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**Pressure Sensitive Chemicals for Use in
Marking and Detection of Personnel (U)**

Robert A. Allen
ALLIED CHEMICAL CORPORATION

TECHNICAL REPORT AFATL-TR-67-180

OCTOBER 1967

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PRESSURE SENSITIVE CHEMICALS FOR USE IN
MARKING AND DETECTION OF PERSONNEL (U)

Robert A. Allen

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FOREWORD

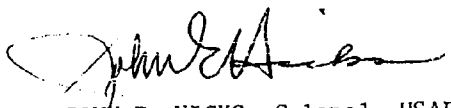
(U) This report is an Interim Report covering the period July 1966 - February 1967. It consolidates and summarizes the results of research and development efforts by the contractor, Allied Chemical Corporation, P.O. Box 1069, Buffalo, New York under United States Air Force Contract AF 08(635)-6100. The contracting agency was the Air Force Armament Laboratory, Research and Technology Division, Air Force Systems Command, Eglin Air Force Base, Florida. Assigned by AFATL (ATCB) as project engineers were Robert E. Mayo, Captain USAF and Frederick T. Dehner, Captain USAF. This report covers the joint research effort of the author and Mr. Elmore L. Bement.

(U) The contractor, hereby, gratefully acknowledges the help of Armstrong Cork Company of Lancaster, Pennsylvania who provided some of the chemical components.

(U) This report contains no classified information extracted from other classified documents.

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(U) Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



JOHN E. HICKS, Colonel, USAF
Chief, Bio-Chemical Division

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UNCLASSIFIED ABSTRACT

Research studies were conducted to develop a pressure sensitive personnel detection system. This work resulted in a unique chemical system and device which when actuated by pressure yields an audible response and has a marking capability. Demonstration was made at Eglin Air Force Base during February 1967 in cooperation with monitors of the Air Force Armament Laboratory AFATL (ATCB).

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SECTION I

INTRODUCTION

(C) The Air Force Armament Laboratory established the requirement for the development of an economical pressure sensitive chemical system to aid in the non-injurious detection of personnel movement. The immediate objective was development of a chemical system that when activated by pressure yields an audible response. The system when incorporated into a device of proper design would be sufficiently sensitive to be triggered by a pressure differential of not more than 100 pounds. A distinct audible response which could be detected by the unaided ear at a distance of at least 100 yds. was specified and the device would have to remain active under a wide variety of environmental conditions. Secondly, the device should show an eventual capability of emitting concurrently a marking compound to further identify the triggering agent.

(U) Fundamental work on this problem was conducted by the author and his associate during the period July 1966 - February 1967. Approximately four months were devoted to research with the balance of time utilized in development of specific devices and production of demonstration quantities.

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SECTION II

SUMMARY

1. (C) INTRODUCTION TO CHEMICAL SYSTEM

After an early investigation of a nitrogen tri-iodide chemical system which proved to lack stability, investigation of a more complex chemical system was undertaken. It was found that potassium permanganate doped with sodium peroxide and a desiccating agent (molecular sieve) and properly partitioned from glycerine and/or ethylene glycol yielded a relatively stable heat generating system which could be pressure activated. It was further found that an organic diazo oxide derived from 4-nitro-2-aminophenol could be isolated to the dry powder state with reasonable stability and safety. This diazo oxide is a deflagrant, burning rapidly without emitting noise when heated in its unconfined state, but reacts to heat with explosive violence (emits loud noise) when confined. These factors when properly combined led to the development of an audible detection device meeting the objectives of this initial investigation.

2. (C) PHYSICAL DESCRIPTION

A device of particular design and labelled Device D was the focal point of a demonstration at Eglin Air Force Base during February 1967. Briefly, the device can be described as a two inch long, thin wall, polyethylene tube, one quarter inch in diameter and heat sealed flat at each end. Internally the tube contained a powder-bead mix at each end. In the center of the tube was placed a No. 5 pharmaceutical gelatin capsule which previously has been filled with the diazo oxide. The powder was a dry mixture of potassium permanganate-sodium peroxide - No. 4A molecular sieve¹ in the weight ratio 95:5:1. The beads² were random 2,000-3,300 micron wax spheres (micro-capsules) containing a 50:50 weight ratio of glycerine-ethylene glycol solution. To enhance the audible response and for demonstration purposes the external polyethylene wall of the device was reinforced in the area of the diazo capsule site using one turn of common office transparent adhesive tape. Figure 1 shows the external configuration of Device D and its internal components.

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- 1 Supplied by Union Carbide Corporation, Linde Division.
 - 2 Supplied by the Research and Development Center, Armstrong Cork Company, Lancaster, Pa.

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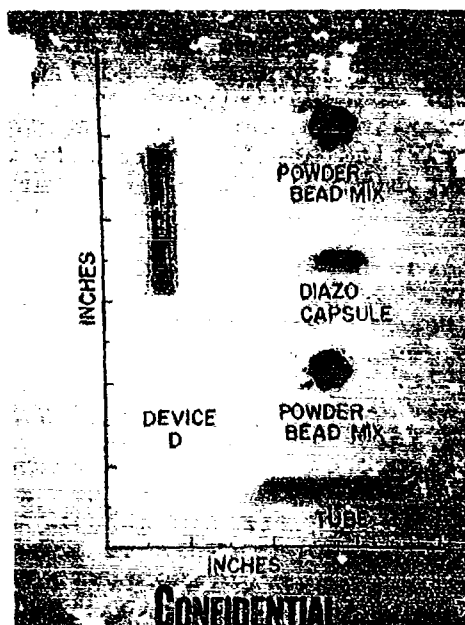


Figure 1. Assembled Device D and Components

3. (C)CHEMICAL ACTION

Device D is activated when the wax beads are crushed. The glycerine-glycol exudes onto the surfaces of the permanganate-peroxide crystals where in the presence of minute traces of water from the pores of the molecular sieve a rapid oxidation takes place. The heat generated by this oxidation is propagated to the gelatin wall of the diazo capsule. The diazo oxide explodes when the temperature of the confining wall exceeds 125-130°C.

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4. (C) (U) PROPERTIES OF THE DEVICE

(C) Under controlled laboratory conditions, Device D has an audible response in excess of 100 yards, is capable of being triggered by a pressure differential of less than 100 pounds, and at temperatures above 70°F yields an audible response in less than 3 seconds. It was also shown to be capable of trigger by normal foot pressure on mowed lawn and some field grass conditions. Of secondary importance, feasibility for dissemination of a marking compound was demonstrated. Other noteworthy characteristics are its stability when immersed in water, camouflage capability, and its oven storage stability at 50°C.

(C) To further evaluate the capability of Device D, demonstrations under random environmental conditions were conducted at Eglin Air Force Base. Temperatures ranged from 40°F to about 70°F but were generally in the 50°-60° range. The audio demonstrations revealed the sound could be detected in open areas at distances of at least one half mile without detection aid and at least 500 yards in forest cover. Both performances were made in the presence of aircraft background noise. With the ambient temperatures generally well below 70°F, the triggering times for Devices D were erratic ranging from roughly one second to more than one minute. This random action was attributed to both temperature and non-uniformity in assembly of the device. In another demonstration simulating aircraft dissemination, the devices were thrown from a 285 foot tower onto packed dirt road and sandy terrain. Less than 3% were activated at impact. Demonstrations of pressure sensitivity showed the devices to be adequately sensitive on hard surfaces but were not sensitive to normal foot pressures in the sand, grass and some dirt road conditions at Eglin. In the soft terrain, the foot was unable to exert sufficient unit pressure to crush the glycerine-glycol beads.

(U) The results of the marking compound demonstration were less than desired. These devices consisted of a Device D placed in a three quarter inch by two and one-half inch flexible polyethylene bag together with a color precursor powder and heat sealed. The explosion burst the bag raising a cloud of powder. The powder was converted to deep blue color on the target by spraying the target with vinegar. It was found that the powder was too thinly dispersed at distances beyond three or four feet to adequately mark a target. This fact together with the excessive trigger times that permitted a person to escape beyond this distance before the dissemination could occur resulted in an unsuccessful demonstration.

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SECTION III

DISCUSSION

1. (C) NITROGEN TRI-IODIDE SYSTEM

Nitrogen tri-iodide has been a laboratory curiosity for many years and many references to its instability have been reported in the literature. It is a very sensitive, explosive compound that can be triggered by extremely small pressures. It needs no auxiliary triggering agents such as heat generators.

A brief study of the nitrogen tri-iodide compound, $\text{NH}_3 \cdot \text{NI}_3$, was made. An attempt to incorporate it into a gelatin-gum arabic microcapsule was unsuccessful. The $\text{NH}_3 \cdot \text{NI}_3$ slowly decomposed and interfered with the coacervation (wall-forming) step of the encapsulation process.

As an alternate solution to this containment problem, a small quantity of partially dried $\text{NH}_3 \cdot \text{NI}_3$ mass was inserted into the bottom of a No. 5 pharmaceutical gelatin capsule, the capsule closed, and then allowed to dry under ambient laboratory conditions. After drying, the capsule was stepped on using the normal walking pressure of the foot. There was an instantaneous explosion and loud audible report. Devices of this type were then placed in storage only to find that within three or four days the $\text{NH}_3 \cdot \text{NI}_3$ slowly deteriorated into a completely inactive state. Attempts to prolong its activity by introducing molecular sieves into the capsule showed only a nominal increase in the shelf life. Storing the capsules in a refrigerator did prolong the active life appreciably but this was considered an impractical solution. Further investigation along this tack was abandoned.

2. (C) EXPLOSIVE COMPOUNDS

Concurrently with the $\text{NH}_3 \cdot \text{NI}_3$ study, an investigation of the deflagration and/or explosive properties of organic diazo compounds was initiated. Listed in Table I are nineteen compounds that were synthesized in the laboratory; the initial objective being to obtain a crystalline compound that could be microencapsulated and exploded when triggered.

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TABLE I. (C) DIAZO COMPOUNDS

Compounds that were converted to diazo oxides or salts and studied in the laboratory:

2-amino-4-(methylsulfamyl)-phenol
2-amino-3,4,6-trichlorophenol
2-amino-4-chlorophenol
2-amino-4-sulfophenol
2-amino-4-chlorophenol-6-sulfonic acid
2-amino-4-nitro-6-sulfophenol
2-amino-5-nitrophenol
2-amino-4-chloro-5-nitrophenol
4-nitro-2-aminophenol
4-amino-2-nitrophenol
sulfanilic acid
2-aminophenol
1-amino-2-hydroxy-6-nitronaphthalene-4-sulfonic acid
4,6-dinitro-2-aminophenol
aniline
para-nitroaniline
ortho-nitroaniline
para-nitroaniline-ortho-sulfonic acid
para-aminosalicylic acid

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Of the compounds listed in Table I only twelve could be isolated readily to dry powder form. These twelve diazo oxides or inner salts were tested for activity by heating small quantities on a hot plate and/or striking on an anvil with a moderate hammer blow. All of the compounds were less pressure sensitive than could be tolerated in the visualized device. However, from these tests, six showed sufficient activity to be given further consideration. These six were the diazo oxides of:

2-amino-4-methyl-sulfamylphenol (MP 115°C)
5-nitro-2-aminophenol (MP ca. 120°C - exploded in MP apparatus)
4-chloro-5-nitro-2-aminophenol (MP - unknown)
4-nitro-2-aminophenol (MP ca. 128°C - exploded in MP apparatus)
4-amino-2-nitrophenol (MP 162°C)
4,6-dinitro-2-aminophenol (MP 163°C)

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The activity of the compounds when subjected to melting point tests further influenced the choice among these compounds. For example, a milligram quantity of the 5-nitro-2-aminophenol diazo oxide completely shattered the melting point apparatus. It was considered too brisant and is reported to be fickle.

Based on the overall activity characteristics of these compounds and the contractors knowledge of their economics, the diazo oxide of 4-nitro-2-aminophenol was chosen as the explosive agent. A laboratory procedure for preparation of the material is given in Table II.

TABLE II. (C) PROCEDURE FOR LABORATORY SYNTHESIS
OF DIAZO OXIDE OF 4-NITRO-2-AMINOPHENOL

Into a 400 ml. beaker, fitted with an A-type agitator, and immersed in an ice bath, is added 100 ml. of cold water and 15.4 gms. (0.1 mole) of 4-nitro-2-aminophenol. The mixture is agitated to a uniform slurry. When thoroughly mixed, 30 ml. of 20° Be' hydrochloric acid is slowly added. Crushed ice is added directly to the mass to reduce the temperature to 0°C. Then 7.3 gms. (0.106 mole) of dry, powdered sodium nitrite is added rapidly in a manner to insure immediate immersion and minimum loss of nitrous oxide. A bright yellow precipitate is formed. The reaction mass is tested to insure the pH is acid to Congo red test paper and that a test on starch iodide paper shows an excess of nitrous acid. Agitation is continued for at least one-half hour with the temperature kept at 0-5°C. The mass is filtered on a vacuum filter consisting of a Buchner funnel and vacuum flask connected to a vacuum source. Care is taken to prevent excessive suck-down and cracking of the filter cake. The cake is washed with 400 ml. of water followed by 50 ml. of benzene denatured ethyl alcohol. The alcohol-wet filter cake is then placed in an oven to dry at 40-50°C. Precaution is taken to avoid dusting the dry diazo oxide product.

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Attempts to microencapsulate the diazo oxide were unsuccessful. The oxide appeared to react with the wall material rendering the oxide inactive and preventing proper wall formation of the capsule. Attention then turned to placing the diazo oxide powder into standard pharmaceutical, two-piece gelatin capsules and observing the reaction to heat. Loud audible reports were produced. Since none of the diazo oxides produced audible responses when ignited in the unconfined state, they are considered deflagrating agents. The audio response is the result of rapid pressure buildup and sudden fracture of the confining walls.

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3. (C) POTASSIUM PERMANGANATE - GLYCERINE SYSTEM (DEVICE A)

Having settled on a particular, heat sensitive diazo compound, attention turned to heat generating systems and mock-up of pressure sensitive detection devices. As a starting point, the well known potassium permanganate-glycerine system was chosen. When brought into intimate contact, these two compounds autoignite within eight seconds at temperatures above 70°F. The timing of ignition can be correlated with the ambient temperature. The lower the temperature, the longer the ignition time.

A device (Device A) was assembled by placing approximately one gram of permanganate grains in a one inch square polyethylene bag and adding a cellulose acetate (CA) two piece capsule³ containing five drops of glycerine. This partitioned the glycerine from the permanganate. Then a No. 4 gelatin capsule packed with the diazo powder was introduced into the bag and the bag heat sealed to complete the device. Foot pressure applied to the device crushed the large CA capsule releasing the glycerine onto the permanganate surface. Auto-ignition ensued and the heat triggered the explosion of the confined diazo. A loud audio report resulted (usually within 10-15 seconds).

This device was generally reliable and reasonably stable. However, the triggering time is considered too slow and the rigidity of the CA capsule renders the device less sensitive than desired. Further, the CA capsule walls are subject to manufacturing imperfections and must be carefully inspected before using.

4. (C) PERMANGANATE - ALCOHOL - SULFURIC ACID SYSTEM (DEVICE B)

Potassium permanganate wetted with ethyl alcohol auto-ignites instantaneously when brought in contact with concentrated sulfuric acid. From these components a Device B was assembled similar to Device A. The bag contained the permanganate wetted with several drops of ethyl alcohol, a small thin-walled glass vial containing 3-4 drops of 100% sulfuric acid, and a gelatin diazo capsule.

3 A 1/4 inch diameter x 3/8 inch long cellulose acetate capsule, similar to a gelatin capsule and obtained through Unipeg Incorporated of Rockville, Maryland.

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Again this device was pressure sensitive to normal foot pressure acting on a hard surface. The audible report was loud and instantaneous. Among the factors that precluded the device from further study were: lack of sufficient pressure sensitivity under soft surface conditions; personnel hazards from spewing concentrated acid; and eventual loss of activity due to evaporation and vapor transmission of the alcohol through the walls of the polyethylene bag by ambient forces.

5. (C) SODIUM PEROXIDE - WATER - GLYCERINE SYSTEM (DEVICE C)

Essentially an instantaneous autoignition occurs when sodium peroxide is brought in contact with glycerine and traces of water. Paradoxically sodium peroxide will slowly inactivate under humid environment. Similar to Device A, Device C was assembled by replacing the permanganate with sodium peroxide and adding a cellulose acetate capsule containing a 50:50 weight solution of glycerine water and a gelatin diazo capsule.

Generally the triggering time of this device is less than three seconds and has a sensitivity similar to Device A. The resistance to water vapor transmission through the closure area of the CA capsule is inadequate and results in fairly rapid inactivation of the trigger system under warm humid storage conditions.

6. (C) DEVICE D

The physical make-up and chemical action of Device D was explained in Section II. Its design is the outgrowth of attempts to rectify the deficiencies of Devices A and C.

Search was made for economical polyhydroxy compounds other than glycerine that would react more rapidly with potassium permanganate. From observations, glycerine tends to resist wetting the surface of the permanganate and secondly, the viscosity of glycerine increases very rapidly with a decrease in temperature. Similar observations were made of the sodium peroxide-glycerine system. Among the common polyhydric alcohols and polyhydroxy compounds, ethylene glycol appeared best able to aid or replace the glycerine in the triggering system. Various ratios of glycerine-ethylene glycol were tested and a 50:50 weight ratio was chosen.

Knowing the speed of the sodium peroxide-glycerine reaction, the stability of the permanganate-glycerine system, and the effect of the glycol addition, these factors were translated into a combination triggering system consisting of

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potassium permanganate-sodium peroxide-glycerine-ethylene glycol-trace water. The system worked. In the laboratory the speed of reaction was reduced to less than a 2 second auto-ignition. To insure that trace amounts of water were retained in the device at all times and that the water was "partitioned" from the peroxide, a small arbitrary amount of molecular sieve powder was added to the system. As a result the system remained stable to storage (50°C for 30 days) and active when triggered.

As noted in Section II, footnote 1, page 2, the glycerine-ethylene glycol microcapsules or beads were obtained from Armstrong Cork Co. They stated they are a licensee of the Southwest Research Institute technique. Armstrong Cork, tailored the beads to the requirements of this device. The beads consist of a wax shell melting above 55°C. The payload or weight of the glycerine-glycol inside the shell is approximately 65%. Armstrong's initial supply of these beads contained a minimum of leaky or defective beads. However, their second shipment had a high percentage of defects. Armstrong acknowledged the poorer performance and assured their future efforts would be equal to the initial shipment.

Attempt was made to obtain a second source of microcapsules from National Cash Register. Although their techniques are not readily adaptable to encapsulation of water miscible compounds, they did supply a research quantity of glycerine-glycol capsules made by an interfacial polymerization method. The capsules were unacceptable because of the high leakage and toughness of the shell.

An alternate device similar to Device D was assembled, replacing all the glycol-glycerine beads with 50:50 glycerine-water wax beads. This device worked equally well when stored at ambient laboratory conditions. However, it had a fickle characteristic of either exploding prematurely or becoming very slow reacting when stored at 50°C. This characteristic was attributed to water vapor passing through the wax capsule wall. When it passed rapidly enough it reacted with the peroxide to form sodium hydroxide, hydrogen peroxide and sufficient heat to melt the wax and release the glycerine. When it passed slowly its only effect was to deactivate the sodium peroxide.

7. (C) OTHER HEAT SYSTEMS

Noted during the course of the investigations were the following heat generating systems:

zinc dust - aluminum dust - water
zinc dust - ammonium chloride - ammonium nitrate - water

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potassium chlorate - starch - sulfuric acid
potassium permanganate - gallic acid - water
potassium permanganate - pyrogallol - water

All of these were eliminated from further consideration primarily because they reacted prematurely while mixing or the problem of partitioning the liquid component was too difficult or the heat generation cycle was too slow.

8. (C) MARKING COMPOUNDS

Since primary attention was given to development of an audible device, only brief study of marking compounds and their dissemination characteristics was made. Compounds tested were:

- a dye - Victoria Green WB⁴
- a color precursor - Color Precursor No. 1⁴
- a UV fluorescent dye - Lumogen Yellow⁵

The method of dissemination was described in Section II.

For the demonstration at Eglin only Color Precursor No. 1 was used. It is a white to light tan colorless powder in its normal state. When treated with a weak acid such as dilute acetic acid or vinegar the powder is converted instantaneously to a deep blue color of high tinctorial value. The precursor can be returned to its colorless form by treating it with weak alkali solution.

The Victoria Green WB is a triphenylmethane dye of very high tinctorial value. It tends to react with the human skin making it difficult to obliterate the green color. The color can be destroyed with strong bleaching agents.

The Lumogen Yellow fluoresces under black light. It tends to cling to the skin even after several washings.

4 Product of Allied Chemical Corporation, Industrial Chemicals Division

5 Product of Badische Anilin und Soda Fabrik A.G.

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SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

1. CONCLUSIONS

The evidence presented herein supports the conclusion that a chemical, pressure sensitive, personnel detection system is feasible and can be developed into an operational device. Considerable insight into the chemical and mechanical prerequisites for such a system has been achieved. Essentially all the chemical-mechanical principles have been demonstrated either in the laboratory or in the field. It is believed that with further effort the sensitivity and reactivity of the Device D system could be optimized to meet the operational military objectives that are beyond the scope of this initial investigation.

2. RECOMMENDATIONS

It is recommended that further design and development work be done to meet the required operational military objective.

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