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# MOISTURE INDICATING LACQUER FOR 20 mm AMMUNITION

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Philadelphia, Pennsylvania

June 1973

MEMORANDUM REPORT M73-20-1

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by

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**DEPARTMENT OF THE ARMY**
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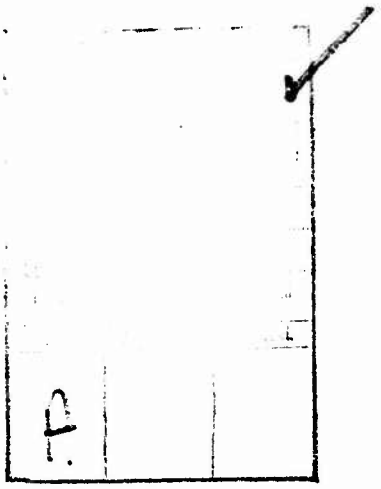

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13. ABSTRACT

Hangfire problems encountered in M61 gun installations on USAF aircraft dictated the need for a moisture-indicating system which could be used to screen 20 mm ammunition. This report describes the development of such a system utilizing a color-producing reaction between ferric ammonium citrate and gallic acid. When formulated into a suitable lacquer system and applied to a cartridge case, water contamination was indicated by a color change from gray to black.

Several other concepts for water-indicating systems were tested and found unacceptable.

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AMCMS Code: 4810.16.0338-6.02
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Munitions Development & Engineering Directorate
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## ABSTRACT

Hangfire problems encountered in M61 gun installations on USAF aircraft dictated the need for a moisture-indicating system which could be used to screen 20 mm ammunition. This report describes the development of such a system utilizing a color-producing reaction between ferric ammonium citrate and gallic acid. When formulated into a suitable lacquer system and applied to a cartridge case, water contamination was indicated by a color change from gray to black.

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## BACKGROUND

A letter written by AMSMU-QA-MR, U.S. Army Munitions Command, dated 20 July 1971, indicated that the 20 mm hangfire problem experienced in certain M61 gun installations in USAF aircraft appeared to result from moisture contamination of the primer and/or propellant. Further, letter from AMSMU-RE-CA, U.S. Army Munitions Command, dated 20 July 1971, included a summary of an explanation of this phenomenon offered by Lake City Army Ammunition Plant (LCAAP). A brief description of the LCAAP explanation follows.

LCAAP indicated that certain M61 gun installations in USAF aircraft generated a sizable quantity of unfired rounds which had been cycled through the gun. The waterproofing seal was broken at the cartridge case/projectile junction in an unknown number of these rounds by forces applied to the projectile during weapon cycling.

An unknown number of down-loaded rounds were subjected to improper handling, resulting in exposure to liquid water. Those rounds with broken waterproofing seals then suffered moisture contamination of the primer and/or propellant. In subsequent use of these water-contaminated rounds an unknown number of hangfires occurred. For these reasons, the Small Caliber Propellants and Pyrotechnics Division laboratory of the Munitions Development and Engineering Directorate suggested to Munitions Command that it may be possible to find an indicating mouth-waterproofing lacquer which would provide visual evidence of cartridge water contamination. This report describes the development of such a lacquer.

## APPROACH

Two basic approaches were employed to obtain the water-indicating lacquer. The first, a physical approach, made use of water soluble dyes which were incorporated into the mouth-waterproofing lacquer. It was expected that these dyes would dissolve in water after physical rupture of the film and cause the cartridge case to be stained, thus providing visual evidence of water contamination.

The second basic approach, a chemical method, employed a reaction requiring water for the formation of a colored product. The reactants were incorporated into various lacquer formulations and applied to several locations on the cartridge case. A second chemical approach involved the use of a pH indicator and an acid or base incorporated in the standard mouth-waterproofing lacquer. It was expected that, if the seal were broken and water contamination occurred, the indicator would change color. Each of these approaches will now be discussed in some detail under the appropriate section.

## Physical Approaches

A "suspended dye" approach was tried which explored the feasibility of employing a water soluble/organic solvent insoluble dye as the color-producing agent. The dye would be relatively colorless when suspended in the nitrocellulose lacquer but, if the film was broken and the lacquer exposed to water, the dye would dissolve producing a brightly colored solution and bleed out onto the cartridge case, thereby providing the required visual discernibility. The water-dye contact would be prevented by the nitrocellulose lacquer until the lacquer film was mechanically ruptured during handling or gun cycling.

The successful incorporation of a dye in the waterproofing lacquer required the use of a dye formulation which contained no chemicals that were known to aggravate barrel fouling or erosion. For this reason, organic dye samples were obtained (from DuPont Co.) which contained no halogens and were not diluted with salts. Diluents used were mostly sugars and, therefore, would decompose under gun conditions. The following dyes were tested for this application:

"Pontamine" Blue AX
Crocein Scarlet N
"Pontamine" Diazo Blue BR
"Pontamine" Brilliant Violet RN

The initial tests with the various dyes dispersed in nitrocellulose lacquer were conducted by painting glass slides with the lacquers. The slides were allowed to dry and then were exposed to water to

insure that no dye dissolved before the lacquer surface was broken. The lacquer surfaces were then cut with a knife blade to simulate a break in the waterproofing seal. Upon subsequent exposure to water, a color change was evidenced as the exposed dye particles in the cut edges dissolved into the water solvent.

The next experimental step was to attempt to achieve these results with the dyes suspended in the standard cartridge case mouth-waterproofing sealant, which is a black tar-like compound. The test procedure was identical; however, no coloration of the water was experienced as in the nitrocellulose lacquer tests. It was concluded that the negative results were caused by the fact that the dye particles were too well protected by the standard asphaltic mouth-waterproofing lacquer.

This result indicated that the dye could not function when present in the asphaltic lacquer. For this reason it was decided to coat the exterior of the case at the projectile case junction with a nitrocellulose lacquer containing a water soluble organic dye.

Several tests were then conducted on 20 mm cartridge cases and projectiles utilizing this nitrocellulose system. The case mouths were waterproofed in the usual manner and the projectiles were partially inserted. A bead of nitrocellulose lacquer with suspended dye was placed on the projectile circumference just below the rotating band, and then projectile insertion was completed. Excess material was wiped from the cartridge, and the cartridge assembly was allowed to dry.

Before fracturing the waterproofing seal the round was exposed to water and no coloration appeared. The seal was then mechanically fractured and again exposed to water. Some bleeding out of the dye occurred, but not enough dye was exposed to readily permit visual identification of the fracture. The test was repeated with increased percentages of dye in the indicating lacquer; however the amount of coloration remained small and the color that did appear was easily washed away by the water. Therefore this system did not meet the functional requirement of a permanent color change.

It was thought that the application of a thin band of absorbent material at the case/projectile juncture would absorb the dye, thereby effecting a permanent color change. Several coated paper strips

were applied to test cartridges; however they could not be sufficiently waterproofed themselves to retain their adhesion to the cartridge case. A test was then run with collodion as the absorbent thin band, and it also failed to remain on the cartridge case after fracture and water exposure.

In conclusion, the nitrocellulose lacquer with suspended dye indicator system was abandoned in favor of the following systems.

## Chemical Approaches

### The Acid-base Indicator System

This system was based on the ability of acid-base indicators to change color or take on a color in the presence of an appropriate acid or base in solution. The acid or base selected would have to be water soluble and exhibit a final pH within the indicator transition interval.

Innumerable water soluble indicators and acids and bases are available. Initial trials were made with citric acid incorporated directly into the nitrocellulose lacquer. Citric acid was chosen because it would not be likely to cause barrel fouling due to its organic composition. The test procedure was similar to that of the suspended dye approach with the exception that an acid indicator was dissolved into the water that was applied after the mechanical fracture of the waterproofing seal. A color change was evidenced; however, application of a large quantity of water washed the acid from the fractured seal area and no further color change resulted.

An attempt was made to employ an acid or base which was only moderately water soluble in order to prevent its washing away. Potassium bicarbonate was selected, and the same test procedure was employed. Again, the results were not satisfactory.

The fact that not enough acidity or basicity could be retained on the cartridge case and, also, that the color change was not permanent indicated that this was not an acceptable approach.

## Microencapsulation of Reactive Chemicals

Both this approach and the final one were conducted in conjunction with the Capsular Products Division of the National Cash Register Co. of Dayton, Ohio. In order to utilize a color-producing reaction as a moisture-indicating lacquer, it was necessary to microencapsulate one of the reactants and to incorporate the other reactant in an unencapsulated form into the lacquer formulation. This was done to prevent a water-induced reaction prior to physical rupture of the lacquer film. It was thought that mechanical rupture of the lacquer film would cause the microcapsules containing the reactant to rupture. Subsequent exposure to water (i.e., rain, fog, etc.) would provide the solvent medium for a permanent color-producing reaction, thereby providing the required visual indication of seal rupture and subsequent moisture contamination of the round.

The first system to be investigated involved octyl gallate and ammonium metavanadate. The octyl gallate (OG) was microencapsulated in gelatin, utilizing an acrylic resin and toluene solvent system. A lacquer was formulated containing the encapsulated OG and unencapsulated ammonium metavanadate (AMV) suspended in the nitrocellulose lacquer. Use of encapsulated OG successfully eliminated the possibility of prereaction during the lacquer formulation and application process.

Films of this lacquer formulation were mechanically broken and treated with water; however, no color change resulted. It was felt that this system failed because the film rupture did not produce a significant number of ruptured capsules; thus, only a small amount of OG was available for reaction with AMV and no detectable color change was produced. This property of microencapsulated reactants led to its abandonment as a satisfactory approach to the development of a water-indicating lacquer.

## The Ferric Ammonium Citrate-Gallic Acid (FAC-GA) System

This final and successful approach incorporated reactive ingredients as in the microencapsulation method, but the ingredients were blended directly into the binder without encapsulation. The two essential components were ferric ammonium citrate and gallic acid. These components react to produce ferric gallate, which is blue-black in color.

The binder composition played a key role in the success of this system. The binder had been formulated such that it was partially water soluble and partially organic solvent soluble. This property provided the following advantages. The reactive ingredients are only water soluble and, therefore, the system can be formulated and applied in an organic media without pre-reaction of the ingredients. The binder, however, remains permeable to water and, therefore, on exposure to water a reaction will occur. The system does not wash off the cartridge case because it is only partially water soluble; thus the permanence of the color change is insured.

Also, lateral transfer of water through the binder greatly increases the reaction site area, thus facilitating visual identification. As a result of this property, however, it became necessary to water-proof the indicator system. A topcoat of Saran resin was chosen for this function. The final formulations for the components of this system are given in the Appendix.

The indicator system may be applied to the cartridge case/projectile rotating band junction by spray coating, brushing, or roller coating, and it will remain stable for at least 5 days, thus providing sufficient time for application of the final Saran coating. The topcoat (Saran) may also be sprayed. Figure 1 shows two 20 mm rounds which have the FAC-GA coating before and after water contamination.

In order to obtain a workable system the total coating thickness was maintained at 0.005 inch. This value is within the chamber clearance tolerances on 20 mm Army weapons; therefore no interference difficulties are anticipated.

## CONCLUSIONS

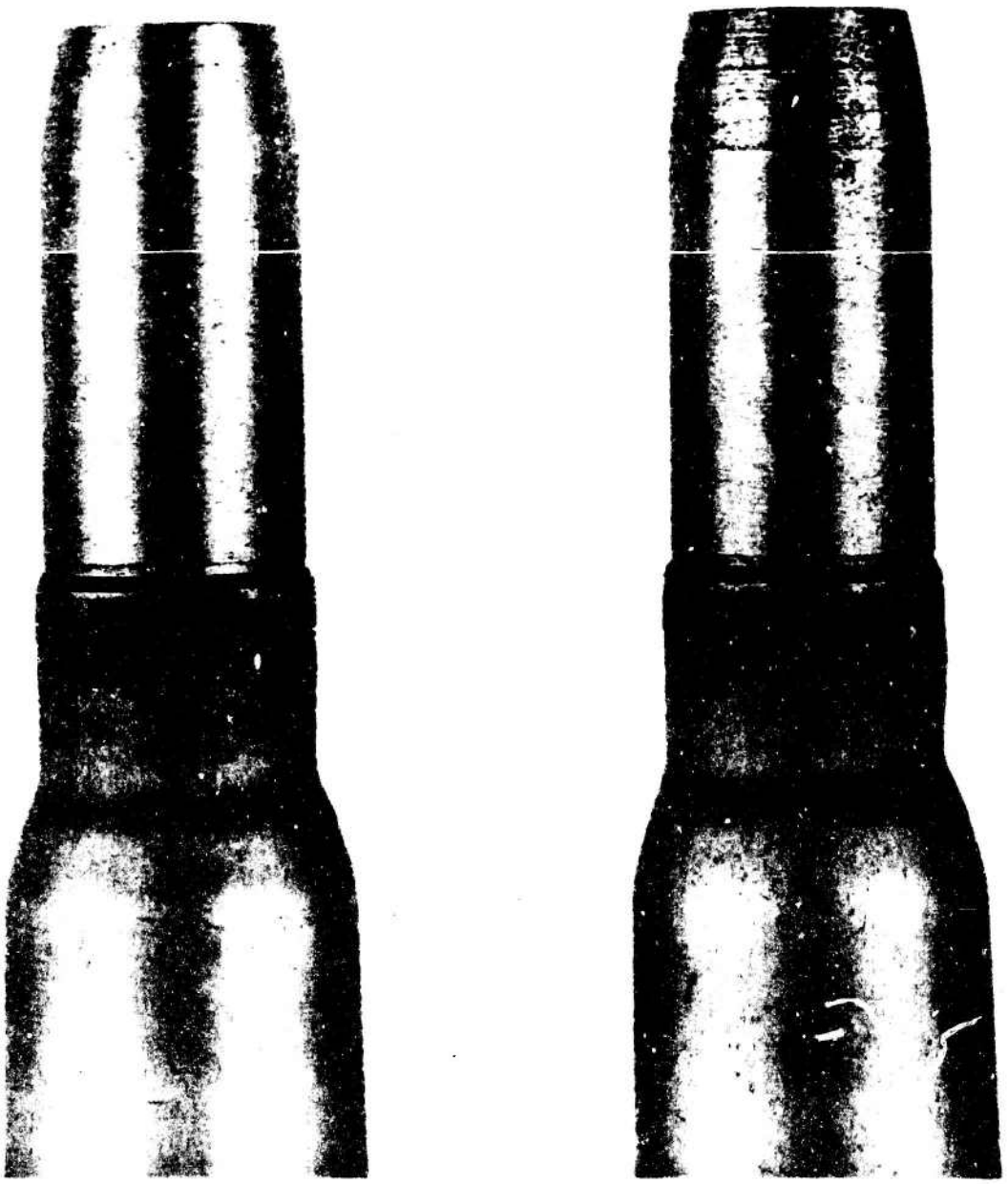
1. A water-indicating lacquer employing a colorimetric reaction between ferric ammonium citrate and gallic acid (FAC-GA) has been developed. This lacquer has been applied to cartridge cases, and initial testing has shown that a noticeable color change is produced if the seal is broken and water contamination occurs.

2. Use of lacquers containing water soluble dyes as a moisture-contamination indicator was unsatisfactory because no permanent color change could be obtained.

3. Use of a lacquer containing an acid or base in conjunction with a pH indicator as a moisture-contamination indicator was unsatisfactory because no permanent color change was produced.

## RECOMMENDATIONS

It is recommended that further testing be performed to determine the feasibility of using the FAC-GA lacquer on production ammunition and to establish that no detrimental ballistic or weapon effects occur because of its use.



A B

Figure 1. 20 mm Rounds with FAC-GA Coating
A - Before water contamination
B - After water contamination

## APPENDIX

## PREPARATION OF FAC-GA SYSTEM

The following is the final procedure for the preparation of a moisture-indicating lacquer, as prepared by the National Cash Register Company.

1. Formulation of Base Coating

a. Gallic Acid (GA) System

Prepare a 20% solids solution containing solids composed of 95% Dantoin Resin 684 and 5% Gallic Acid; e.g., 80 grams $H_2O$, 19 grams Dantoin Resin, 1 gram Gallic Acid.

Blend together 125 grams of Syloid 74 with 268 grams of the above 20% solution. This mixture will have the consistency of a stiff paste. It should be spread in a thin layer on a tray and allowed to remain until dry. After air drying, place in an oven at 100° C for 18 to 24 hours. Store in a moisture-proof container.

Shake the following in a "quickie" mill for six minutes:

10 grams GA system (above)
30 grams Carboset 525 (20% in MIBK)
10 grams MIBK

and store in a sealed container.

b. Ferric Ammonium Citrate (FAC) System

Blend together 130 grams of Microcel C with 442 grams of 20% Ferric Ammonium Citrate Solution. This mixture will have the consistency of a stiff paste. Spread in a thin layer on a tray and allow to dry. After air drying, place in an oven at 100° C for 18 to 24 hours. Store in a moisture proof container.

Shake the folling in a "quickie" mill for six minutes:

10 grams FAC powder (above)
5 grams Purecal 0
15 grams 20% Carboset 525 in MIBK
25 grams MIBK.

Mill two batches to a total weight of 90 grams and add 90 grams of MIBK. Stir in a light-proof container for 48 hours. Store in a dark bottle

c. Preparation of Water-sensitive Coating for Spraying.

Mix the following:

10 grams of GA system
20 grams of the FAC system
5 grams of MIBK
14 grams of 20% Carboset 525 in MIBK

Blend in a Waring blender for several minutes; transfer to a jar and stir for four hours.

The mixture is now ready for use and should not be stored for more than 24 hours.

The system described above has been stored for up to five days without deterioration but, as a general rule, the 24-hour limit is reasonable for production purposes and lessens the probability of moisture contamination. This base coating (not to exceed 3 mils) must be thoroughly dry before a topcoat is applied.

A test of the coating grind or smoothness of the base coating should be made on a glass slide with an air brush. Rough coatings are a result of the following factors: insufficient grinding of solids, poor spray pattern, or high solids or viscous media. Uneven or rough base coatings will hinder the effectiveness of the topcoat.

2. Preparation of Topcoat

Prepare a 7% solution of Saran resin XD 2364.02 in a 1:1 solvent blend of toluene and MIBK.

Spray this coating on the base or water-sensitive coat in such a manner as to seal the surface and edges of the base coating. Several passes will be required, and complete drying should be achieved between coats.

If the viscosity of the Saran resin solution is too high, a reduction of solids by 1% will usually be adequate to achieve better spray characteristics.

Film thickness should be approximately 0.002 inch.