary, but it shortens the washing time of Kallitypes on extra-heavy layers like Arches Platinum to about 15 minutes. Sodium sulphite as a 1% solution, freshly prepared before use, is quite sufficient for this purpose. The stock solution of my (or Kodak) wash aid can be diluted 1+10 to 1+20 for the same effectiveness.

**Drying**

The prints can be laid out on drying frames at room temperature and then they dry completely smooth. In case of hanging quick drying in the drying cabinet, all papers, the thinner ones more than the heavier qualities, will curl and have to be smoothed out at low heat (mounting press 50-60°C), or under a weight on a flat solid wooden board or glass plate.

This is what the untoned Kallitypie looks like after fixing and drying. The density has increased enormously compared to the wet (unfixed!) print, the previously shrill yellow tone has become a pleasant warm reddish brown. Toning is therefore only necessary if a different image tone is desired or if the print has to last a few decades longer.

For comparison, a gold toned print before fixing. Although the exposure time was 10% shorter, the maximum blackening is higher. The highlight density is lower, the image appears richer in contrast.

**Lesson 2: From Mixing to Development**

Mixing and application of the sensitising solution

Iron III oxalate and silver nitrate are mixed in equal parts in a small beaker. Make sure that the container is dry. Even small amounts of tap water can cause reactions with the silver nitrate. If the solution becomes milky very fast, it is unusable. Four to five drops of both partial solutions are sufficient for an 8x10inch coating (with glass rod).

Not all papers that produce good results in platinum printing are equally suitable for Kallitypes. The process stands and falls with the quality of the printing paper used. I almost exclusively use Arches Platinum, Bergger COT and, since its release, Hahnemühle Platinum Rag. The cotton board of these three brands is about 300g, which makes handling easier and the finished print is not only pleasing to the eye but also to the hand. These papers have the "right" sizing for all noble printing processes, the coating does not float off, iron salts are quickly dissolved in the clearing bath, the long-fibre cotton facilitates allow a wash out of the chemicals quite fast, there is no fear of fading during the individual processing steps.

It should be noted that when switching between the papers mentioned, both exposure times and contrast may change slightly. Identical toning processes can also lead to divergent results in colour saturation.

**Brush or glass rod?**
When applying with a brush, the layer is thicker and the print shows more strength. When applying with a glass rod, the layer is more homogeneous, thinner, the resolution is higher, the impression of sharpness too.

Brush:
The marked area should be coatet as quick as possible. The brush is held loosely, and the solution is spread thickly with criss-cross strokes without exerting much pressure. This is a critical phase for inexperienced users. Different densities of the application or even visible brush strokes can only be evened out as long as the layer is still wet, but as soon as it starts to dry, further handling with the still wet application brush is often unsuccessful. In such cases, especially if the application quantity was a little too high, it helps to have a dry spreader brush at hand, with which the layer can be slightly homogenised if necessary. To do this, let the brush dance on the layer with its tips only, whether with short strokes or circular movements, it doesn't matter, it just has to be quick. Any pressure should be avoided, because this does not spread the emulsion, but only pushes it back and forth. With strongly structured motifs, brush strokes can remain inconspicuous; with homogeneous surfaces, every mistake jumps directly into the eye.

The print shows (upper right edge) that not homogenising with the brush does not help. If small puddles form at the edge where the brush was placed, they are more noticeable after drying than before. When still wet, however, such irregularities are easy to recognise when viewed against the light.

Glass rod:

The glass rod is placed on the paper, a dropper pipette is used to apply the sensitizer to the required length on the rod. Tear-offs of the applied solution are normal. If there is enough solution, it can be brought back to the intended length by pressing on the rod, then pull the rod downwards, place it again behind the emulsion sausage that has been pushed downwards and pull it upwards again to the attachment point with lighter pressure. The emulsion should be applied evenly in two to a maximum of four strokes. Any excess solution at the ends is picked up with kitchen paper. If left to stand, it may be absorbed into the area where the negative is placed.

Faulty application, only visible at the edges, but still unsightly.

**Masking**

If the picture is to stand free without brush strokes, the negative must be masked.

It is quite easy to mask the negative with a red or better black tape or to create a mask with black foil, black cardboard or blackened RC paper. However, with this method, the contact pressure of a glass plate is not sufficient to ensure intimate contact between the negative and the printing paper, resulting in partial blurring. Only vacuum frames pull the sandwich cleanly onto the paper. If printing is to be done from a digitally created print negative, a deep black border can be added during image processing.

Masking is helpful for first attempts to assess the clearing, but it is also sufficient to cover a small corner of the layer during exposure. The light-sensitive substance (here iron III oxalate) must be dissolved as completely as possible to ensure long-term durability. As long as the unexposed edge has a yellowish tone, there is still iron salt in the paper. At the beginning of development, the emulsion edge is still very clearly visible. The reason for a development time of four minutes or more is not an increase in density, but an extensive dissolution of the iron salt. But only in the citric acid clearing bath does the yellow (iron) disappear completely, the excess silver is dissolved in the fixing bath.

A yellow edge indicates iron salt residues. If the clearing is not completed after four to six minutes, the paper is unsuitable for this technique. A grey border is caused by too bright room light when positioning the print negative, or when using translucent masking material, or also when using overused developer (more on this later).

This is how the support must look after processing. Ideally, there is no difference between the paper white and the unexposed edge.

The line at the top left is a pencil mark made at all four corners to mark the area for application of the sensitising solution. For exempt prints, these marks can be easily erased if a soft pencil has been used.

**Freestanding picture without brush strokes**

If only a narrow black border is desired without showing the application by brush or glass rod, the negative is positioned in a black mask.

A blurred edge occurs when the mask is two to three millimetres above the negative. Due to the refraction of light at the edges of the mask, especially with the thick glass negatives, a slight tone may appear on the entire application surface, but this can be removed
after fixation with toner (better bleach and dissolve in fixer). To do this, dilute the bleach about 1+20 and apply it with cotton wool. If you let the print drip off beforehand, the short exposure time of 20-30 seconds prevents the small amount of bleach from being absorbed into the image and spoiling the print. I always apply bleach to one long and one short side of the print. The print lies on a flat surface (e.g. a partition from the washer), which is tilted so that the solution runs down when the application is complete.

Photo: Albert Renger-Patzsch,  glass plate

Toning: MT6 Nelson Gold after fixing

**Contrast control**

If the contrast range of a negative is too low, the print will only appear flat, or too dark and undifferentiated with ample exposure. In such cases, the developer can be made more contrasty by adding dichromate.

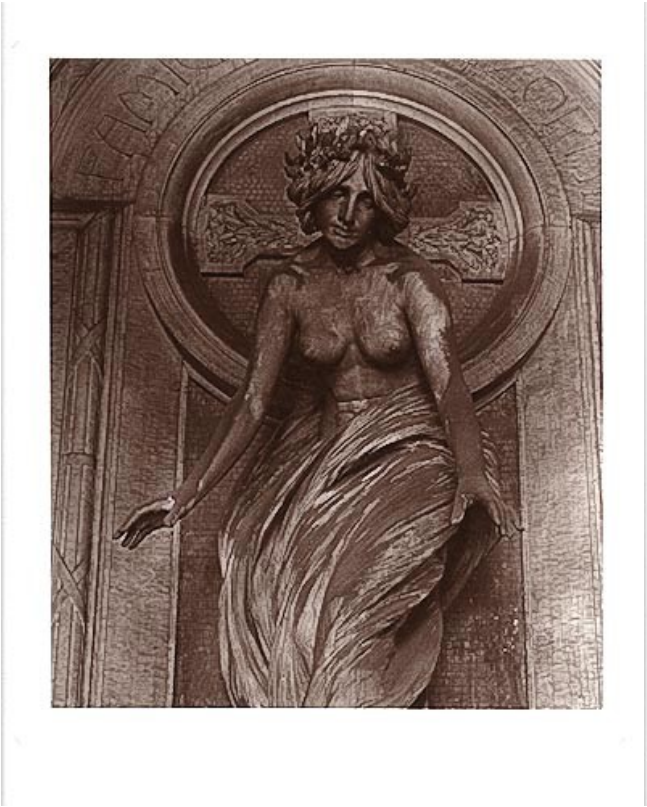
The following negative was made for gum printing, so it is delicate, without high density. At a lamp distance of 12cm (UV tanner) the sample was exposed for two minutes and divided before development. The left part was developed in sodium citrate without addition, the right part in the same developer with addition of 40 drops of sodium dichromate (50% solution) to one litre. It is clear that especially the highlights and mid tones come out brighter. The deep shadows remain unaffected with this amount of chromate if the exposure time is increased accordingly. In the example below, the exposure is correct for the developer with chromate. For the developer without chromate, the exposure time would have to be reduced by about 50%. Potassium dichromate can also be used instead of Sodium dichromate. Sodium dichromate has the advantage of being more soluble, which makes it possible to dose drop by drop. For normal negatives with a copy range of 0.9 to 1.3 logD, 2 to 12 drops are needed. If the contrast range is higher than 1.4 logD, no chromate is needed to increase contrast, but then the effect as an anti-fogging agent (pure image whites) is also lost.

Even if a densitometer is available to control densities, negatives may turn out too thin. While an optimal negative for the process should be aimed for, it makes more sense to control contrast with chromate in the developer than to re-enlarge. If negatives with different contrast ranges are to be processed during a print session, it is advantageous to have two to four developers available with, for example, 2, 4, 8, 12 drops of chromate per litre. If you work with digital negatives, you can quickly reprint a negative that has been adjusted in density and contrast. This eliminates the need to increase contrast with chromates.

Photo: Matthias Stalter
without toning

Even polaroid negatives can be used for Kallitypes in this way, if they are not too thin. If the negative (and not the positive) is exposed at optimum density, two to three drops of dichromate per 100 ml of citrate developer are sufficient.

If a (untoned) Kallitype is too dark due to overexposure, it can be lightened with Farmer's reducer like any other silver print. This reducer contains Potassium ferricyanide and Thiosulfate. A better choice is to use the devidet solutions of a Ferricyanide bleach with a bromide and a fixer.



To be able to control the degree of reducing, bleach in weak hexacyanoferrate solution. For this application I diluted my bleach concentrate 1+100 and bleached by sight. After twenty seconds, I quickly washed and fixed. A fixing time of 20 seconds is now quite sufficient.
This bleached down print does not differ in any way from a properly exposed print in terms of colour tone and maximum blackening.
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If you want to print from a negative where the highlights are too dense caused by overdevelopment and the shadows are too thin, there is no choice. Exposure must be made on the highlights, with the result of a print that looks too heavy with undifferentiated shades.

Photo: Albert Renger-Patzsch

In this case, a simple bleaching does not achieve the goal because the highlights would fade until the shadows are reached by the bleach. Bleach the highlights and upper midtones of the completely washed-out print and protect them with a thiourea toner. After drying, the print will be much lighter than before.

With sulphur toning after strong bleaching, the image tone always changes to a yellowish brown. Thiourea toners with low alkalinity has proven to be the most favourable toning substance.

However, if the shadows are still too heavy, bleaching is carried out again. The previously toned densities are not affected by the bleach, so bleaching only takes place in the areas of the image that contain metallic silver. The bleaching time should be measured in such a way that the deep shadows remain. The bleach should now be diluted to at least 1+500 to allow sufficient time to assess the bleaching process. Then fix, wash and dry.

**Lesson 3: Toning with precious metals**

Platinum and palladium toners are used before fixing. Gold toners, on the other hand, work before as well as after the fixer.

All other toners are to be used exclusively after fixation.

All toning methods presented here increase the life of Kallitypes. Like any other silver print, Kallitypes are sensitive to gases and exposure to light for a prolonged period of time, not more but rather less than silver gelatin prints. At least when printed on high quality cotton paper, Kallitypes can survive a few decades without damage. If you want to safeguard your works for centuries, you cannot avoid precious metal toning. When the silver is completely replaced by gold or platinum, the Kallitype is just as durable as the Platinotype, i.e. until the cotton layer decays.

The developer also plays a significant role in the toning of Kallitypes. The following examples were toned after development in sodium citrate. Examples of toning after other developers follow later.

Kallitype from pyro original negative, without toning

Palladiumt toning

Platinum toning

for comparison Carbon-toning (selenium/sulphur) after fixing

**Gold toning**

Gold toning before fixing increases the density in the shadows and slightly reduces the density in the highlights. The different formulations also produce very different image tones.

After fixing, colour differences between the various toners (when toned through!) are hardly noticeable. All toners known to me tend to a cool magenta tone after citrate developer and to a rather bluish tone after acetate developer. The intensity depends on the duration of toning. Here, too, the maximum blackening increases, but the highlight tone value remains the same, the hue becomes colder.

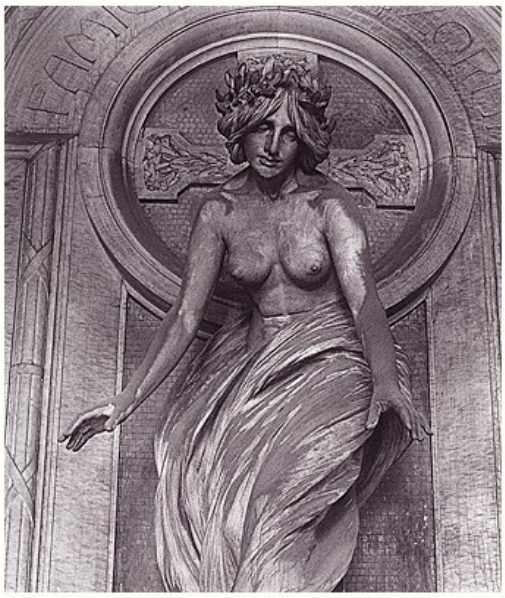
Featured Gold Toner

MT10 gold toner (thiourea/gold) high durability and high capacity, my standard toner

MT9 Gold toner (thiocyanate/gold) separate solutions, not very stable after mixing

MT6 Nelson gold toner (complex production) must be used at approx. 40°C

Horst P. Horst
Left untoned, right toned in MT9 gold toner after fixing



Toning before fixing clearly shows the differences between the gold toners: MT9 on the left, MT10 on the right, toning time four minutes each.

Albert Renger-Patzsch

MT6 Nelson Gold before fixing

MT6 Nelson Gold after fixing

**Changing the tonal range through toning**

In order to make tonal value shifts due to the common toners clear, the exposure time was not adjusted to the property of the intended toner in the following examples. All prints were exposed for two minutes. The negative has a contrast range of 1.3 logD. Developed for five minutes in sodium citrate with the addition of one drop of dichromate per 100ml, clarified for three minutes, fixed for four minutes.

Without toning

Palladium toning

Palladium and gold toning - one and a half and two minutes before fixing

Platinum toning

Gold toning (MT9) prior to fixing

**Unit 4 Papers, developers and toners**

Besides the sodium citrate developer shown above, several other developers are interesting. Of particular note is the sodium acetate developer, which, in contrast to all other formulations, produces an almost neutral image tone.

**Papers**
Arches board
Arches vellum
Bergger COT
Hahnemühle Platinum Rag
Revere Platinum
Fabriano Artistico (pre-treatment is required for most batches).
Weston Diploma Parchment
Cranes Kit White and Kit Ecru (warm)

**Developers**
Sodium citrate
Potassium citrate
Ammonium citrate
Potassium sodium tartrate (Rochelle salt developer)
Sodium acetate
Potassium oxalate (only n mixture with other developers)

**Toners**
Platinum, Palladium
Gold (presented three different formulations)
MT3 Vario (thiourea) MT5 Sepia (sulphur)
Selenium MT1 (only in high dilution, with mostly unattractive yellow tone and loss of density, recommended only in combination with other toners). MT14 Selenium/thiocyanate (cool tone, no loss of density).
MT2 Carbon
MT7 Iron Blue
MT12 Cobalt/Iron
Copper (alkaline toner, especially for callitypie)
Toning by bleaching and redevelopment, for example in Lith or SE6 Blue

Bergger COT 320 Sodium acetate developer

Hahnemühle Platinum Rag, Rochelle salt developer

Developer Sodium citrate

Diploma Parchment

MT10 Gold toner after fixing

Cranes Kit Ecru,

alkaline Copper toner
followed by MT3 E
(without bleaching)

Cranes Kit Ecru,

Sodium citrate developer

Toner MT10 Gold
1:30 minutes

Cranes Kit Ecru,

Acetate developer

Palladium toner

Hahnemühle Platinum Rag

Potassium citrate developer

Hahnemühle Platinum Rag

Ammonium citrate developer

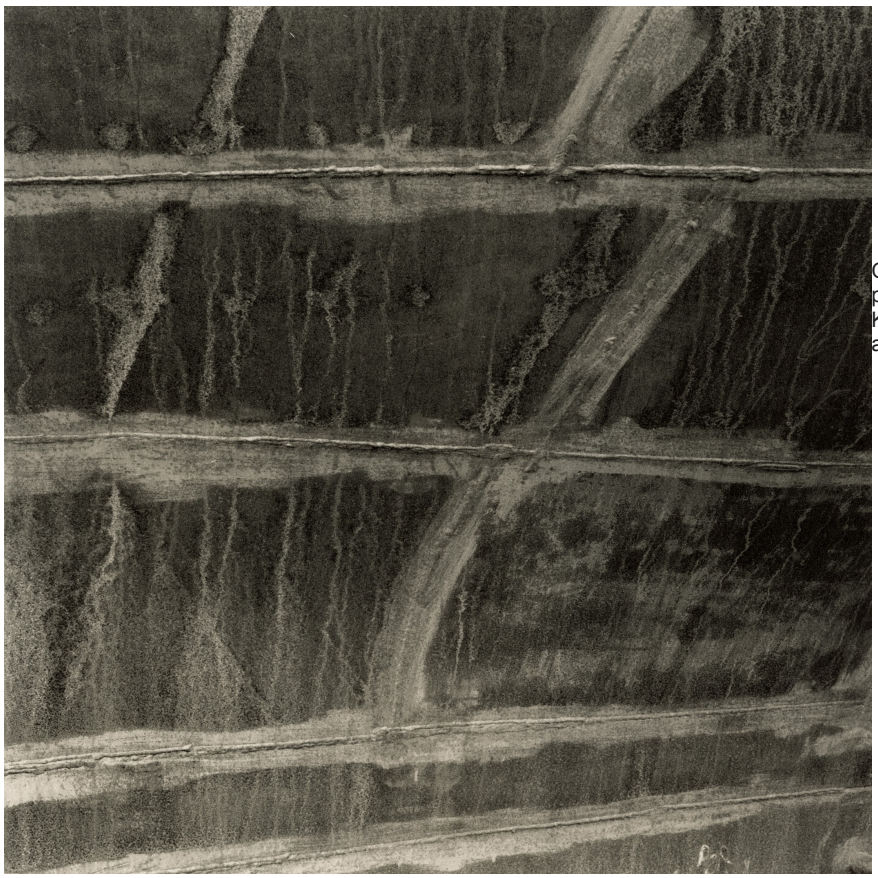
MT10 Gold toner

Bergger COT 320

Acetate developer

MT3 Vario
bleach 1+75 1 minute
toner setting A

followed by
MT10 Gold toner



Hahnemühle Platinum Rag

Oxalate developers used for
platinum/palladium are much too strong for
Kallitype, but mixtures with other developers
are possible.

Mixture of
Potassium oxalate and Sodium acetate

MT3 Vario
bleach 1+50 30 seconds
toner setting A

Hahnemühle Platinum Rag

Mixture of
Potassium oxalate and Sodium acetate

MT3 Vario
bleach 1+50 30 seconds
toner setting E

same treatment as above
but with yellowish toner setting

Hahnemühle Platinum Rag

Mixture of
Potassium oxalate and Sodium acetate
1+3

Hahnemühle Platinum Rag

Mixture of
Potassium oxalate and Sodium acetate
1+3

bleached with Lead nitrate

redeveloped in Lith
A 5ml Lith B 2ml AmCl 3ml
Omega 3ml to 600ml water
45°C 7 minutes

Bergger COT 320

Acetate developer

MT12 Cobalt toner
followed by
MT7 Iron Blue Toner

Hahnemühle Platinun Rag

Rochelle salt developer

MT10 Gold toner 1 minute

Arches Platine

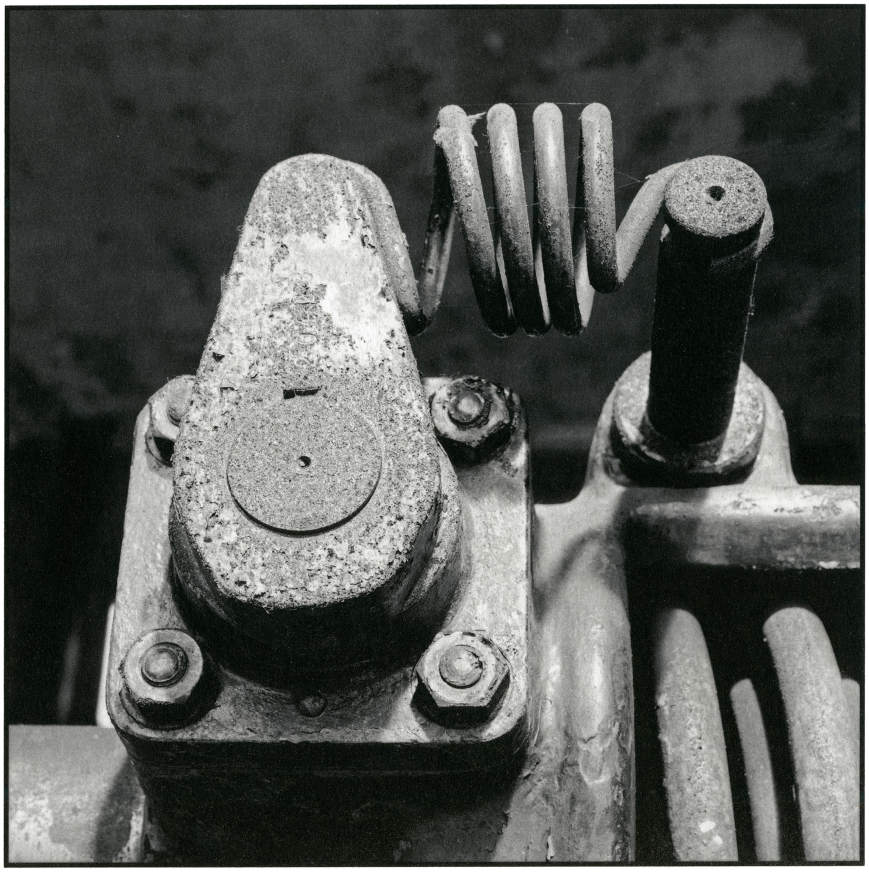
Acetate developer

3 toners

1st MT3 Vario
bleach1+50 50 seconds
toner setting C

2nd MT10 Goldt oner 75 seconds

3rd MT7 iron Blue Toner
5+5+10+5+800ml 40 seconds



Hahnemühle Platinum Rag

Sodium acetate developer

MT10 Gold toner

Hahnemühle Platinum Rag

Mixture of
Potassium oxalate and Sodium acetate
1+3

MT3 Vario toner
bleach1+100 30 seconds
toner setting C

Arches Platine

Mixture of
Potassium oxalate and Sodium acetate
1+3

MT3 Vario toner
bleach1+100 30 seconds
toner setting C

Hahnemühle Platinum Rag

pre-coated with Silica
in order to achieve a deeper black

Acetate developer

Toner MT3 and MT10

MT3 Vario
bleach 1+75 50 seconds
toner setting E

MT10 Gold toner 4 minutes

Bergger COT 320

Sodium citrate developer

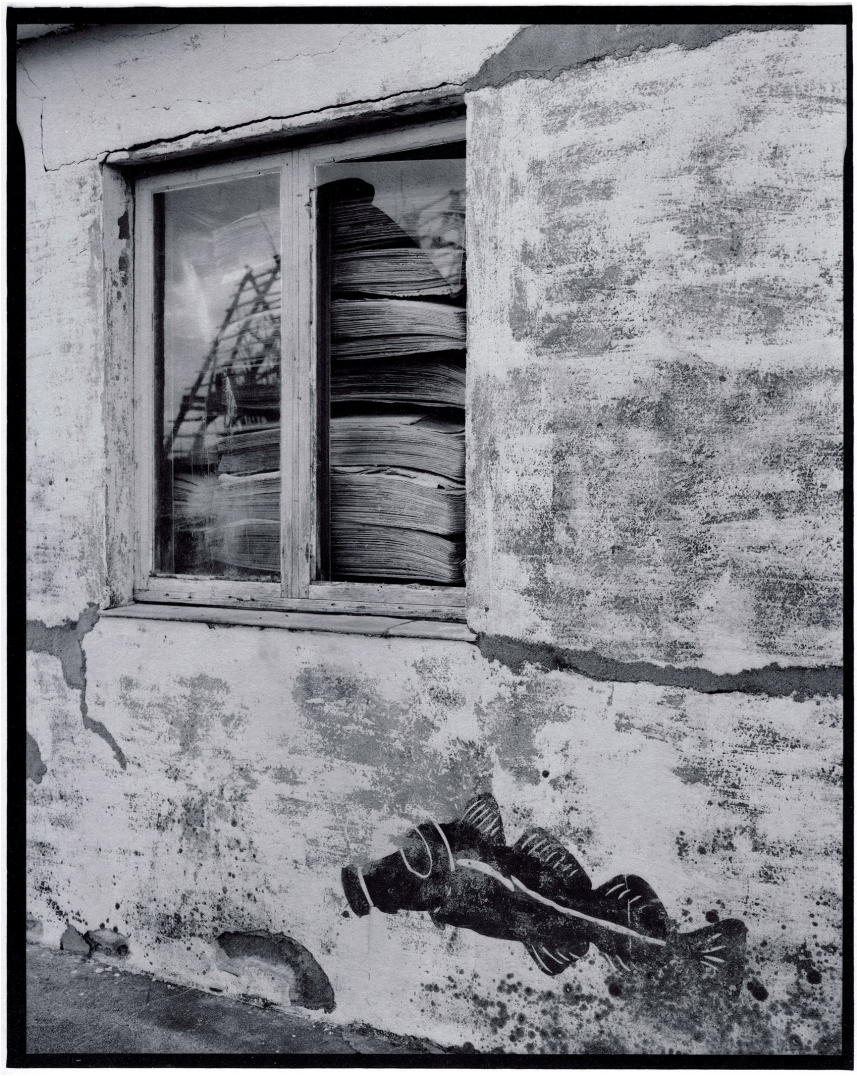
MT3 Vario toner
bleach 1+100 50 seconds
toner setting D

Magnani Revere Platinum, Sodium acetate developer

Platinum toner1:30 minutes, followed by alkaline Copper toner10 seconds

Alkaline Copper toner followed by MT7 Iron Blue Toner

MT3 Vario bleach 1+75 30 secs setting E, MT10, Gold toner 4 Minuten



Markus Rottländer

printed from a 8x10 Pyro negative

Magnani Revere Platinum,

Sodium acetate developer

MT10 Gold toner 1 min prior to fixing

Arches Platine

Sodium acetate developer

MT12 Cobalt-Iron toner

Hahnemühle Platinum Rag

Acetate developer

MT12 Cobalt
16-7+7+7+1000ml 1:40 minutes

followed by MT10 Gold toner 5 minutes

Arches Platine

Acetate developer

MT12 Cobalt
25+10+10+10+1200ml 70 seconds

followed by MT10 Gold toner 6 minutes

Bergger COT 320

Acetate developer

MT14 Selenium/Tthiocyanate

MT7 Iron Blue Toner

Bergger COT 320

1 drops Na2  5% to 2ml sensitising solution

Sodium acetate developer

no toning

Hahnemühle Platinum Rag, one drop Na2  5% to 2ml sensitising solution, Acetate developer, no toning

Bergger COT 320, Ammoniumcitrate developer

Kozo, Sodium acetate developer, MT3 Vario toner: bleach 1+50 30 seconds, toner setting  C

Kozo

Sodium citrate developer

MT1 Selenium toner 1+200 1 minute

Arches Velin

Sodium citrate developer

MT2 Carbon toner
bleach 1+200 90 seconds
toner 1+100 2 minutes

Fabriano Artistico

NatriumcitratentwicklerSodium citrate developer

Hahnemühle Platinum Rag

Acetate developer

Lead nitrate bleach

Easy Lith A+B+Omega+water
10+8+4+1400ml 52°C 4 minutes

Bergger COT 320

Acetate developer

MT10 Gold toner 2 minutes

Bergger COT 320

Acetate developer

alkaline Copper toner,
followed by MT3 A
(without bleaching)

Bergger COT 320

Acetate developer

alkaline Copper toner,
followed by MT3 C
(without bleaching)

Bergger COT 320

Ammoniumcitrate developer

MT10 Gold toner
prior to fixing 6 minutes

Arches Platine

Acetate developer

pre-toning in Palladium 20 seconds

and after fixing
alkaline Copper toner 1:30 minutes

Arches Platine

Acetate developer

alkaline Copper toner 1 minute

followed by Iron Blue Toner
3+3+6+3+250ml 1 minute
Lead acetate - toner 1,5% 25 seconds

Printnegativ von Lithprint

Bergger COT 320

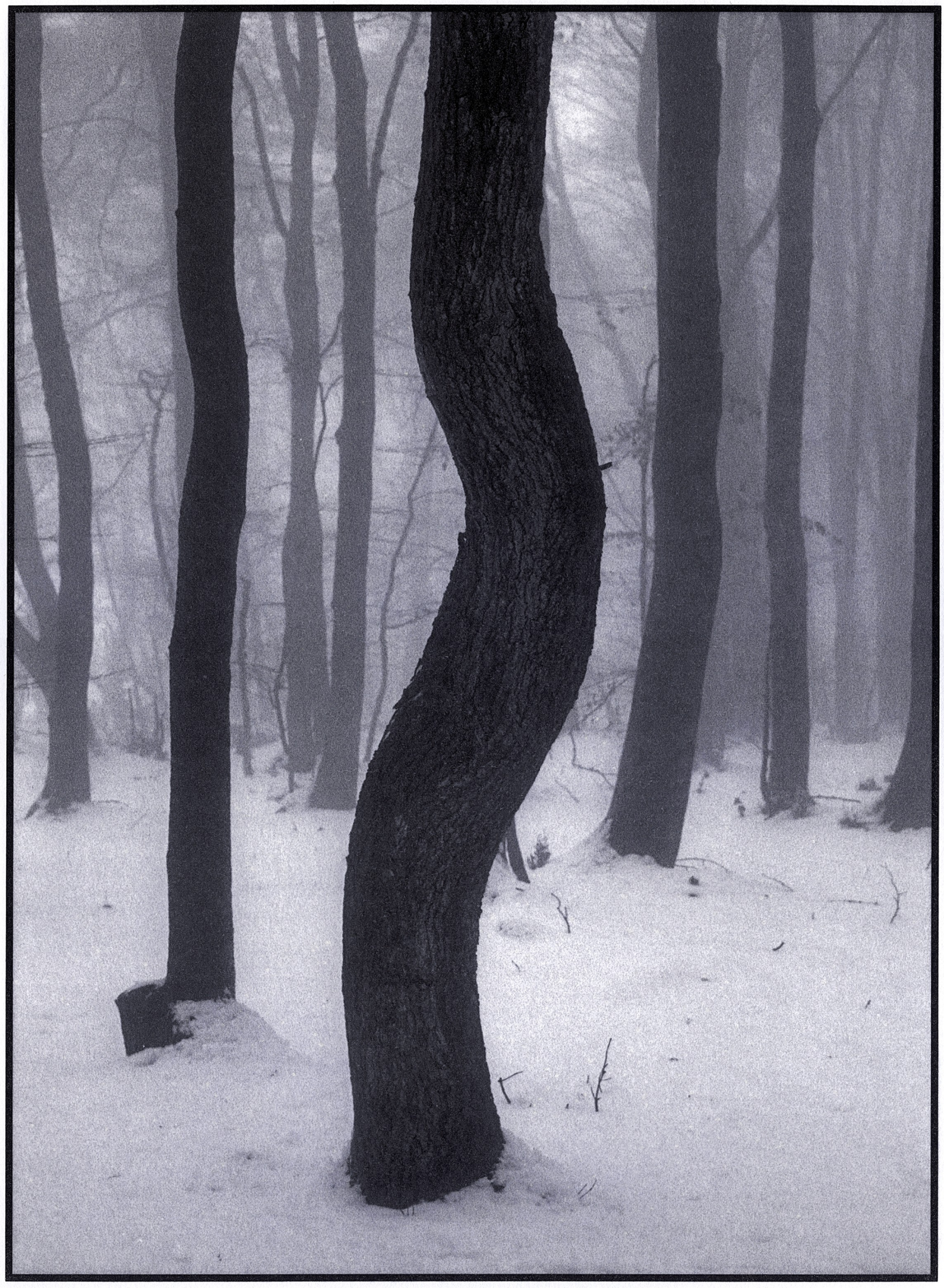
Acetate developer

alkaline Copper toner
followed by MT3 C

Kozo

Acetate developer

MT3 Vario
bleach1+60 1:15 mins
toner setting D



Hahnemühle Platinum Rag

Acetate developer

MT7 Iron Blue Toner 2+2+5+2+500ml 30 seconds, followed by Lead acetate 3% 20 seconds