# Making, Coating and Processing a Simple Gelatin Emulsion

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[This piece was written as a chapter for <u>The Book of Alternative Processes</u>, 2nd Edition, Christopher James, 2008. This particular document includes my suggestions regarding editing. Because of this it includes some material not included in the book. There are photographs of some emulsion related equipment at the bottom of this document.]

IMPORTANT: Text printed in blue is considered optional. These concepts may be removed due to limited space without seriously changing content and flow of this chapter.

# **Some History**

By the 1870s, there were three major variants of the wet-plate collodion process: preserved (moist) collodion, dry collodion and collodion emulsion. Unlike the former two, collodion emulsion relied on actually adding silver nitrate to the halides in the collodion binder before it was applied to the plate. The basic concept that evolved from making collodion-based emulsions was the beginning of emulsion photographic technology for the next 150 years.

The use of gelatin as a photographic binder was suggested by several people but the first experiments with making silver halide emulsions with gelatin were conducted as early as 1853 by Marc Gaudin. His emulsion, which he called *Photogene*, was based on the combination of iodide and silver and was not successful. It was not until January 17<sup>th</sup>, 1868 when W.H. Harrison published the first serious article on gelatin dry plates in the <u>British Journal of Photography</u>. Though imperfect, Harrison's work helped to create new interest in the possibilities of the process, particularly in England.

In 1871 Dr. Richard Leach Maddox introduced the idea that gelatin emulsions should contain silver bromide, rather than silver iodide; the basis of modern gelatin emulsions for development. Improvements, such as washing the emulsion (J. Johnson 1873) and ripening by heat to increase sensitivity (C.E. Bennett, 1874) were major contributions to the evolution of the medium. By 1877 gelatin technology was advanced enough to produce commercially manufactured plates though many professional photographers were not yet ready to make the transition from the wet plate collodion process. By 1880 gelatin emulsion plates were being manufactured on both sides of the Atlantic and being used by amateur and professional alike.

Like the daguerreotype, albumen on glass and collodion processes that preceded them, all gelatin emulsions begin as mostly sensitive only to blue, violet and ultra violet light. However, depending on the procedure, early gelatin emulsion plates could be from two to ten times more sensitive than the typical collodion negative. The sensitivity of early gelatin plates was even expressed numerically by how many times faster they were compared to a wet plate.

Making gelatin emulsions in the mid-1880s was not particularly difficult, once the basic concepts were understood. Hand coating these early plates was also very easy since they could be applied to glass plates under red light. Today, an individual can, without expensive equipment, make a similar emulsion that is perfectly suited for hand coating plates for negatives, positive transparencies, opaltypes, orotones and gelatin tintypes.

The purest gelatin used in the earliest days of dry plates was probably not so different than good quality food grade gelatin available today. It was processed from the hides, bones and hooves of cattle. As the speed and spectral sensitivity of emulsions was increased, the purity of this bovine gelatin became more of an issue and photographic grade gelatin was improved. Photographic grade gelatin was, and still is, much more pure than the food grade variant. It is probably not a coincidence that the Genesee Pure Food Company (later to be known as the Jell-O Company) was established in Le Roy, NY only a few miles from Rochester and the Eastman Kodak Company, the leading buyer of raw unprocessed gelatin.

# THE EMULSION EXPERIMENT BELOW CAN BE DELETED FROM THIS ARTICLE IF SPACE IS LIMITED.

# **Basic Theories of Emulsion Making**

It's difficult to know just how much information to include in a basic set of instructions but the following will give the beginner a good start. Though it's really a suspension, the end product of this technique has become known as an emulsion. The goal is to produce a liquid that suspends the precipitated silver halide particles and keeps them from falling to the bottom of the beaker. Before actually making a silver bromide emulsion, it's very instructive to do the following experiment with silver nitrate and common salt to understand the mechanics of the process.

# **Equipment and Materials**

- 1. safety goggles
- 2. latex gloves
- 3. two 200 ml Pyrex beakers
- 4. plastic, stainless or glass stirring rod
- 5. heat source (alcohol lamp, hot plate)
- 6. eye dropper
- 7. 2 grams silver nitrate crystals
- 8. 4 grams sodium chloride (common table salt)
- 9. 5 grams gelatin (food grade is fine for this)

[Caution: Safety goggles and latex gloves should always be worn when handling silver nitrate to prevent possible eye injury and indelible stains on skin.]

#### Part A.

In a clear glass 200 ml beaker dissolve 2 grams sodium chloride (table salt) in 100 ml distilled water. In a second beaker, dissolve 1 gram silver nitrate in 50 ml distilled water. Using an eye dropper put all of the silver solution, drop by drop into the chloride solution while stirring with a glass rod. The silver nitrate will bond with the chloride creating a precipitate of white silver chloride. This will eventually fall out of solution to the bottom of the beaker as a white paste. [Both Thomas Wedgwood and Nicephore Niepce applied this silver chloride paste to paper and exposed it to light to create printed-out photolytic silver images.]

#### Part B.

Place 5 grams photographic grade gelatin in a 200 ml beaker with 100 ml distilled water. Allow the gelatin to swell for 15 minutes. When swollen, heat this solution until the gelatin melts completely. Add to this 2 grams sodium chloride and stir until dissolved. In a second beaker, dissolve 1 gram silver nitrate in 50 ml distilled water. Using an eye dropper, put all of the silver solution, drop by drop into the chloride solution while stirring with a glass rod. This time the silver chloride precipitate will stay suspended in the gelatin mixture creating a milky white emulsion.

# Types of Gelatin

There were three basic forms of processed bovine gelatin in the late 19<sup>th</sup> century; glue, food grade and photographic grade. Glue is not suitable for photographic emulsions. It is possible to use some food grade gelatin provided that it is of reasonable purity, though photographic grade should always be a first choice.

Poor quality food grade gelatins may contain compounds that increase sensitivity of the emulsion causing non-image fogging. When using food grade gelatin, you will probably need to add more than the formula requires to achieve the same jelling characteristics as the photographic grade. This is not a problem as additional gelatin can be added at any time as required to set into a firm jelly at room temperature.

Gelatins are occasionally assigned a bloom rating, important to the setting potential of your emulsion. In the nineteenth century, gelatins were offered as simply hard or soft. If you are able to purchase gelatin with a known bloom rating use the lower bloom (soft gelatin) for the first melt and the higher 250 bloom (hard gelatin) for the reserve gelatin added after ripening. A small percentage of softer gelatin in an emulsion allows the film to be penetrated by the chemicals more easily.

# THE FOLLOWING ENTRY ON THERELATIONSHIP OF SILVER TO HALIDES MAY BE TOO TECHNICAL AND CAN BE DELETED IF NECESSARY.

# Relationship of Silver to Halides

The emulsion described herein is a silver bromo-iodide variety. The bromides are in excess to the iodides, and the total halides must be in excess to the silver. This is why the silver is always added to the bromide rather than the other way around. This excess of bromide to silver is important during the combining of the silver as it prevents potential fogging in the finished emulsion.

When looking at the formula featured on page \_\_\_\_ however, it appears that the weight of silver nitrate is actually larger than the total weight of the halides. This is because to accurately compare the ratio of silver to halides (bromide combined with iodide) one must convert the gram weights of the silver and halides to the molecular weight of those compounds being used.\* This depth of understanding is only necessary if you are formulating your own emulsion formula or substituting one halide for another.

[\*A metaphor might be that the sweetening power of a sugar substitute might be stronger than plain sugar. Equal amounts of these two sweeteners will have a very different effect. Therefore, it will take less weight of the substitute to have the same effect as sugar because of its greater sweetness.]

# **Sensitivity of Gelatin Emulsions**

The speed in which you combine the silver solution with the gelatin halide solution (called **precipitation**) has much to do with the speed, density potential and resolution potential of the final emulsion. You have several ways to control this particular variable; the percent and purity of gelatin in the gelatin halide solution, speed of agitation when combining the silver with the halide, the opening of the orifice on your syringe and how quickly the silver streams from the orifice. *The slower the silver is introduced, the larger the silver halide grain size and the more sensitive the emulsion.* 

When making your first emulsions, sensitivity is much less important than making a clean, fog free plate, with a good range of tones. A very slow, fine grained emulsion is made by literally pouring the silver solution from a beaker, into the gelatin halide solution in a continuous stream while stirring.

# Ripening and Digestion; its Effect on Gelatin Emulsions

Once the silver solution has been added to the gelatin halide solution, the sensitivity of a simple bromo-iodide emulsion can also be increased by a process called **ripening**. The hot emulsion is kept at a constant temperature to promote the growth of larger silver halide particles that are more sensitive to light than smaller particles. The hotter the temperature of the emulsion and the longer the ripening period, the larger the silver halide crystal will become. After ripening, the emulsion is chilled, washed and subjected to another heat treatment called **digestion**. This is another opportunity for the silver halides to grow by heating the emulsion for a given period of time.

Eventually heat digestion was replaced by chemical sensitization with ammonia, sulfides or other additives. It is digestion, however, that continues to increase the sensitivity *every time a gelatin emulsion is reheated*. If an emulsion is reheated too many times, or at too high a temperature, it will eventually become so sensitive that non-image fogging is the result. Reheating the emulsion also causes the gelatin to lose its setting characteristics which are absolutely crucial for successful coating and subsequent processing.

#### Washed Emulsion

When silver nitrate is mixed with a bromide and iodide, silver nitrate reacts with the halides creating a precipitate of light-sensitive silver bromo-iodide. In a process called double decomposition; a by-product of water soluble nitrates is also formed. When making developing-out emulsions with potassium bromide, the resulting water soluble potassium nitrate must be washed away. The first step in washing the emulsion is called **noodling**.

# Chilling & Noodling

The potassium nitrate is washed out of the emulsion by first allowing the hot emulsion to chill in a refrigerator to a stiff jelly. Once chilled, the jelly like emulsion is cut into shreds with a silver or stainless steel fork or squeezed through the small openings of a die or mesh to produce emulsion noodles. These noodles give the emulsion a greater surface area which aids in releasing the water soluble potassium nitrate when they are washed in very cold water. Distilled Water is safer than tap water, though most tap

water works fine. After washing the noodles they are drained of excess water and remelted leaving behind only light sensitive silver bromo-iodide in a gelatin binder.

Noodle washing was eventually replaced by dialysis and later, the application of special washing gelatins that precipitated the emulsion so that the potassium nitrate could be siphoned off the top of the emulsion. For the amateur emulsion maker, old fashioned noodle washing is still the easiest approach.

#### Making the Emulsion

The following instructions are based on a typical mid-1880s formula for making about 350 ml of a slow gelatin bromo-iodide emulsion for landscape work. You can make this emulsion more sensitive by adding the silver solution slower combined with ripening and digesting the emulsion longer; but it's better for the novice to start with slow, clean working emulsions.

# Equipment and Materials Needed:

# Equipment

Much of this equipment can be used for other historic photographic processes. The hot plate/stirrer, for example, is one of those pieces of equipment that is a great help for mixing all sorts of things. Pyrex glass beakers in assorted sizes are essential in every historic process darkroom. The crock pot and potato ricer are however specific to gelatin emulsion making, an activity very similar to cooking.

- 1. safety glasses or goggles
- 2. latex gloves
- 3. accurate gram scale (.01-25 grams)
- 4. Pyrex "tempered glass" laboratory beakers; one 500 ml & two 300 ml
- 5. 1½ quart Pyrex "tempered glass" loaf baking dish for chilling
- heat source\*
- 7. thermometer (digital thermometers are cheap and perfect for emulsion making particularly when they have a built-in alarm)
- 8. 3 quart glazed ceramic or stainless steel mixing bowl
- 9. stainless steel wire mesh drainer big enough to rest in the opening of the aforesaid mixing bowl
- 10. small brown ceramic cheese crock with wire locking ceramic lid
- 11. 1 square foot of black plastic sheeting (must be opaque)
- 12. 1 gallon bag made of black plastic sheeting (must be opaque)
- 13. large plastic syringe (60ml)\*\*
- 14. heavy duty stainless steel potato ricer
- 15. refrigerator (a small dormitory type is perfect or use your household refrigerator) and ice cube tray
- 16. electric crock pot with at least two temperature settings (for digestion)
- 17. 25" square piece of sheer white nylon or polyester cloth (for washing noodles)
- 18. 10" length strong cotton string
- 19. stainless steel spoon
- 20. glass or stainless stirring rod
- 21. deep red safe light
- 22. darkroom timer with sweep second hand

\*You can use a sauce pan on a hot plate or the crock pot (also listed) for precipitating and ripening the emulsion, but a laboratory hot plate w/magnetic stirrer is perfect for emulsion making and well worth the expense. You can purchase them second hand on line.

\*\* The Terumo brand 60 ml/cc Syringe with Catheter Tip is perfect except that the opening of the tip is too wide. Go to the hardware store and buy a tube of glue that comes with a separate tapered tip of the same size. Make a pin hole in the end of the tip with a hot needle and push this firmly over the catheter tip of the syringe.

#### Materials

The quantities listed below are for making one batch of approximately 350 ml emulsion. Naturally it is a better plan to buy larger amounts any time you buy these materials as they are generally less expensive when purchased in quantity.

- 1. 1 liter distilled water
- 2. 10.5 grams potassium bromide
- 3. 0.4 grams potassium iodide
- 4. 12 grams silver nitrate crystals
- 5. 21 grams gelatin (photographic grade is best, but you can use food grade)
- 6. 5 grams chrome alum (added as needed)
- 7. 0.065 grams thymol
- 8. 5 ml 95% grain alcohol

[Caution: Safety goggles and latex gloves should always be worn when handling silver nitrate to prevent possible eye injury and indelible stains on skin.]

#### The Procedure

Take the time to read and visualize all of the following steps before you attempt to make the emulsion. You may want to practice step 5 with plain water to feel comfortable with the technique. Try to spray 40 ml from the syringe in a continuous stream within one minute. Preparing all the materials and equipment prior to working under safelight conditions will make the procedure much easier to perform.

- 1. Put 3 grams photographic grade gelatin into a 500 ml Pyrex glass beaker with 85 ml distilled water. Allow at least fifteen minutes for the gelatin to become fully swollen and easily flattened (or squished) between the fingers. This is called the "first melt" gelatin.
- 2. Put 18 grams photographic grade gelatin into a 300 ml beaker with about 80 ml *cold* distilled water; or enough to just cover the gelatin. Allow this gelatin to absorb enough water to make it soft as tested between the fingers. This is called the "reserve" gelatin which will be drained of excess water and then added to the emulsion after the first melt.
- 3. Dissolve the swollen first-melt gelatin by placing the beaker in a hot water bath such as a small sauce pan with water on a hot plate, or in a crock pot with just enough water to the level of the gelatin solution. You may also use a hot plate stirrer as long as the solution is kept in motion with the magnetic stirrer set on slow. Using a thermometer, try to keep the temperature around 120 degrees F.

4. Put 10.5 grams potassium bromide and 0.4 grams potassium iodide in the first melt gelatin and stir the solution until the halides are fully dissolved.

[Every operation after this should be done under red safe light. The darker the safe light and the less time you have the emulsion exposed to it, the better]!

5. **Precipitation:** Prepare the silver solution by dissolving 12 grams silver nitrate in a 300 ml Pyrex glass beaker with 85 ml distilled water. Heat this silver solution to around 120 degrees F (50 C) and draw half of this solution into the syringe. Slowly squirt the heated silver solution at a rate of 40 ml per minute in a continuous stream *with the tip below the surface of the gelatin halide solution* while you stir it continuously (this is where a hot plate stirrer comes in handy). Refill the syringe and continue until all the silver solution has been added to the gelatin-halide solution. As you combine the silver with the gelatin-halide solution you will see the two clear liquids change into a milky white silver bromo-iodide emulsion.

[When making more sensitive emulsions you can be more accurate if you use a musician's metronome to keep you on track as you gently and continuously push the plunger of the syringe. Try to make the plunger pass a ml marking on every third or fourth click of the metronome. You can set your own rhythm to as slow as you want.

- 6. **Ripening:** After all the silver has been added, ripen the emulsion by maintaining the temperature at around 120 degrees F for 15 minutes with constant gentle stirring.
- 7. Adding Reserve Gelatin & Digestion: While the emulsion is ripening, begin draining all the excess water from the reserve gelatin. Too much water in the reserve gelatin can cause weak, thin images. When ripening is complete, add the reserve gelatin to the emulsion and stir until the new gelatin is completely dissolved. When you first add the reserve gelatin the temperature of the emulsion will fall. Bring the temperature of the emulsion back to 120 degrees F. and dissolve the reserve. Once the emulsion has reached 120 degrees F maintain this temperature for about 5 minutes with gentle agitation to digest.
- 8. **Chilling:** Pour the hot emulsion into the glass Pyrex loaf pan and carefully slide this into the black plastic bag. Secure the opening of the bag so that no light can fog the emulsion. Place the bagged emulsion in the refrigerator for several hours or until completely set to a stiff jelly. The reason a shallow dish is used for chilling the emulsion is so that it will set the gelatin to a stiff jelly faster and more evenly than if left in the beaker.

[In the following steps it is advisable to wear latex gloves; not because of potential silver stains, but to prevent contamination of the emulsion from chemicals that might be on your hands.]

9. Under red safe light remove the emulsion from the refrigerator and pull the dish from the bag. The emulsion will look white under the safe light (it is actually

bright yellow). Scoop out the firm jelled emulsion with the stainless steel spoon and put it into the potato ricer.

- 10. **Noodling & Washing:** Place the 25" sheer white nylon fabric in the stainless steel wire mesh drainer and lay the drainer in the mixing bowl. Squeeze the ricer to extrude emulsion noodles that will fall into the center of the fabric. When the emulsion is completely noodled into the fabric, gather the edges of the cloth and secure the bag of noodles with cotton string. Fill the mixing bowl with very cold distilled water (add a few ice cubes) and move the noodle filled fabric in the water with you're your hands for about five minutes. Let the noodles soak for five more without agitation. Change the water two more times and wash the noodles as before.
- 11. **Re-Melt & Finals:** Drain the washed emulsion noodles thoroughly for at least 15 minutes. It is important to remove as much excess water as possible because too much water will dilute the emulsion causing cause weak, thin images. Place the drained noodles in a clean 500 ml Pyrex beaker. Re-melt the emulsion using the electric crock pot at around 120 degrees F and add "finals." The finals listed below are chrome alum, added to make the emulsion set to a stronger film to withstand processing, alcohol to aid in coating and thymol, to prevent bacteria growth. Add the chrome alum solution drop by drop with agitation of the emulsion or the emulsion will seize into a golf ball size lump.

[Chrome alum hardener: The original 1887 formula *does not* include chrome alum. It is included here to help the novice emulsion maker produce an emulsion that does not frill or melt during processing. You may choose to leave chrome alum out of the formula. It can always be added to the emulsion immediately before coating plates.

No chrome alum in the emulsion may result in a very fragile film that might melt off the plate if processed in solutions that are too warm. Too much chrome alum on the other hand will prevent the gelatin from absorbing the processing chemicals resulting in thin images and prolonged developing times.

The hardening effect of chrome alum continues long after the emulsion has dried on the surface of the plate, so it's best to use the plates within a week or so to keep development time short. There is no way to include the absolute correct amount because the characteristics of each source of gelatin are going to be different. The quantity of chrome alum may need to be decreased or increased as needed. *Make only as much chrome alum solution as you will need as it goes bad quickly.*]

#### Finals

2 ml of a chrome alum solution (1.25 grams chrome alum into 25 ml distilled water) 5 ml 95% grain alcohol 0.065 grams thymol

When the finals are added and fully incorporated into the emulsion pour the entire contents into a brown ceramic cheese crock, cover the opening with a piece of black opaque plastic and secure the ceramic lid with the wire spring. Place the emulsion filled crock in the refrigerator for future use. Remove only what's needed when coating a batch of plates by scooping out the chilled emulsion with a stainless steel spoon. The stock emulsion will last many months if kept cool and protected from white light.

This emulsion is fairly slow by modern standards and only sensitive to blue, violet and invisible ultra violet light. Assume an ISO rating of between 3-10.

# Coating Glass Plates with Gelatin Emulsions

Coating glass plates with gelatin emulsions is a little different than working with a solvent based binder such as collodion, which relies on evaporation for the coating to set to a firm film. Gelatin emulsions must be heated to a liquid form, and once applied to the glass support, be able to set back into a firm jelly at an average room temperature. This so called "set time" or "setting time," is governed by the ambient temperature of the room, the bloom and percent of gelatin and the quantity of alum added as a "final" to the emulsion.

Shortening the setting time was almost always done by quickly lowering the temperature of the emulsion. The most common approach was to level a piece of marble or thick glass and place the coated plates upon the surface until the emulsion cooled and became firm. The setting of gelatin emulsions on paper supports was generally accomplished by chilled air.

The earliest commercially made gelatin dry plates were coated by pouring the emulsion by hand. An 1884 account of the coating operation at the Cramer Dry Plate Works in St. Louis was described as "eight busy men, with pitchers of emulsion on one side, a pile of glass on the other and in front of them, a peculiar leveling stand." \*

\* Philadelphia Photographer, Jan 1884, p 11

The following instructions are based on the techniques of the early 1880s, before the invention of cascade or transfer coating and continuous belt chilling chambers. This system of hand coating with a chilling table is not difficult to master and enables one person to make dozens of plates in one sitting. The only real limitation is the capacity of the drying box.

# Equipment and Materials Needed

#### Equipment

- 1. Glass cutter
- 2. Cork backed straightedge
- 3. Small sharpening (whet) stone
- 4. 2" wide natural bristle paint brush
- 5. Soft natural hair make up brush
- 6. 2 wood plate racks (see Appendix )
- 7. liquid dish detergent
- 8. rottenstone or calcium carbonate powder
- 9. ½ yard fine cotton or linen cloth cut into 5" and 8" squares
- 10. Small stainless steel container with lid (an old stainless steel film developing tank is perfect)
- 11. electric crock pot with at least two temperature settings
- 12. small ceramic tea pot, antique invalid cup or small glass beaker with a lip
- 13. Photo Flo (as needed)

- 14. electric laboratory hot plate
- 15. deep red safe light
- 16. paper towels
- 17. 2 1'x1' marble tiles or ¼ glass plates
- 18. small spirit level
- 19. 2 wood leveling stands (see Appendix)
- 20. drying box (see Appendix)

# **Cutting & Cleaning Glass Plates**

Window glass is good enough for hand coated photographic plates. Make sure you purchase these from a framing supply house in unopened boxes. The plates should be interleaved with paper to prevent surface scratches. If you have never cut glass before, pay a visit to a stained glass shop, purchase the best glass cutter you can afford and ask for a demonstration.

Cut the glass to the desired size and never lay the plate surface down on anything or it will become scratched. Place the plate upright against a wall or in a wood rack. It is extremely important to remove the razor sharp burr on all the edges on both sides with a small hand sharpening stone. Dust off the powdered glass dust with a stiff natural brush. This is also a good time to check if the plates fit your holders before you coat them.

Apply a drop of detergent to each side of the glass and wash the plate under warm running water rubbing the surface thoroughly with a small square of cotton cloth. If the plates are particularly dirty you may add a dusting of fine rottenstone or calcium carbonate to each side with the detergent. Keep washing in running warm water without the cloth until the water sheets off evenly. Handling by the edges only, place the plate upright in a rack on a piece of blotting paper to dry. Once dry, breath on the surface of the plate and rub the condensation with a clean piece of the larger cotton cloth squares until you see no streaks.

# Heating and Pouring the Emulsion (under red safe light)

The whole coating procedure requires very little time; less than ten seconds for a 5x7 plate from the initial pour to placing the plate on the chilling table. It is similar to coating collodion plates, though not exactly. *Naturally all of the following is performed under red safe light conditions*. Before you turn off the white lights, level your chilling tables using the spirit level.

- 1. Remove the emulsion from the refrigerator. Scoop out enough cold gelatin emulsion for several plates and place this in a stainless steel container (developing tank) with a light trap lid. Put the tank containing the emulsion in a heated crock pot and with enough water to keep the emulsion warm without having the container float or flip over. Heat the emulsion until it is very liquid. The actual temperature will depend on the pouring qualities of each batch of emulsion.
- 2. When the emulsion is thoroughly liquefied, pour some through a large square piece of clean, fine cotton fabric into the pouring cup (I prefer the antique invalid cup, but a small glass beaker will do). Allow the emulsion to settle so that the

bubbles rise to the top and pop. The pouring cup can be kept warm in the crock pot between pouring plates.

- 3. Slightly heat the plate of glass by placing it on the surface of a warm (not hot) laboratory hot plate covered with two layers of paper towels. While the plate is still warm hold it in the left hand with the fingertips supporting the back of the plate. Give the plate a quick dusting with the make-up brush.
- 4. Holding the pouring cup in the right hand, pour a sufficient quantity onto the center on the plate (pouring too little is worse than pouring too much). Keep the plate level so that the creamy emulsion forms a perfect circle. (illustration)
- 5. Gently tilt the plate so that the emulsion flows progressively to all four corners without going over to the back side. Once the plate is completely covered, gently let some of the emulsion flow off one corner of the plate and back into the pouring cup and immediately afterwards pour some of the excess off from the opposite corner into the pouring cup. (illustration)
  - Each corner from which you poured the excess will drip a small amount of emulsion that rolls to the back of the plate. Do not worry about this, it is typical of hand coated gelatin plates and this artifact can be seen on historic examples. Any emulsion on the back of the plate can be removed with a razor blade after the emulsion is dry or after the negative is processed. (illustration)
- 6. Gently rock the plate for a couple of seconds so that the emulsion redistributes evenly on the surface. Immediately place the plate on the leveled chilling slab leaving a lip to hang over the edge of the slab for subsequent removal. The emulsion should still be warm and still fluid enough to level itself on the surface of the plate before it begins to set to a firm even coating. (illustration)
- 7. As the gelatin begins to set up you will probably see some dimples on the surface and possibly some dust. Move the plate to the next chilling table until the emulsion is firm enough to place upright on a rack in the drying box. You can test the firmness of the emulsion by touching one corner with your finger, though with experience you'll eventually have a sense when they're ready to be removed from the slab.

The slower gelatin plates are dried the better. Fast drying can cause ridges in the surface of the emulsion. Make sure the drying box is absolutely light tight but fitted with adequate ventilation. Collect the dry coated plates the next day and place them in a light proof box interleaved with clean paper until needed.

## **Processing Gelatin Emulsion Plates**

THE EVOLUTION OF DEVELOPERS, IN BLUE, CAN BE DELETED FROM THIS ARTICLE IF SPACE IS LIMITED.

The earliest developers used for gelatin emulsion plates were based on either **ferrous oxalate** or **pyrogallic acid**, known simply as **pyro**. Unlike the calotype, albumen negative or collodion processes that preceded gelatin technology, all of the developing

agents for processing gelatin emulsions were used in an **alkaline** state. Ferrous oxalate was one of the first developers used for gelatin plates, though it fell from favor by the end of the century. Pyro was introduced in the 1850s to develop collodion negatives and has continued to attract devotees well into the 21<sup>st</sup> century for development of silver based film stock. Pyro development produces warm brown silver deposits with a slight yellow stain.

By the 1890s, **hydroquinone** and **metol** based developers were being offered by photographic suppliers. Metol developers produced cool, blue-black silver deposits and developed very quickly though with little density. Hydroquinone development resulted in warm black silver deposits that developed slowly with greater density potential than metol. By the late 19<sup>th</sup> century, most photographers chose either pyrogallic acid or a combination of metol and hydroquinone, simply called "MQ."

A typical developer formula for processing gelatin emulsions has the following elements; the **reduction agent** (pyro or metol/hydroquinone), an alkaline **accelerator** (ammonia or sodium carbonate), a **restrainer** (usually potassium bromide) and a **preservative** such as sodium sulfite. By knowing the function of these components, a photographer could tweak the formula to suit specific needs and correct exposure problems to some degree.

Development of emulsion plates is most easily done in a white tray so that the progress of development can be easily viewed by safe light. All other chemical operations can be done in any type of tray, though Pyrex glass is always the best choice as it is easily cleaned. Development is by a red safe light. The effect of over or under exposure can be seen during development and the knowledgeable photographer has the opportunity to adjust the developer as needed to produce the best possible results. It was typical in the 19<sup>th</sup> century for the photographer to have small dropper bottles of accelerator (ammonia or calcium carbonate) and restrainer (potassium bromide) solutions at the ready near the processing sink.

# Processing the Negative [under red safe light]

The exposed plate is placed, emulsion side up, into the white tray containing enough developer to cover the plate. The developer should be used at a temperature of around 65-68 F. It is necessary to rock the tray during the entire development so that fresh developer is always in contact with the emulsion.

When developing by inspection, the most common mistake is to stop development too soon. The maximum density areas of a negative always look much darker when working under safe light. It will be necessary to lift the plate from the tray and inspect the progress by looking through the plate, illuminated from behind by the safe light. No development time can be assigned as there are too many variables with hand made emulsions. An average, properly exposed landscape negative develops gradually with the sky visible first, followed by architecture and eventually well lighted foliage. Do not expect to see details in the deep shadows of foliage due the insensitivity of a bluesensitive emulsion. Development can be carried out for 20 minutes if needed.

Once development is judged to be complete, the plate is washed under gentle running water for two minutes or in a tray with two changes of water and gentle agitation. Do not use an acid stop bath as this may shrink the emulsion causing frilling. Fix the negative in a tray of sodium thiosulfate for five minutes with occasional agitation. Wash

the plate in several changes of fresh water or running water for at least 20 minutes and then place on a rack in a dust free place to dry.

#### **Troubleshooting**

**Peeling Problems:** On occasion, photographers had problems with the emulsion lifting from the edges of the plate; an effect called "frilling." Assuming the glass support was properly cleaned, this usually happened when the developer was either too alkaline or the temperature of the developer was too hot. A simple 2% alum hardening bath before or after development, or a little alum added to the fixing solution was usually enough to prevent frilling. If peeling is a persistent problem, pre-coat your glass plates with a 2% subbing solution of hard gelatin containing 1/2% chrome alum using the same method described for coating with the emulsion.

**Image does not appear or takes too long to develop:** The plate was either not exposed or the emulsion was too hard due to excessive chrome alum. A pre-soak in 70 degree F water for about five minutes will usually soften the emulsion for development. You may also add a drop or two of glycerin to the pre-soak. Adding a few drops of ammonia to the developer will also help, though excessive ammonia will soften the gelatin too much, causing frilling and fog.

**Fog:** There will always be some degree of fog present in gelatin emulsions. Too much fog is a problem. The most common causes of fogging are pre-exposure of the emulsion or the coated plate to white light or a faulty safe light. If the thin shadow cast on the negative by the plate holder is without fog; the problem is overexposure in the camera. Fog is also caused by using too warm or too alkaline a developer. Poor quality gelatin also causes fogging.

Thin, flat images: It is common for the beginner to produce thin images. This is usually due to overexposure combined with underdevelopment. Overexposure is responsible for detail in both the deep shadows and strongest highlights of the image. Using cold or dilute developer, not developing long enough or with enough agitation will also result in weak, thin images. Density is gained by proper exposure combined and prolonged processing with a strong developer and adequate agitation. Excess water in the emulsion from not draining the reserve gelatin or the washed noodles effectively is common cause of weak, thin images.

**Too much contrast:** High contrast is the result of underexposure and over development. If there is no shadow detail despite prolonged development, it is a clear sign of underexposure.

#### **Formulae**

Kodak Dektol can be been used effectively for processing the emulsion described earlier in this section. Begin by using it undiluted and dilute with water if you feel the maximum highlight density is too strong. You may also make your own MQ (metol/hydroquinone) style developer. Kodak D-49 was originally formulated for processing bromide prints though it can be used undiluted for negatives made with ordinary blue-sensitive emulsions. When making alkaline developers, the ingredients should be added to the hot water in the order listed and each ingredient fully dissolved before the next is added.

#### D-49 Developer

```
500 ml distilled water (around 120 F)
3.1 grams metol
45 grams sodium sulfite
11 grams hydroquinone
45 grams sodium carbonate
2.1 grams potassium bromide
Cold distilled water added to make a total 1000 ml
```

As with all MQ developers, metol and hydroquinone are the active developing (reduction) agents. Potassium bromide is the restrainer. Sodium carbonate is the accelerator and sodium sulfite, the preservative. If you want more density than extended development will provide, increase the hydroquinone. You may also raise the pH by adding ammonia or more sodium carbonate. This is most easily done by adding drops of household ammonia.

A good starting point is about 4-6 drops in 100 ml developer. Pour the developer from the developing tray into a glass beaker, add the ammonia to the developer solution and then pour the developer back into the tray. Raising the pH with ammonia or sodium carbonate will make the gelatin soften and more permeable so that the developer can be more effective, though too much will cause the emulsion to fog, lift from the glass and cause frilling.

Decreasing the potassium bromide restrainer in the formula will also cause the developer to work faster, though by doing this there is always a chance of causing fog. Tweeking the developer formula as needed eventually becomes intuitive.

```
Sodium Thiosulfate Fixer (working solution)
1000 ml tap water
150 grams sodium thiosulfate
```

#### **APPENDIX**

There are three things you will need to construct before coating gelatin emulsion plates; 2 plate racks, 2 wood leveling stands for the chilling tables and a plate drying box.

# Photographic Plate Racks

Vintage plate racks can be purchased at antique shops and on internet auctions, though availability is uncertain. (illustration)You can make a nice rack by drilling pairs of holes into the top of a wooden board and fitting a series of wood dowels. (illustration) The size plate you wish to coat will dictate the size of the materials.

Leveling Stands for Chilling Tables

#### Materials

```
two 8"x8" pieces of 3/4" birch plywood
six 11/2"x 5/16" bolts
six 5/16" nuts
six 1/4" flat washers
six 5/16" coupler nuts
```

A leveling stand is essentially a short adjustable tripod, with wide, flat top. Drill three 5/16" holes through the plywood. [Fig?] Countersink the holes so that the heads of the bolts sit below the surface of the plywood when installed. Install the bolts and attach the washers and nuts to the bolts on the underside of the plywood. Thread the coupler nuts on the end of each bolt. The coupler nuts allow adjustment of the leveling stand from below. The marble (or glass) chilling plate rests upon the leveling stand to complete the chilling table.

#### Plate Drying Box

In time you may want to make a sturdy wood box with filtered ventilation, but for your first experiments a cardboard drying box is easy to make and will do the job. You will need one, good quality, corrugated cardboard box and a couple extra sheets of single weight cardboard for constructing the ventilated light trap. The size of the box is dictated by the size and quantity of the plates you wish to coat in one session. While any tape will work, water soluble gummed paper backed tape (available at art supply stores) never fails over time.

Simply put, the box must allow adequate ventilation without exposure to white light. Holes must be cut into either end of the box and then fitted with a light trap as illustrated. Tape the lid closed with black tape after you fill the box with plates and for extra protection pace a piece of dark cloth over the top.

#### **Further Reading**

The 19<sup>th</sup> century photographic journals are a great wealth of gelatin emulsion technology, particularly for the individual making and coating their own emulsions on paper and glass. English language publications include the *Photographic News*, *British Journal of Photography*, *British Journal Almanac*, *Philadelphia Photographer*, *American Annual of Photography and Photographic Times Almanac* and *Anthony's Photographic Bulletin*.

The following out-of-print texts should be sought by every emulsion enthusiast. The more common titles are often quoted like scripture by a growing subculture of emulsion makers.

<u>The Practical Working of the Gelatin Emulsion Process</u> Captain W. De W. Abney, Piper & Carter, 5 Castle Street, Holborn, E.C. London, 1880

Modern Dry Plates or Emulsion Photography Dr. J.M. Eder, E. & H. T. Anthony & Co. No. 591, Broadway, NY 1881 (English edition)

Wilson's Photographics E.L. Wilson, No. 853 Broadway, NY, 1881

The Photographic Negative W.H. Burbank, Scovill Manf. Co., NY, 1888

<u>Photographic Emulsions, Their Preparation and Coating on Glass, Celluloid and Paper, Experimentally and on the Large Scale</u> E.J. Wall, American Photographic Publishing Co., Baker, 1929

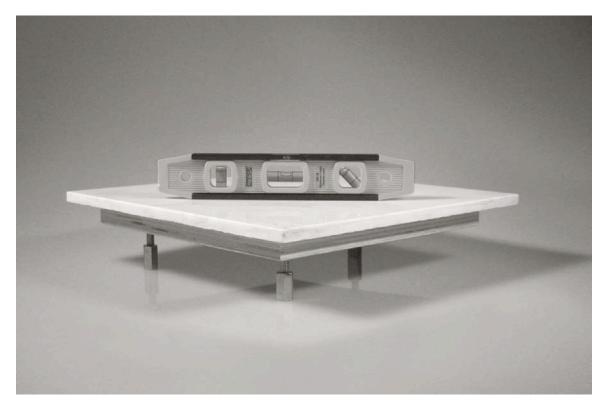
<u>Photographic Emulsion Technique</u> T.Thorne Baker, American Photographic Publishing Co., Boston, 1948

Photographic Emulsion Chemistry G.F. Duffin, Focal Press, London & NY, 1966

<u>Silver Gelatin; A User's Guide to Liquid Photographic Emulsion</u> Martin Reed & Sarah Jones, Argentum/Aurum Press, 25 Bedford Ave., London, 1995

Finally, there is the internet, where you will find a huge amount of information posted by individuals with a wide range of experience. In most cases however, you will be better served to read the original texts and evolve though your own personal experience.

Revised Wednesday May 9th, 2007



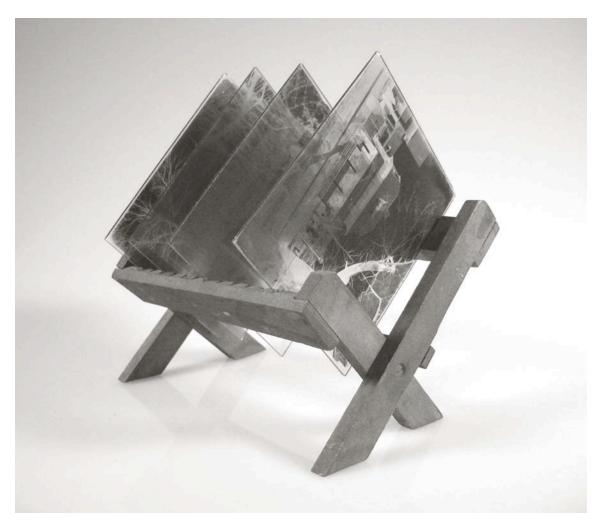
Leveling Table made with 1" square sheet of marble, plywood and three adjustable levelers.



Detail of adjustable leveler. It is made by inserting a bolt into a countersunk hole from the other side of the plywood. The washer and nut secure the leveler to the wood plate and the hexagonal connection nut adjusts for height.



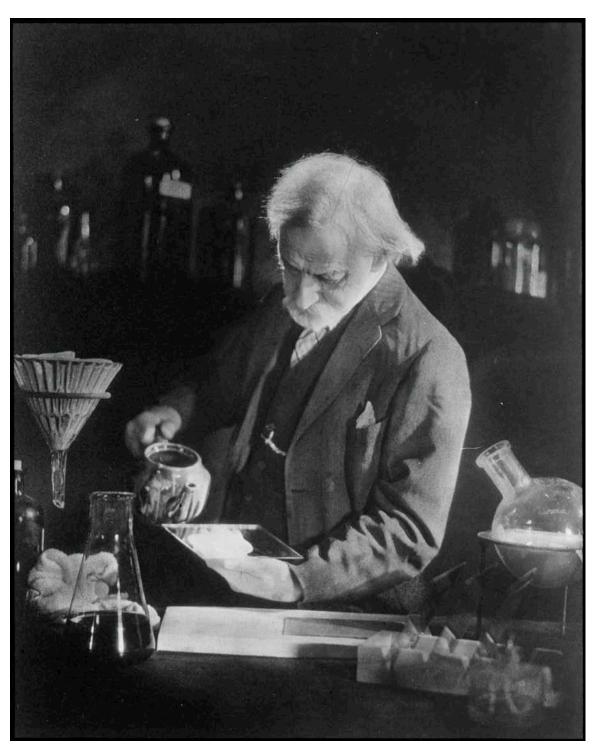
Two cheese crocks for cold storage of gelatin emulsions. Make sure the seal is still intact or place a sheet of opaque black plastic over the opening before closing the lid. The invalid cup [front] is used for pouring warm emulsions onto glass plates. It is perfect for this operation because the fluid pours from the bottom, keeping any bubbles from flowing onto the plate. A modern gravy separator can also be used, but it doesn't have the barrier over one half of the upper opening.



Wood plate drying rack. These can be purchased at antique shops, on ebay and there are a few people making reproductions for photographers using the wet collodion process.



Stainless steel potato ricer. This is perfect for noodling the chilled emulsion. Be sure the noodles fall into very cold water to prevent diluting the emulsion.



Coating gelatin emulsion plates using a tea pot.



The next five images show the sequence of pouring warm gelatin emulsion onto a warmed  $4 \times 5''$  glass plate. [These pictures were photographed in white light for better visibility.] After coating the plate, it is placed upon the leveling table to "set" the gelatin. Once the emulsion is firm and no longer mobile, the plates can be placed upright in a drying cabinet until the emulsion is completely dry. The final image showing draining from the opposite side was not included in the book which I considered a mistake as it is a critical step to make an even coating.







