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Aberdeen Proving Ground, Maryland 21010

TECHNICAL REPORT ARCSL-TR-82029

RIOT CONTROL AGENT DECONTAMINATION: EVALUATION OF COMMERCIAL PRODUCTS

by

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Chemical Branch
Research Division

June 1983

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A variety of commercial cleaning, bleaching and disinfecting products were examined for their potential utility in the decontamination of CN, CS, and CR. Some of these products were found to be quite useful in reacting with or promoting the reactions of these agents. Specific recommendations are made regarding the use of these materials.

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PREFACE

The information in this report was generated by work funded through an In-House Laboratory Independent Research program, Project 1L161101A91A. This report is concerned wth decontamination of riot control agents. The experimental data are recorded in notebook no. 9831. The period covered was from August 1978 to August 1980.

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RIOT CONTROL AGENT DECONTAMINATION:
EVALUATION OF COMMERCIAL PRODUCTS

1. INTRODUCTION

The deployment of riot control agents by either military or civilian law enforcement agencies is often the only viable method of preserving order while minimizing personal injury and property damage during civil disorders. Unfortunately, this results in the contamination of public areas with riot control chemicals which must be decontaminated as soon as order is restored. The current recommendation for decontamination of areas contaminated with riot control agents is to scrub the affected areas throughly with detergent in water.[1] While in some cases this is sufficient, it is not often possible to reach all the affected surfaces. Porous surfaces will retain a significant amount of contamination after washing.

There have been several laboratory studies to develop chemical decontaminants for α- chloroacetophenone (CN),[2] o-chlorobenzylidenemalononitrile (CS),[2-6] and dibenz(d,f) (1,4)-oxazepine (CR).[7,*] Several chemicals have been observed to react with these materials but none are suitable for large-scale decontamination of public areas because they are expensive, noxious, difficult to store, difficult to use, or corrosive. This study was undertaken to determine whether any commercially available cleaning products could be of value in this decontamination problem.

2. EXPERIMENTATION

All commercial products were obtained from local retail stores (see appendix). In many cases, equivalent products are available under several different brand names. An effort was made to avoid duplicating product types, and most products are widely available in the US. CS and CR were obtained from Munitions Division, CSL. The CS was purified by vacuum sublimation to produce large white crystals, m.p. 55° to 56°C. The CR was recrystallized from isooctane, yielding yellow crystals, m.p. 72 to 73°C. CN (phenacyl chloride) was obtained from commercial sources and used without further purification.

To evaluate the reactivity of the various preparations, a solid deposit of the agent was first formed in the bottom of a vial. This was done by preparing a solution containing the agent (1.0% w/v) and nonadecane (0.1% w/v) in chloroform. A 1.0-ml aliquot of this solution was placed in a 3-ml vial and allowed to evaporate to dryness leaving 10 mg of agent and 1 mg of nonadecane as a film on the bottom. One ml of the potential decontaminant was then added to the vial. After waiting a suitable length of time, 1.00 ml of chloroform and ca. 100 mg of sodium chloride were added to the vial and the mixture shaken. The chloroform layer was then analyzed for residual agent by gas chromatography.

All three agents were analyzed by the same gas chromatographic system. This consisted of a Hewlett-Packard 5730A chromatograph with a 4-mm by 183-cm glass column. The column was packed with UC-W98 (5%) on 100/120 Chromasorb W. The carrier gas was helium flowing at 60 ml/min. The injection and flame ionization detector (FID) temperatures were 250°C. The oven temperatures were programmed as follows:

CN: 130°C for 2 min after injection followed by 130 to 230°C @ 16°/min
CS and CR: 150 to 230°C at 8°/min (no program delay).

The signal from the FID was processed by a Hewlett-Packard 3380S recorder/integrator.

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* Unpublished UK data, 1965-1967.

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3. RESULTS AND DISCUSSION

Table 1 summarizes the results of the tests for CN decontamination. These data are consistent with the previously reported observation that CN undergoes slow hydrolysis in alkaline solution.[10] The most significant impediment to the alkaline hydrolysis of CN is its poor solubility in aqueous systems. Thus, it is observed that in aqueous trisodium phosphate alone, CN shows no significant reaction in spite of the high alkalinity of the solution. In Fantastik, however, which is only moderately alkaline, CN undergoes a slow but quite measurable hydrolysis. When trisodium phosphate (TSP) is dissolved in Fantastik, the combination of the detergency of the latter and the alkalinity of the former combine to produce substantial hydrolysis.

Table 1. Behavior of CN in Various Media
Produced from Commercial Products

Number of medium	Medium	Reaction in 5 hours
		%
1.	Fantastik (neat)	20
2.	Fantastik saturated with Tintex color remover	30
3.	Fantastik saturated with Clorox 2	10
4.	Zephiran (0.85% in (TSP))	20
5.	Trisodium phosphate (1%)	0
6.	Fantastik with 1% (TSP)	65
7.	Zephiran (0.85%) and TSP (1%) in water	35
8.	Fantastik saturated with Vanish	0

Table 2 summarizes the results of the test with CS. CS has been observed to undergo both hydrolysis[11] and oxidation.[3] Like CN, the use of a high detergent formulation such as Fantastik is very effective in promoting CS hydrolysis although the reaction is still quite slow. Nonchlorine oxidants such as perborate (Clorox 2) and peroxysulfate (Vanish) were ineffective against CS. The most dramatic results were obtained with those formulations containing Tintex color remover (TCR). The active ingredient in this product is sodium dithionite ($Na_2S_2O_4$). Sodium dithionite is a crystalline solid which is stable at room temperature in dry air. In acid solution, it quickly decomposes into sulfate, sulfite and elemental sulfur. It is considerably more stable in alkaline solution where it functions as a strong reducing agent.[12] It is this property of dithionite solutions which makes them useful for decolorizing fabrics tinted with vat dyes. Alkaline dithionite will also react quickly and quantitatively with dissolved oxygen and has been employed in the quantitation of the same. It was initially thought that dithionite was promoting the reduction of the cyano groups in CS to amines. This, however, is inconsistent with the known chemistry of nitriles[13] which require reactive hydrides or catalytic hydrogenation to effect such reductions. Furthermore, the product(s) of the CS-dithionite reaction are very soluble in alkaline water which would not be expected of amine products.

Table 2. Behavior of CS in Various Media
Produced from Commercial Products

Number of medium	Medium	Reaction in 5 hours
		%
1.	Fantastik (neat)	89
2.	Fantastik saturated with Tintex color remover	100
3.	Fantastik saturated with Clorox 2	54
4.	Downy fabric softener (1% in water)	0
5.	Downy (1%) saturated with Tintex color remover	100
6.	Downy (1%) saturated with Clorox 2	11
7.	Spic & Span (7%) in water	0
8.	Spic & Span (7%) saturated with Tintex color remover	100
9.	Spic & Span (7%) saturated with Clorox 2	8
10.	Zephiran (0.85%) in water	0
11.	Zephiran (0.85%) saturated with Tintex color remover	100*
12.	Zephiran (0.85%) and TSP (1%)	9
13.	Fantastik saturated with Vanish	0
14.	Zephiran (0.85%) saturated with Vanish	0
15.	Tintex color remover (saturated in water)	100**

* Reaction after 15 min was 30%
** Reaction after 15 min was 78%

Kerber and Starnik[14] have reported that acrylonitrile reacts with dithionite as follows:

$$S_2O_4^{=} + H_2O \rightleftharpoons HS_2O_4^- + OH^- \qquad (1)$$

$$HS_2O_4^- + H_2O \rightleftharpoons HSO_2^- + HSO_3^- + H^+ \qquad (2)$$

$$HSO_2^- + CH_2{=}CHCN \rightarrow {}^-O_2S\text{-}CH_2\text{-}CH_2CN \qquad (3)$$

$$^-O_2S\text{-}CH_2\text{-}CH_2CN + CH_2{=}CHCN \rightarrow \begin{array}{c} CH_2\text{-}CH_2CN \\ | \\ SO_2 \\ | \\ CH_2\text{-}CH_2CN \end{array} \qquad (4)$$

While the analogy to the CS/dithionite system is obvious, a sulfone product would not be water soluble. Kerber and Starnik, however, ran their reactions with the acrylonitrile in excess. In the studies reported here, the dithionite was maintained in large excess over CS. As such, reaction (3) would predominate and the water soluble sulfinic acid would be the primary product. At present the evidence in support of this reaction pathway for CS/dithionite is only circumstantial. Studies are continuing in this laboratory to provide additional information on this system.

As the data in table 3 indicates, CR was very resistant to decontamination by the commercial products tested. There is some indication that acid catalyzed hydrolysis in aqueous detergent or isopropanol will proceed slowly. The alkaline system of Fantastik and trisodium phosphate was ineffective. Likewise, perborate (Clorox 2) and peroxysulfate (Vanish) oxidants produced no reaction. Fichlor, a chlorine bleaching powder, rapidly destroyed the CR. Mention

Table 3. Behavior of CR in Various Media Produced from Commercial Products

Number of medium	Medium	Reaction in 5 hours
		%
1.	Fantastik (neat)	0
2.	Fantastik saturated with Tintex color remover	12
3.	Fantastik saturated with Clorox 2	0
4.	Zephiran (0.85% in water)	7
5.	Fantastik saturated with Vanish	0
6.	Fantastik with 1% TSP (1%)	0
7.	Zephiran (0.85%) and TSP (1%)	5
8.	Axion (saturated solution in water)	0
9.	Isopropanol/water (7:3) saturated with Fichlor	33
10.	Isopropanol/concentrated HCl (7:3)	30
11.	Isopropanol/water (7:3) with excess Tintex color remover	52[a]
12.	Isopropanol/water (7:3) with excess Fichlor	100
13.	Isopropanol/30% hydrogen peroxide (7:3)	0
14.	Water/30% hydrogen peroxide with excess Tintex color remover	30[a,b]
15.	Isopropanol/30% hydrogen peroxide with excess Tintex color remover	100[a]

[a] "Excess" means 2 to 3 times the amount required for a saturated solution.
[b] Reaction after one minute was 100%.

is made in a 1969 report* that sodium dithionite was efficacious in reducing the irritation of skin by CR but the observation does not appear to have been pursued. In this study, aqueous dithionite (TCR) showed some effect but complete reaction was never achieved. When solid Tintex color remover is added to the vial containing the solid CR and a mixture of isopropanol

* Unpublished CSL review.

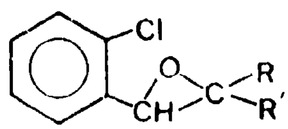
and hydrogen peroxide added, a vigorous reaction occurs. Considerable heat is produced and a foul smelling gas is evolved. While much of this is undoubtedly due to the redox reaction of the dithionite and peroxide, analysis showed the CR to be destroyed within 1 minute. The gas chromatogram showed several product peaks which changed over a period of several hours. Likewise, the color of the reaction mixture changed from its initial yellow-orange color through orange, dark red and finally to violet-brown over the same period of time. It is clear that this system is undergoing several concurrent and/or sequential reactions with a multitude of products. No efforts were made to identify them.

4. CONCLUSIONS

The decontamination of CN may be effected by a strongly alkaline detergent solution such as trisodium phosphate in Fantastik. Trisodium phosphate is somewhat corrosive, making skin protection essential. Some damage to aluminum, paint and finished wood surface may be expected.

Sodium dithionite-based products offer considerable promise in the decontamination of CS. The reaction appears to be quite rapid, produces a highly soluble product and the excess dithionite will decompose into harmless products. Because of its bleaching capabilities, some damage to fabrics may be expected.

The problem of CR decontamination is a difficult one and will require further investigation. While Fichlor is effective, its use is contraindicated for several reasons. Fichlor is quite corrosive and may be expected to do considerable damage to environmental surfaces. Further, the reaction of CR with Fichlor may produce chlorinated aromatics which are considered undesirable environmental contaminants. Finally, CR is often deployed in conjunction with CS. It has already been observed that CS can react with strong oxidants to produce epoxides of the type



and that such compounds show vesicant activity. Sodium dithionite does appear to react with CR and further studies in this area may result in optimization of this reaction to the extent that it may be useful in both CS and CR decontamination. Clearly, the promotion of the CR/dithionite reaction by peroxides is unsuitable for use in any location other than a laboratory fume hood.

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APPENDIX

DESCRIPTION OF COMMERCIAL PRODUCTS USED IN THIS INVESTIGATION

Detergents

Fantastik: Trademark, Morton Norwich Products; an "all purpose" household cleaning product consisting primarily of anionic detergents and mild organic solvents.

Spic & Span: Trademark, Proctor & Gamble Co.; a granular household cleaner consisting of anionic detergents and phosphate salts which appear to buffer the solution at pH ca. 9.

Zephiran: Trademark, Winthrop Laboratories; a disinfectant consisting primarily of cationic detergents of the benzalkonium family.

Downy: Trademark, Proctor & Gamble Co.; a fabric softener for home laundry use. The primary component of most such products are cationic detergents.

Reactive products

Tintex color remover: Trademark, Knomark, Inc.; a color stripper for pretreating fabrics prior to dying. The active ingredient is sodium dithionite ($Na_2S_2O_4$) with buffers (pH 8 to 9) and a small amount of detergent.

Vanish: Trademark, Drackett Products Co.; a toilet bowl cleaner and disinfectant whose active ingredient is sodium monopersulfate ($NaHSO_5$).

Clorox 2: Trademark, The Clorox Co.; an "all fabric" laundry bleach containing sodium perborate as the oxidant.

Fichlor: Sodium N,N-dichloroisocyanurate; a chlorine bleaching powder widely used in water treatment and commercial fabric bleaching.

TSP: Trisodium phosphate; offered in paint and hardware stores under a variety of trade names; aqueous solutions are strongly aklaline and are used to clean and degloss surfaces prior to repainting.

Axion: Trademark, Colgate-Palmolive Co.; a home laundry pre-soak containing modified hydrolytic enzymes of bacterial origin.

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