

BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
Washington

METHODS OF SILVERING GLASS

In response to numerous requests for methods of silvering mirrors, the following formulae and directions have been collected. The methods described are selected from the large number of silvering formulae available, and are those which are in most common use.

CLEANING THE SURFACE TO BE SILVERED

This is the most important part of the process, whatever formula is used. The glass surface must be chemically clean. A greasy surface, which has not previously been silvered, should be cleaned with some such solvent as alcohol or ether. Following this, the surface should be scrupulously cleaned with nitric acid.

Make a swab by winding absorbent cotton on the end of a glass spatula or glass rod, with sufficient thickness of cotton so that there will be no danger of scratching the glass with the rod. With such a swab and pure nitric acid, to which a little distilled water may be added, clean every part of the surface; considerable pressure should be used in rubbing with the swab. Do not let any part of the glass become dry in this process; if it does, swab and clean again. Rinse off the nitric acid, for which ordinary water may be used at first, followed by distilled (or rain) water. Finally leave the mirror in a tray or other container, covered with distilled water, until ready to silver. No part of the mirror should be allowed to become dry.

With large mirrors, the mirror itself is made the container. In the case of circular mirrors, a band of paraffined paper is tied tightly around the disk and "cemented" to the glass with a soldering iron. A wall of wax or putty is frequently built up around the edges of large rectangular mirrors for the same purpose.

After cleaning with nitric acid, many advise a second cleaning with a strong solution of caustic potash, followed with an application of French chalk, and rinsing as above. The nitric acid alone will be found sufficient, provided the cleaning is thoroughly done, and plenty of pressure used in the swabbing.

1 gram = 0.0353 ounce.

3.1416
9

28.2744 sq. inches

$\frac{28.27}{4} = 7.07$ grams.

.0353
7.07

2471

2471

249571 ounce

14 grams.
 $\frac{6}{84}$ silver nitrate
cc.

$\frac{1}{4}$ ounce silver nitrate

3 ounces water.

$\frac{1}{8}$ ounce caustic potash

1 ounce water.

In commercial silvering many manipulators follow the cleaning with nitric acid by a vigorous swabbing with a saturated solution of stannous chloride (SnCl_2) which is carefully rinsed off with warm water. This is regarded as an essential feature in most of the "secret processes" used in the trade.

PURITY OF CHEMICALS USED

All chemicals used in the formulae must be of high purity, of the grade known in the trade as C.P.; the use of impure reagents will result in failure.

Distilled water is best; if this is not available use rain water. In some localities it will be found that the water from the taps will answer instead of distilled. A test on a small mirror will decide this point. If the solution turns a light pink or blue when the silver nitrate is dissolved in the water, the water is probably too impure for the purpose.

BRASHEAR'S PROCESS

This process is probably used more than any other for silvering the surface of large mirrors used in reflecting telescopes, and laboratory mirrors where a thick coat is desired.

For most work the following proportions will be found adequate:

$$\frac{\text{Square cms.}}{40}, \text{ or } \frac{\text{Square inches}}{6} = \begin{array}{l} \text{(No. of grams} \\ \text{) silver nitrate} \\ \text{(required.)} \end{array}$$

For very thick coats, and for astronomical mirrors, many prefer a more liberal allowance of silver nitrate, about as follows:-

$$\frac{\text{Square cms.}}{27}, \text{ or } \frac{\text{Square inches}}{4} = \begin{array}{l} \text{(No. of grams} \\ \text{) silver nitrate} \\ \text{(required.)} \end{array}$$

CAUTION. In using the Brashear process keep the solutions, and do the silvering, at a temperature of about 15°C . or 65°F . In hot weather it is advisable to use ice to keep the temperature of the solutions below 18°C . (72°F .). If warmer than this the resulting coat is apt to be soft, and there is danger of the formation of small amounts of silver fulminate, which is very explosive.

The Reducing Solution:

Rock candy	90 grams
Nitric acid (sp. Gr. 1.22)	4 cc
Alcohol	175 cc
Distilled water	1000 cc

This reducing solution is preferably made up in advance; the older it is, the better it will work. If necessary to use it at once, the action may be improved by boiling it, adding the alcohol after it has cooled.

The Silvering Solutions. (Make up just before silvering)

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|----|---|----------|
| A. | Distilled water | 300 cc |
| | Silver nitrate | 20 grams |
| | Strongest ammonia, as may be needed (see below) | |
| B | Distilled water | 100 cc |
| | Caustic potash | 10 grams |
| C | Distilled water | 30 cc |
| | Silver nitrate | 2 grams |

In solution A, after the nitrate is all dissolved, add ammonia gradually. The solution will at once turn a dark brown. Continue adding ammonia, drop by drop toward the close of the process, until the solution just clears up; avoid an excess of ammonia. Then pour in Solution B; the mixture will again turn dark brown or black. Again add ammonia, drop by drop toward the close, and stirring constantly, until the solution just clears up again. It should now be a light brown or straw color, but transparent.

Next add slowly, stirring constantly, as much of the reserve silver solution, C, as the mixture will take up without turning too dark; it is important that the nitrate of silver be in excess. Continue this till there is quite a little suspended matter, which the solution refuses to take up. Filter through absorbent cotton.

When ready to silver, pour into this mixture about 6 cc of the reducing solution for each gram of silver nitrate used, and pour at once upon the mirror, which has been lying covered by about the same amount of water as is used in the solutions; this water need not be poured off.

The process will be finished in from three to eight minutes, depending on the temperature of the solutions, which should never exceed 18°C. (72°F). It is well to make preliminary tests in small beakers or drinking glasses to get the time necessary as the coat is apt to bleach if process is continued too long. Keep solution in motion so that the thick sediment which forms will not deposit on the silver coat. A very light swabbing with loose absorbent cotton, over every part of the mirror, will be found advantageous in large mirrors, as soon as the coat begins to form. Avoid exposing the surface to the air for more than a second or two at a time to observe progress.

Get the spent solution off quickly at the close of the process; rinse thoroughly, first with ordinary and then with distilled water; swab lightly with absorbent cotton while rinsing if there is much "bloom" on the surface.

THE FORMALDEHYDE PROCESS

The Reducing Solution.

Distilled water	200 cc
Merck's formaldehyde	40 cc

The Silver Solution.

Distilled water	1000 cc
Silver nitrate	21.6 grams

Add strong ammonia gradually and clear up fully.

Mix these two solutions thoroughly and quickly and pour on the mirror; keep solution in motion. When the solution is clear with the exception of small black grains like gunpowder, and these appear to be depositing on the silver coat, the process is complete. Rinse thoroughly. Temperature about 20°C (77°F).

THE ROCHELLE SALTS PROCESS.

There are many formulae which employ Rochelle Salts or tartaric acid as a reducing agent. These formulae are the ones ordinarily employed in silvering mirrors commercially, where the silver coat is on the back surface of the glass, and is protected by copper plating or by painting. The glass sheets are cleaned with nitric acid, or by allowing them to stand in a concentrated solution of potassium dichromate in dilute sulphuric acid. After rinsing, the sheets are rubbed with a tin chloride solution, and rinsed. The following formula has given good results in use:-

The Reducing Solution.

Make up a saturated solution, at about 18°C., of Rochelle Salts in distilled water.

The Silver Solution.

Dissolve 42.8 grams of silver nitrate in as little water as possible, just clear up with strongest ammonia, filter through cotton, and dilute to 250 cc.

At

At 30°C
use 40 cc
20 cc
400 cc

At 30°C
30 cc of the reducing solution
20 cc of the silver solution
400 cc distilled water

The process is complete in from 40 to 60 minutes, depending on the temperature. For semi-transparent coats (half-silvering) stop the process sooner. If coat is not thick enough, rinse off the spent solution and any sediment and repeat with a fresh solution.

THE CATHODIC PROCESS

The cathodic method of silvering is particularly useful as a laboratory process for half-silvering and for silvering delicate articles, those which must be kept dry, and in cases where portions of the surface are to be left clear.

The apparatus consists of a glass bell-jar ground on the bottom to fit a metal plate, generally aluminum, to which it can be sealed by melting wax (about six parts of beeswax to four parts of rosin) around the edges. A clean and polished plate of silver, without projecting points, is mounted on an aluminum rod enclosed in a glass tube, and forms the cathode, the bottom plate acting as the anode. If the bottom plate is of glass, a rod must be cemented in to act as the anode. The cathode should preferably be large enough to cover the object to be plated; the object is placed on a stand so as to be 2 or 3 cm. from the cathode.

The anode and cathode are connected to a transformer giving 5,000 or 10,000 volts, or to an induction coil, and the apparatus exhausted till the cathode's dark space extends beyond the object to be plated. The object to be plated must be clean and dry, and if it is transparent a piece of white paper with a black cross marked on it should be placed beneath so that the progress of the plating can be observed. Metals other than silver can be plated by replacing the silver with a plate of the metal required.

DRYING, BURNISHING, ETC.

Stand on edge to dry; remove water at edges with blotting paper.

For front surface silvering, burnish, after mirror is perfectly dry, by making a pad of softest chamois skin wrapped around a wad of cotton. Rub a very little of best optical rouge into this pad, and go over entire surface in circular strokes. Dust mirror and pad occasionally during the burnishing to avoid scratches.

Burnishing is not necessary for back surface silvering, as in ordinary looking glasses. The silver coat may here be covered with one or two coats of shellac, and later covered with paint or other protector.

THICKNESS OF THE FILM

Nobili's rings may be used to determine the thickness of the film. Place a very minute crystal of iodine on the silver surface; obviate the effect of air drafts by placing over it a small beaker, which should not, however, fit the surface tightly. Count the rings which will form around the crystal in a few minutes.

No. of ring	Thickness of film	
To first dark ring	0.000,018 mm	
" second bright ring	37	thin
" third " "	74	
" fourth " "	110	thick
" fifth " "	147	
" sixth " "	184	
" seventh " "	220	very thick

GENERAL INFORMATION AND REFERENCES

Rubber gloves will be found a convenience.

Nitrate of silver stains on the hands, if freshly made, can be removed by bathing the hands in hot hyposulphite of soda, or in a dilute solution (cold) of potassium cyanide (very poisonous; should not be used if there are cuts or abrasions on the hands.)

The merest trace of chlorine, free or in combination, will cause failure. In silvering small mirrors, where small amounts of solution are used, use care that the solutions are not contaminated by the salt in the natural perspiration of the hands.

Brashear's Method, Astrophysical Journal, 1, 252, 1895.
Draper's Method, Smiths. Contrib., 1356, XIV
Liebig, Annalen der Physik, 1867, Sup. V., 153
Chattaway, Chemical News, Sept. 27 and Oct. 4, 1907.
Curtis, "Methods of Silvering Mirrors", Publ. Astron. Society of the Pacific, 23, 13, 1911.

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A ROCHELLE SALTS PROCESS

Solution A

Silver nitrate - - - - - 10 grams.

Distilled water - - - - - 100 grams.

To this add concentrated ammonia until the precipitate first formed is just redissolved (care used in mixing well).

Then add drop by drop 10% solution of silver nitrate in water until the solution is opalescent.

Dilute to 1 liter, filter and bottle.

Solution B

Silver nitrate - - - - - 2 grams.

Rochelle salt - - - - - 1.66 grams.

Distilled water - - - - - 1 liter.

Bring the solution of silver nitrate in distilled water to a boil. Then add Rochelle salt dry. Boil 5 minutes; stirring all the time. Filter hot and keep in dark bottle.

To Silver

Use equal parts A and B at room temperature. The deposition of silver on the glass surface will be more rapid and complete, if that surface is several degrees warmer than the solution.



