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Use of Acidified Hydrogen Peroxide to Remove Excess Gun Blue from Gun Blue-Treated Cartridge Cases and to Develop Latent Prints on Untreated Cartridge Cases

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ABSTRACT: A formula used by firearms and toolmark examiners to clean lead residue from bullets was also found to remove excess gun blue coating from metal cartridge cases. It consists of a mixture of acetic acid and hydrogen peroxide and works by oxidizing the coating back into solution. This explanation allows one to determine the optimum proportion of the mixture. Acidified hydrogen peroxide formulas were also found to "clean" (oxidize or etch) metal cartridge cases in areas where there was no sebaceous latent print material (or other substances that resist oxidation). The subsequent treatment of these "cleaned" cartridge cases with gun blue to enhance the latent print contrast was also considered.

KEYWORDS: forensic science, gun blue, latent fingerprint, acidified hydrogen peroxide, cartridge cases, metal etching, metal deposition, oxidation-reduction

Recently, the Maryland State Police laboratory had a case involving a cartridge case that had been overdeveloped with gun blue. (See below for an explanation of the gun blue process). Their firearm examiner (JK) was not certain if this had covered or damaged any identifying toolmarks on the cartridge case. One of their fingerprint experts (JS) was asked if there was any method to remove enough of the gun blue coating to facilitate the firearms examination. They called the U.S. Secret Service (USSS) for assistance but no immediate answer was provided. In the meantime, the firearm examiner (JK) tried a formula he recalled from an Association of Firearm and Toolmark Examiners (AFTE) publication (1). The formula is used to remove excess lead residue from copper-jacketed bullets. In fact it is called a "lead solvent." It worked on the gun blue overdeveloped cartridge case, particularly on the base where the firing pin impression was to be examined. The formula consists of 10 mL glacial acetic acid, 2 mL 30% hydrogen peroxide, and 70 mL distilled water giving a total volume of 82 mL.

The USSS decided to study how this solution acts as a gun blue removing formula. It was quickly found to work by an oxidation-reduction (redox) process: acidified hydrogen peroxide oxidizes the copper and selenium components of the gun blue coating converting it to soluble species. It was also found to oxidize the metal of cases except where sebaceous prints exist (or other oxidation-resistant materials). This promoted the study of its use as a latent print visualizing reagent. This study considered only fingerprints placed on post-fired cartridge cases (rather than prints placed on cartridges prior to firing and visualizing them after firing).

Experimental

Preparation of Cartridge Case Test Samples—Sebaceous prints, obtained by rubbing a finger on one's forehead, were placed on brass, nickel-coated brass, and aluminum cartridge cases. A portion of these cartridge cases were processed with a mild superglue treatment on the same day. The dilute gun blue and acidified hydrogen peroxide tests were also done on the same day for convenience. Mild superglue treatment, in this case, was a five-min treatment of the cartridge cases in a closed chamber (4 L volume) with superglue (about a 2 cm diameter size drop in an aluminum cup) and a small beaker of warm water (50 mL). This does not necessarily produce visible prints. Supergluing prior to an etching process was first mentioned in 1984 by Almog and Gabai (2). Their process involved an electrochemically accelerated etching process involving a "copper and alloys electropolishing solution."

Chemicals—The chemicals used were Gun Blue (Brownells Formula 44/40 Instant Gun Blue), glacial acetic acid (J.T. Baker), 3% hydrogen peroxide (CVS—local brand), 5% acetic acid vinegar (TownHouse—Safeway brand), concentrated (37%) hydrochloric acid (Mallinckrodt), and distilled water.

Reagents—The following reagents were prepared: 1. (Dilute Gun Blue)—A 1.25% (v:v) (approximately) gun blue solution was made by taking 1 part gun blue and adding 80 parts distilled water (3). 2. (The AFTE H_2O_2/HOAc Formula)—10 mL glacial acetic acid, 20 mL 3% hydrogen peroxide, and 52 mL distilled water giving a total volume of 82 mL. Here HOAc represents acetic acid. 3. (An H_2O_2/HOAc Optimized Formula)—14.1 mL 5% vinegar and 20 mL 3% hydrogen peroxide giving a total volume of 34.1 mL. and 4. (An H_2O_2/HCl Optimized Formula)—1.2 mL concentrated HCl (37% or 10.2 Molar), 20 mL 3% hydrogen peroxide, and 13 mL distilled water giving a total volume of 34.1 mL.

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Preparation of Gun Blue Overdeveloped Cartridge Cases—Brass cartridge cases with freshly placed sebaceous prints (superglued and non-superglued) were dipped into the diluted gun blue solution and stirred occasionally. This was continued past the stage where latent prints become visible (one normally stops at this stage) until they were so coated that they were barely visible. If stirring is not done, a spongy or mossy coating of finely divided materials forms which can be shaken off or rubbed off. These overdeveloped cartridge cases were then placed in a water bath for about two minutes and then allowed to air-dry to harden the gun blue coating. Light rubbing removed any weakly adhered coating. Nickel-coated brass and aluminum cartridge cases were not tested since these do not take the gun blue coating as well as the brass cartridge cases.

Procedure to Remove Excess Gun Blue—Each gun blue overdeveloped brass cartridge case was dipped in one of the three acidified hydrogen peroxide solutions and gently stirred. When sufficient gun blue was removed (determined by visual examination), the cartridge case was placed in a water bath for two minutes after which it was air-dried.

Procedure to Visualize Latent Prints by Etching—Each of the three types of cartridge cases (brass, nickel-coated brass, and aluminum) with freshly placed sebaceous prints (superglued and non-superglued) was dipped in one of the three acidified hydrogen peroxide solutions. After the print became visible (determined by visual examination), the cartridge case was immediately placed in a water bath for about two minutes. It was then removed to air-dry. All tests were done with fresh solutions (no solution was used twice except where indicated).

Results and Discussion

Results Involving the AFTE H_2O_2/HOAc Formula—The AFTE formula listed above as Reagent No. 2 (which uses 20 mL of 3% hydrogen peroxide and 52 mL water) is the same as the original AFTE formula (which uses 2 mL 30% hydrogen peroxide and 70 mL water). Using household 3% hydrogen peroxide was more convenient. When the gun blue overdeveloped brass cartridge cases were dipped in this solution, bubbles immediately began to appear around the cartridge case and the gun blue coating began to come off in small flakes. These bubbles result from the decomposition of hydrogen peroxide into oxygen and water. This process is discussed below. Gentle stirring assisted in the removal. The latent print eventually became apparent, but it was rather fragile and could be rubbed off. Upon removing and letting the cartridge case dry, the remaining gun blue coating hardened. In our case, the superglue pretreatment showed some improvement on the final result. The quality of the recovered print was poor and, therefore, overdevelopment should be avoided in the first place.

The Gun Blue Process—To understand what is going on, the gun blueing process has to be understood first. Based on discussions with chemists from the gun blueing industry, gun blueing solutions contain three principle (active) ingredients: an acid, a cupric salt, and selenious acid. Selenious acid is a powerful oxidizing agent that can oxidize metals such as copper, zinc, aluminum, and iron. Cupric ions are also oxidizing agents that can oxidize zinc, aluminum, and iron. When cupric ions and selenious acid are reduced by these metals, a blue-black Cu-Se coating is formed on their surface. Acid is needed since, among other things, the equation for the reduction of selenious acid requires acid. The Cu-Se coating occurs only where the metal is clean; it does not deposit well on surfaces with an oil or grease coating or with lipids from latent prints. Most gun blue solutions are made for steel; but there are also some for aluminum and some for brass, bronze, and copper. All contain the three basic ingredients.

In practice, a mild superglue (cyanoacrylate ester or CAE) treatment should be done first (2–4); if this does not reveal latent prints, then the dilute gun blue treatment should follow (see Experimental section). A 1:80 dilution is suggested. One should try to avoid overdevelopment by removing the cartridge case once the print becomes apparent. Overdevelopment may damage the latent print and may not be recovered by the gun blue removal process. Figure 1 shows a properly developed latent print on a cartridge case.

Some have observed that the gun blue coating process continues even after the cartridge case is gently rinsed and dried. This may be due to using a gun blue solution that is too concentrated. The use of a basic bath made of sodium bicarbonate has been suggested (5) to stop the process. The BKA, who originally introduced the gun blue idea, applied a lacquer spray (private communication from Don Coffey, US Army Crime Laboratory, Frankfurt, Germany). The Dutch have also used a lacquer spray (private communication from Anton Theeuwen and Josita Limborgh, Netherlands Ministry of Justice, Forensic Science Laboratory) as well as the Irish (6). A lacquer spray sometimes has the advantage of giving the developed cartridge case a "wet look" which often enhances the contrast of the developed print.

For a discussion of a rather early work on the use of a 3% silver nitrate solution to reveal latent prints on brass slugs (used to simulate coins), see the work of Reed (7). This solution works because silver ions oxidize (etch) copper, zinc, aluminum, and iron into solution and replace them with silver metal. Gun blue solutions oxidize (etch) and deposit a (blue-black) Cu-Se coating while silver nitrate solutions oxidize (etch) metals and deposit a (gray-black) silver metal coating. Both can reveal prints on metal cartridge cases.

An Explanation for the Removal of the Gun Blue Coating—The reason acidified hydrogen peroxide formula worked to remove the gun blue coating may be based on the fact that it is such a powerful

FIG. 1—*Gun blue-treated brass cartridge case with a fresh sebaceous print on it and mildly superglued.*

oxidizing agent that it can oxidize the components of the gun blue coating, changing them to soluble species. To see this, one needs to look at the electrochemical redox (oxidation-reduction) properties of acidified hydrogen peroxide and the copper-selenium (Cu-Se) coating imparted by the gun blue solution. The oxidation and reduction half-reactions involved and their corresponding standard potentials E° are (8):

	half-reaction	E°	
reduction:			
	$H_2O_2 + 2H^+ + 2e^- = 2H_2O$	1.776 volts	(1)
oxidation:			
	$Se + 3H_2O = 4H^+ + H_2SeO_3 + 4e^-$	-0.7400 volts	(2)
oxidation:			
	$Cu = Cu^{++} + 2e^-$	-0.3419 volts	(3)

Assuming that the Cu-Se coating consists of elemental copper (Cu) and selenium (Se) in a 1:1 ratio and each element can be independently oxidized, then the overall reaction to remove the Cu-Se coating using acidified hydrogen peroxide is:

	ΔE°	
$2H_2O_2 + Se = H_2SeO_3 + H_2O$	1.036 volts	(4a)
$H_2O_2 + 2H^+ + Cu = Cu^{++} + 2H_2O$	1.434 volts	(4b)
$3H_2O_2 + 2H^+ + \text{Cu-Se} = H_2SeO_3 + Cu^{++} + 3H_2O$	1.169 volts	(4c)

The net standard potential of $\Delta E^\circ = 1.169$ volts for this overall reaction is based on two factors: (1) the assumption that Cu and Se in the 1:1 Cu-Se coating are oxidized independently as if they were two separate elements and (2) the fact that the number of electrons involved in reactions 4a, 4b, and 4c are 4, 2, and 6, respectively. Therefore, the net potential is a weighted sum of the two potentials:

$$1.169 = (4/6) \times 1.036 + (2/6) \times 1.434 \text{ volts} \tag{4d}$$

The rather high reduction potential value (1.776 volts) of the first reaction (eq. 1) indicates that acidified hydrogen peroxide is a powerful oxidizing agent. Since the potentials for the overall oxidation of Se, Cu, and Cu-Se (reactions 4a, 4b, and 4c, respectively) are positive, acidified hydrogen peroxide, therefore, "can potentially" send elemental Se (eq. 2) and Cu (eq. 3) back into solution as selenious acid and cupric ions, respectively. The qualifier "can potentially" means that the reaction is thermodynamically favorable, but will proceed only if the activation energy is low (i.e., kinetically favorable). The fact that acidified hydrogen peroxide removes the gun blue Cu-Se coating indicates that this thermodynamically favorable reaction appears to be kinetically favorable. Note that according to eq. 4a, the oxidation of Se in the Cu-Se coating does not involve an acid, but according to the half-reactions (eqns. 1 and 2), it is needed. Acid, therefore, acts as a catalyst in this reaction. If Cu-Se is the insoluble cupric selenide CuSe, then the acidified hydrogen peroxide is such a strong oxidizing agent that it apparently dislodges the selenide ion (Se^{2-}) from CuSe and oxidizes it to elemental selenium (Se) [$E^\circ(Se^{2-}, Se) = 0.92V$] and further to selenious acid. It also oxidizes any deposited elemental copper (Cu) back into solution as cupric ions (Cu^{++}). Thus, ΔE° in equation 4c would still be positive.

Effect of the Gun Blue Coating on Toolmarks—Returning to the original question of examining the toolmarks on the fired, this study indicates that the gun blue treatment, if done to overdevelopment, will cover toolmarks. Since gun blueing is an oxidation process (as well as a deposition process), it actually etches the metal and replaces what it etches with the Cu-Se coating. Therefore, it can potentially destroy a weak toolmark. Our findings support the claim made by Bentsen, et al. (6) that, in most cases, the gun blue treatment, if not done to overdevelopment, does not affect toolmarks.

Derivation of an H_2O_2/HOAc Optimized Formula (Reagent No. 3)—The AFTE gun blue removing formula described above was developed for the purpose of removing lead residue from copper-jacketed bullets. As an oxidizing solution for removing gun blue coatings, it is not quite optimized; that is, it does not meet the requirement of eq. 4c that three molecules of hydrogen peroxide are needed for every two hydrogen ions (H^+) to oxidize the assumed 1:1 Cu-Se coating. To see how to obtain a proper mixture, one has to express all concentrations in moles/liter (molar concentration, M). Since the molecular weight of acetic acid (HOAc) is 60 g/mole, and that of hydrogen peroxide (H_2O_2) is 34 g/mole, the molar concentration of the AFTE formula is 2.03 M in acetic acid and 0.215 M in hydrogen peroxide. Here we used the fact that the percent concentration of hydrogen peroxide is weight:volume and the density of glacial acetic acid is about 1. There are about ten times more HOAc molecules than there are of H_2O_2. Because the gun blue removal equation (eq. 4c) requires three H_2O_2 for every two H^+ (from HOAc), an optimal formulation should reflect this. An optimized formula which uses only household vinegar and household hydrogen peroxide is Reagent No. 3:

14.1 mL 5% HOAc (household 5% vinegar)
20 mL 3% H_2O_2 (household 3% hydrogen peroxide)
34.1 mL total volume

This solution is 0.345 M in HOAc and 0.516 M in H_2O_2 and, therefore, there are three moles of H_2O_2 for every two of HOAc. Different total volumes of this formula can be made as long as the volume ratio of vinegar to hydrogen peroxide remains at $(14.1/20) = 0.706$.

Since the atomic weights of copper and selenium are 64 and 79 g/mole, respectively, the "molecular" weight of the "1:1 independent" Cu-Se is 143 g/mole. The number of moles of Cu-Se that the above reagent (No. 3) reacts with completely is 0.172 M (half of [HOAc]). Thus, the above solution should be able to remove $(143)(0.172 \text{ M})(34.1 \text{ mL}) = 839$ mg of Cu-Se. This assumes that the hydrogen peroxide only enters into this reaction and none decomposes to oxygen and water (see discussion below).

Using a weak acid like acetic acid tends to slow down the reaction since not all protons from the acid are available to react (collide) at any one time. As the reaction proceeds, the undissociated molecules release more protons until they are all used up in the reaction. The protons from a strong acid, on the other hand, are all available at the beginning of the reaction and get consumed as the reaction proceeds. To test the effect of using a strong acid, we replaced the acetic acid with an equivalent amount of a strong acid (hydrochloric acid).

Derivation of an H_2O_2/HCl Optimized Formula (Reagent No. 4)—Since a 5% HOAc solution is 0.833 M, the 14.1 mL that are used in optimized Reagent No. 3 contain (14.1 mL) × (0.833 M) = 11.8 mmoles of HOAc. Therefore, using 1.2 mL of 10.2 M HCl (concentrated) retains the 11.8 mmoles and adding 13 mL water retains the molarity (0.345 M). Reagent No. 4, therefore, replaces 14.1 mL HOAc with 1.1 mL 10.7 M HCl and 13 mL water. It is 0.345 M in HCl (and therefore in H^+) and 0.516 M in HOAc.

Results Involving the Two Optimized Formulas (Reagent No. 3 and 4)—Brass cartridge cases overdeveloped with gun blue and dipped in the optimized formula involving HOAc (Reagent No. 3) produced the same results as those using the AFTE (Reagent No. 2) but at a faster rate. The optimized formula involving HCl (Reagent No. 4) worked too quickly, stripping the cartridge case clean including the sebaceous print that was originally placed on it. Thus it appears that acetic acid is the preferred choice since it tends to slow down the reaction. Figure 2 shows a mildly superglued brass cartridge case overdeveloped with gun blue after treatment with Reagent No. 3. The cartridge case should be removed once the print becomes visible. This is because, as the solution removes the gun blue coating, it becomes weaker and contaminated with selenious acid and cupric ions (from the gun blue coating) which can re-deposit as selenium and copper.

Acidified Hydrogen Peroxide as an Alternate Latent Print Visualizer—A rather important after-the-fact thought arose during the gun blue removal studies. This was that acidified hydrogen peroxide should also oxidize (clean, non-lipid containing areas of) cartridge cases made of: brass—these consist of copper (Cu) (60–90%) and zinc (Zn) (10–40%) metals; nickel-coated brass—these consist of a nickel (Ni) metal coat on brass but may also have a nickel oxide (NiO) film due to air oxidation; and aluminum—these consist of aluminum (Al) metal but may also have an aluminum oxide (Al_2O_3) film due to air oxidation.

This is because the standard potentials (8) for the oxidation of these metals are positive as shown below (with lead (Pb) and iron (Fe) included):

half-reaction	E°	
oxidation: $Zn = Zn^{++} + 2e^-$	0.7618 volts	(5)
oxidation: $Ni = Ni^{++} + 2e^-$	0.257 volts	(6)
oxidation: $Al = Al^{+++} + 3e^-$	1.662 volts	(7)
oxidation: $Fe = Fe^{++} + 2e^-$	0.447 volts	(8)
oxidation: $Pb = Pb^{++} + 2e^-$	0.1262 volts	(9)

The oxidation of iron (Fe) is included for those lacquered steel cartridge cases which respond (though rarely) to a gun blue treatment. The oxidation of lead (Pb) is included since it was the target of the original formula.

Consequently, acidified hydrogen peroxide should (a) remove lead (Pb) as it was intended to do (1); but also, and more importantly, (b) "develop" latent prints on cartridge cases by a "cleaning" process which cleans the cartridge case everywhere except for areas containing sebaceous latent prints (and areas covered with oil, grease, or other oxidation-resistant substances). This second point is offered as an alternative to other "cleaning" solutions such as one recently reported by the Israel National Police (private communication from Sarena Wiesner, Israel National Police, Jerusalem, Israel) probably involving chromic acid (made by adding concentrated sulfuric acid to sodium dichromate). Such oxidizing solutions (which do not deposit metals) clean by an etching process.

The desired ratio of components of a solution for use to oxidize a cartridge case or to remove lead residue from bullets is $[H_2O_2]:[HOAc] = 1:2$. Thus the optimized formulas (No. 3 and 4) would work, but there would be an excess of H_2O_2.

Results Involving Reagents 3 and 4 as Etching Reagents—Tests were conducted for each of the three types of cartridge cases using the two reagents (No. 3 and 4). Prints were "developed" only on the brass and aluminum cartridge cases. Within seconds, the solution cleaned the cartridge case everywhere except where the prints (or other oxidation-resistant materials) were. As expected, this process was faster with the HCl-based reagent (No. 4) but there was also a tendency to remove the sebaceous print placed on it. Figure 3

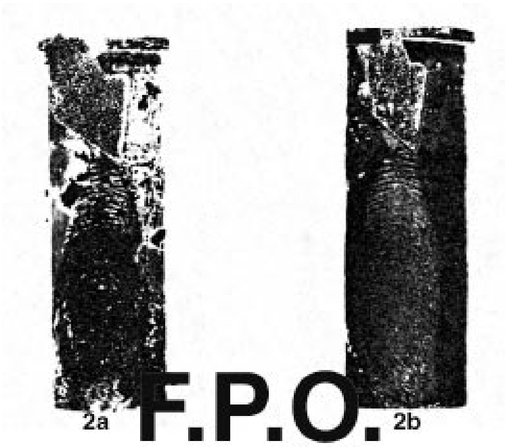

FIG. 2—*Brass cartridge case with a fresh sebaceous print on it, mildly superglued, and overdeveloped with gun blue (a) before and (b) after treatment with Reagent No. 3.*

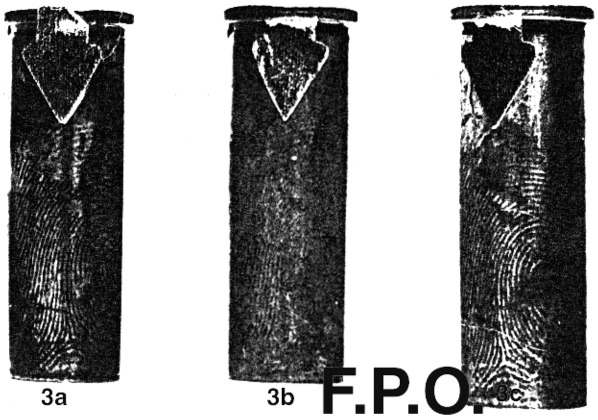

FIG. 3—*Sequence of treatments of a brass cartridge case with a fresh sebaceous print on it after mild super gluing, (a) after treatment with Reagent No. 3, (b) after diluted gun blue treatment, and (c) after a light lacquer treatment.*

(a) shows a mildly superglued brass cartridge case with a sebaceous print on it after treatment with Reagent No. 3.

A brass cartridge case dipped in either of the two solutions (No. 3 or 4) releases cupric and zinc ions into the solution giving it a faint blue-green color. Such solutions become weaker and contaminated with cupric and zinc ions. Therefore, the cartridge case should be removed as soon as the print becomes visible since there would be a tendency for the cupric ions to redeposit as copper. This is due to the solution becoming weak and not able to prevent the reduction of cupric ions by the zinc in the brass. Such contaminated solutions should also not be used on other brass, nickel-coated brass, or aluminum cartridge cases. For example, if a second brass cartridge case is placed in the contaminated solution, some of the copper ions in solution can deposit on the second cartridge case everywhere except where the prints (or other lipid materials) are. If an aluminum cartridge case is dipped in this solution, copper and zinc deposit on it giving it a faint blue-green patina everywhere except where the prints are. This tendency for copper to redeposit is why it is not recommended to keep a cartridge case too long in a "cleaning" type solution or to dip more than one cartridge case in it (unless one wants a metal-etching/copper-deposition process).

An advantage to using "cleaning" type solutions is that, if carefully monitored, they do not deposit a coating like gun blue or silver nitrate solutions. In some cases, however, the coating color may be needed to enhance the contrast of a weakly developed print. In such cases, one can keep it in the solution longer or dip the "cleaned" cartridge case in the dilute gun blue solution until the necessary contrast results (do not overdevelop) and then wash it in water and air-dry it. See Fig. 3(c). In our case, a light lacquer spray enhanced the contrast and stabilized the print. See Fig. 3(d). On several cartridge cases prepared with fresh prints (with and without supergluing), the etching process worked without destroying (and colorizing) the deposited prints. Consequently, in our case at least, the etching process of the first treatment and the (further) etching and simultaneous coating process of the second treatment apparently did not affect the deposited print. The strength of each solution (the etching solution and the gun blueing solution) and the time of treatment (judged by carefully viewing the process) can vary. This promising dual treatment clearly deserves further testing.

Comments on the Decomposition of Hydrogen Peroxide—Hydrogen peroxide (without acid) can decompose as follows:

$$2H_2O_2 = 2H_2O + O_2 \qquad (10)$$

This is a thermodynamically feasible auto-redox reaction since the oxidation and reduction components give a positive potential for the overall reaction:

$$\begin{array}{lll} & E^\circ & \\ H_2O_2 + 2H^+ + 2e^- = 2H_2O & 1.776 \text{ volts} & (11) \\ \underline{H_2O_2 = 2H^+ + O_2 + 2e^-} & 0.69 \text{ volts} & (12) \\ 2H_2O_2 = 2H_2O + O_2 & 1.086 \text{ volts} & (13) \end{array}$$

The overall reaction is rather slow and is even halted by the addition of acid. (Acid reduces the production of O_2 in the oxidation of H_2O_2). However, several materials can promote the production of O_2 from neutral H_2O_2. Such materials include gold dust, platinum, manganese dioxide, silver, hydrogen bromide, and lead. These materials act as catalysts since they remain unchanged. In acidic hydrogen peroxide, metals like silver and lead also get consumed along with the production of oxygen bubbles.

Thus, besides etching certain metals, acidified hydrogen peroxide can also decompose producing oxygen bubbles. Stirring removes bubbles that may hinder the etching process. For the concentrations considered here, the decomposition of hydrogen peroxide does not significantly deplete the amount needed to do its etching action.

Conclusion

Acidified hydrogen peroxide has not only been shown to remove excess gun blue from metal cartridge cases that have been overdeveloped by the gun blue treatment, but also to visualize sebaceous latent prints placed on post-fired metal cartridge cases by an etching (oxidizing) "cleaning" process. Subsequent gun blue treatment of such "cleaned" cartridge cases may enhance the contrast of the developed (etched) print.

Acknowledgments

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