

Chapter 14 - Testing

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Testing the Ferric Oxalate

updated December 2000

This test is comprised of three tests and two functions. The accomplishment of both functions is critical to the passing of the Ferric Oxalate solution. A solution should be made up containing only Ferric Oxalate without any additives so as to not introduce other possibilities. Later actual sensitizer solutions can be tested to determine if any additives adversely affect the function of the Ferric Oxalate.

Note: Testing, as with any handling of Ferric Oxalate, should be carried out in safelight illumination.

In order to pass this test, the Ferric Oxalate solution must first NOT turn color. This demonstrates a desired lack of ferrous material that may be present from contamination or exposure fogging. Second, the solution must turn color to indicate the proper conversion to ferrous when called for.

To test the Ferric Oxalate solution:

PART I - TEST (#1, #2 or #3)

- ✓ Place some Ferric Oxalate solution into a shot glass.
- ✓ Add a crystal of Potassium Ferricyanide (one or two crystals is plenty to do the job).
 - ★ Nothing should happen. Test Part I PASSES if nothing happens.
 - ★ TEST FAILS if the solution darkens (do not consider any natural orange or red color from the Potassium Ferricyanide, the failure color is likely to be blue, gray, or indigo). The solution likely contains Ferrous Oxalate.

TEST #1

TEST #1 FAILS:

- ▶ If TEST #1, FAILS and the solution is OLD SOLUTION, the solution is defective. This can be caused from heat, exposure to light or old age.
 - ✓ Make a fresh solution from the stock powder and test again (TEST #2).
- ▶ If TEST #1, FAILS and is NEW SOLUTION, then the stock powder is defective.
 - ▶ If the defective stock powder is OLD POWDER, then it has probably been stored incorrectly. Heat, moisture or light exposure can cause a powder to go bad.
 - ▶ If the defective stock powder is NEW POWDER, then the powder should be returned to the vender for replacement.

TEST #1 PASSES:

- ★ If TEST #1 PASSES, continue test PART II with the same solution.

TEST #2

TEST #2 FAILS:

- ▶ If TEST #2 FAILS and the stock powder is OLD POWDER, then it has probably been stored incorrectly. Heat, moisture or light exposure can hurt a powder.
 - ✓ Make a new solution using new stock powder and test again (TEST #3).
- ▶ If TEST #2 FAILS and the stock powder is NEW POWDER, then the powder is defective and should be returned to the vender for replacement.

TEST #2 PASSES:

- ★ If TEST #2 PASSES, continue test PART II with the same new solution.

TEST #3

TEST #3 FAILS:

- ▶ If TEST #3 FAILS, then the new powder is defective and should be returned to the vender for replacement.

TEST #3 PASSES:

- ▶ If TEST #3 PASSES, continue test PART II with the same new solution from the new stock powder.

PART II - TEST

- ✓ PART II Place shot glass with Solution in UV light.
 - ★ The solution should turn deep indigo blue.
 - ★ If the solution does not turn a deep indigo blue, then the Ferric Oxalate is bad.
 - ▶ If this is a NEW SOLUTION from a NEW POWDER, first try adding some more Potassium Ferricyanide. If there is still a failure to turn blue and the Potassium Ferricyanide is good, then the powder is not Ferric Oxalate. Return the powder to the vender for replacement. (If this happens again, find a new vender.)
 - ▶ If this is a NEW SOLUTION from an OLD POWDER (that has worked before), then somehow the solution was made incorrectly. Make a new solution from the old powder and re-test.
 - ▶ If this is an OLD SOLUTION from an OLD POWDER (that have both

worked before), then the Potassium Ferricyanide must be bad. (This is unlikely , but there is no other reason.)

- ★ When the solution turns dark blue only after exposed to light, then the TEST has PASSED and the Ferric Oxalate is OK to use in a sensitizer solution.

Fogging Test

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It is important to work with light sensitive materials in conditions which do not adversely affect those materials. Of the most common is fogging from stray light or an inappropriately chosen work light.

Testing for fogging is straight forward and should be performed whenever a new work light or new material is put into use. It is especially important to have conditions which have passed the fogging test when running a clearing test since fogging could be mistaken for incomplete clearing.

Before having an opportunity to write up a testing procedure for fogging, a great procedure was mentioned by a fellow Pt/Pd printmaker. The following fogging test description and explanation is presented with permission from its author, Tom Ferguson.

Tom's web site is at <http://www.thefstop.com/tf.html>

I'm a big fan of people doing tests themselves, rather than simply taking other peoples "word". One learns soooo much more that way! There are simply too many variables in this world. Does my light bulb enclosure block more UV than yours?? Are South American light bulbs different in output than USA bulbs?? Is a Philips 75W bulb different than a GE?? So I do suggest that anyone coating paper do a simple fog test.

Coat a piece of paper (in the dark) and find the exposure without a negative (in your usual UV light source) that gives the first barely visible gray in a processed and dried print. Next coat another piece (in the dark) and give it this same exposure, but don't process it. Now cut this paper into 3 pieces. Put a large heavy coin on each piece of paper and leave it in your room with the desired lights on. Label and remove one after 15 minutes, another after 30 minutes, the last after an hour.

Process and dry the test prints, and see if the coin's shadow shows as a lighter tone than the rest of the test print.

Why go to the trouble of pre exposing to "first barely visible gray"? I know, a few books don't bother with this step! Most, if not all, light sensitive materials have a "threshold" that must be passed before they start to react. What follows will not be correct to a knowledgeable scientist, but the ideas are sound.

It takes some number of photons hitting the emulsion to turn it on. Say that number is 10. If your safe light gives 9, then you don't see fogging. But then you put it, with your negative, into the UV and expose. That highlight you wanted as paperbase gets 5 photons threw the negative, It has now gotten 14 photons, and prints as a slight gray, rather than paperbase. Opps, fogging where you didn't think it would be. If we pre expose the test paper to a "first barely visible gray", then any additional photons affecting the paper will be visible.

Test for Clearing

updated September 1999, December 2000

Clearing times may be ascertained by this test. It is recommended that this test be performed every time a new paper or coating chemistry is to be tried. Also use this test before a new type of clearing agent is used. It is highly recommended to first perform the [Fogging Test](#) as fogging could be mistaken for a lack of clearing.

Notes: Safelight illumination must be used for all coating and processing during this test. Only turn on other lights after paper is in the wash water.

This clearing test procedure has been updated in September 1999 with the addition of the use of an indicator of Potassium Ferricyanide after an exposure after the clearing procedure under evaluation. This indicator was designed and suggested by John Melanson.

Caution: This indicator is extremely sensitive and may produce a false reading. It is suggested that an uncoated control be used as a reference. The solution strength of the indicator can be reduced to 1%.

The addition of this indicator has made this test much more sensitive to detecting an incomplete clearing. John Melanson has demonstrated that several clearing solutions which had removed all traces of yellow and gray and appeared cleared, showed blue or gray values with this indicator.

PROCEDURE:

- ✓ Place a strip of removable tape on the selected paper.
Note: Removable tape is handy for this, but use caution to not damage or roughen the paper surface when removing.
- ✓ Coat the paper with a very definite edge to the coating formed by the tape. Do not let the coating mixture puddle.
- ✓ Remove the tape.
- ✓ Dry carefully so as not to push the mixture into a puddle at the edge.
Note: Puddling may cause a density at the edge of the puddle that typical clearing may not remove as quickly.
- ✓ Mark the edge with a dotted pencil line(s). The purpose is to locate the position of the edge after clearing.
- ✓ Without any exposure, process the paper using the clearing procedure under evaluation, including any other processing typically given including development.

- ✓ Dry (hair dryer with heat may use used).
- ✓ Evaluation is made at this point under white light.
Note: Direct sunlight may be too intense to evaluate. Shaded sunlight is OK.

- ✓ Look very closely along the edge delineated by the pencil line(s) for any differences.
 - ★ Insufficient clearing will be seen as yellow, brown, or gray density in the coated area.
 - Yellow or brown may indicate a presence of unreduced ferric oxalate or metal salts.
 - Gray may indicate residual metal salts.
 - Examples of colors can be found in the Clearing Study.

- ★ Even the slightest difference will indicate incomplete clearing. If not cleared, the indicator portion of the test can be omitted. If there is no difference between the coated and uncoated areas, continue with the indicator portion of the test.

INDICATOR PORTION OF TEST:

- ✓ Expose with UV light source for twice the typical exposure.

- ✓ Place drops of 1% solution of Potassium Ferricyanide $K_3Fe(CN)_6$ onto a clean Q-tip or similar applicator, then spread onto the coating so as to straddle the edge of the coating, moving only from uncoated to coated area.
Note: A puddle of a drop directly applied may allow activated indicator to move across the edge.

- ✓ Wash for about 5-10 minutes to remove any yellow-orange base color of the Potassium Ferricyanide.

- ✓ Dry (hair dryer with heat may use used).

- ✓ Evaluation is made under white light.
Note: Direct sunlight may be too intense to evaluate. Shaded sunlight is OK.

- ✓ Look very closely along the edge delineated by the pencil line(s) for any differences.
 - ★ Insufficient clearing will be seen as blue or gray density in the coated area.
 - Blue indicates a presence of ferrous reduced from ferric oxalate by the light.
 - Gray is thought to be from residual metal salts.
 - Examples of colors can be found in the Clearing Study.

- ★ If there is no difference between the coated and uncoated areas, then clearing was successful. Even the slightest difference will indicate incomplete clearing.

It is best to determine and use the least amount of time necessary to clear a paper. However do remember that if the strength of the clearing agent is increased too high, then the paper may be adversely affected. It is also suspected that weak acid clearing baths work best.