



Surfactant-Based Chemical and Biological Agent Decontaminating Solution Development

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Report Documentation Page

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Background

- **Current Decontaminating (Decon) Solutions (Navy HTH and Army DS-2) Are:**
 - Toxic
 - Corrosive to Materials
 - Environmentally Unfriendly
- **Joint Service Family of Decon Systems (JSFDS) Program Requirement for New Decon**
 - Must Be As Effective As Current Decons
 - Effective Against Biological Agents
 - Compatible (Noncorrosive) with Military Materials
 - Environmentally “Green” and Nontoxic to User



Objectives

- **Develop a Surfactant-Based Decon Solution That Will Meet JSFDS Requirements**
- **Most Challenging Requirements**
 - Neutralization Time of 15 Minutes for VX, HD, and TGD
 - Pot Life of 12 Hours
 - Disinfect Vegetative and Endospore Forming Bacteria, Fungi, and Viruses in 15 Minutes
 - Noncorrosive to Military Materials
 - Nontoxic and Environmentally “Green”



Decon Formulation Components

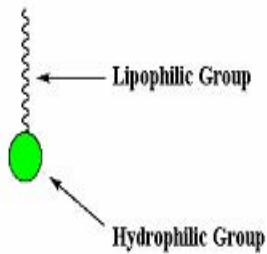
- **Surfactants**
 - Use to Replace Organic/Halogenated Solvents to Solubilize Chemical Agents
 - Must Be Stable Against Oxidation and Hydrolysis
 - Utilize to Increase Biocidal Efficacy by Lowering Interfacial Tension Between Spores and Biocide
- **Peroxygen Compound**
 - Utilize as Environmentally Green Reactant for Both Chemical and Biological Agents
 - Some Peracids Available in Neat Form (Peracetic acid) and In-Situ (Commercial Detergent Technology)
- **Catalyst**
 - Promote Oxidation of the Sulfur in V and HD
 - Reported to Promote Hydrolysis at Lower pH (for G Agent Decontamination)



Decon Formulation Components - Surfactants

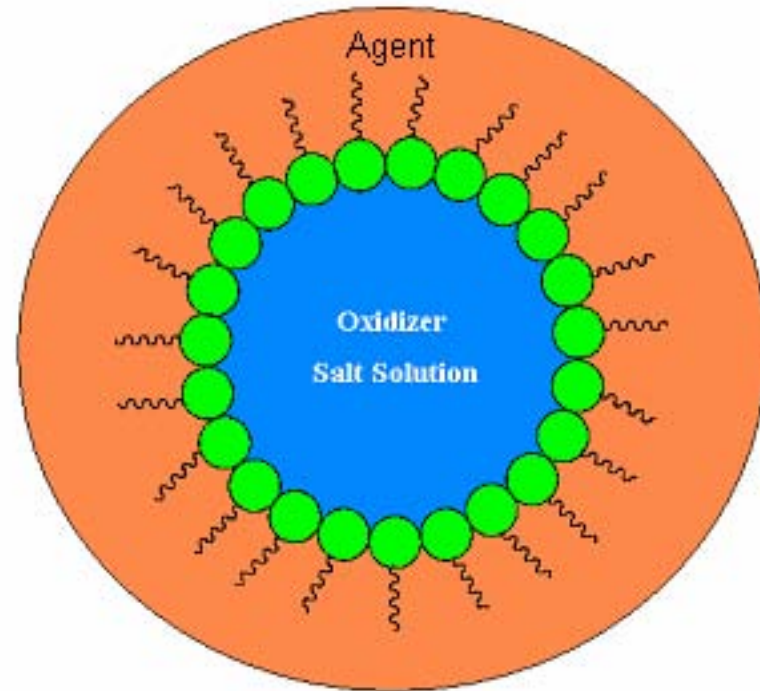
- **Basics of Microemulsion Formulation**

Surfactant



Mix These 3
→
Components

Agent - Nonpolar Molecules (Liquid)
Oxidizer - Polar Molecules (Liquid)





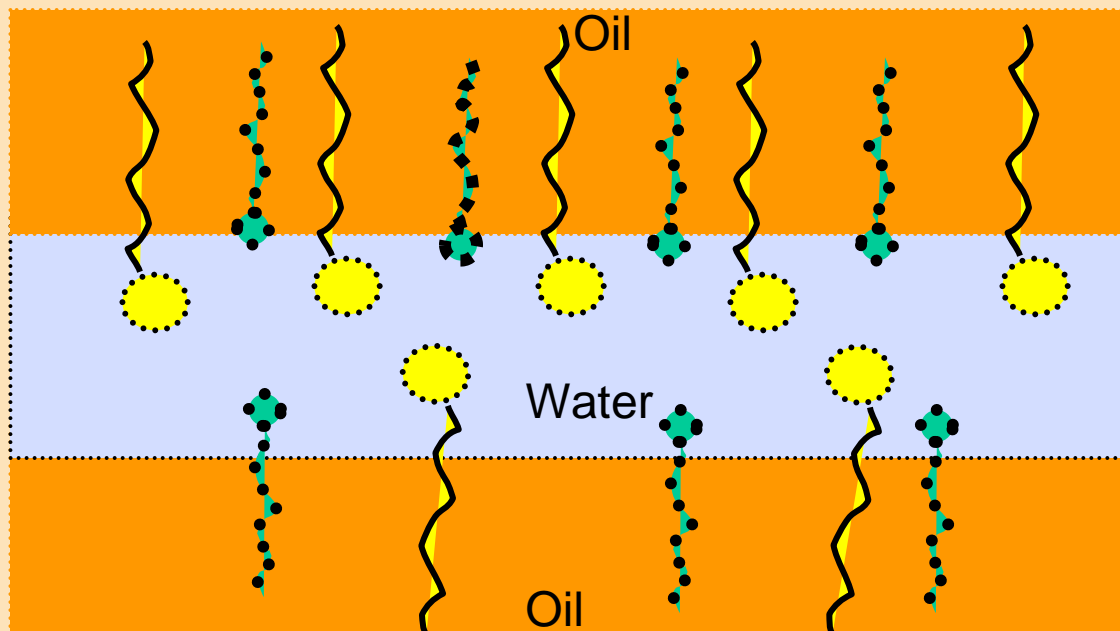
Water-in-Oil Microemulsion



Decon Formulation Components - Surfactants

● MICROEMULSION FORMULATION

-  Identify surfactant(s) slightly soluble in the aqueous phase
-  Identify surfactant(s) slightly soluble in the oil phase
- Combine oil soluble and water soluble surfactants to reduce interfacial tension between oil and water phase to produce microemulsion





Decon Formulation Components - Surfactants

- **Surfactant / Solubility Screen**

J A B C D E F G H I



Oil/Water = 20/80; Surfactant = 3%; Oil = Dibutylsulfide; Water = water + 500-mg Na carbonate

J: control

A: C₁₆ Diphenyloxide sulfonate

B: C₁₄₋₁₇ sec-Alkyl sulfonate

C: C₆₋₁₆ Cocamidopropylamine oxide

D: C₁₀ Amine oxide

E: C₁₂ Amine oxide

F: C₁₄ Amine oxide

G: C₁₂₋₁₈ Amine oxide

H: C₁₈ Amine oxide

I: di-C₁₀ Amine oxide



Decon Formulation Components - Surfactants

- **Surfactant Scan – Amine Oxides**

J **A** **B** **C** **D** **E** **F** **G**
S1/S2= 100/0 90/10 75/25 50/50 25/75 10/90 0/100



O/W = 20/80; (S1+S2) = 3%; V(T) = 5-mL; O = Dibutyl sulfide, W = water + 500-mg Na carbonate

J: control (no surfactant),

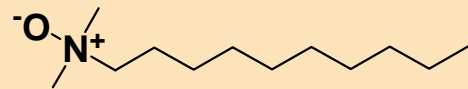
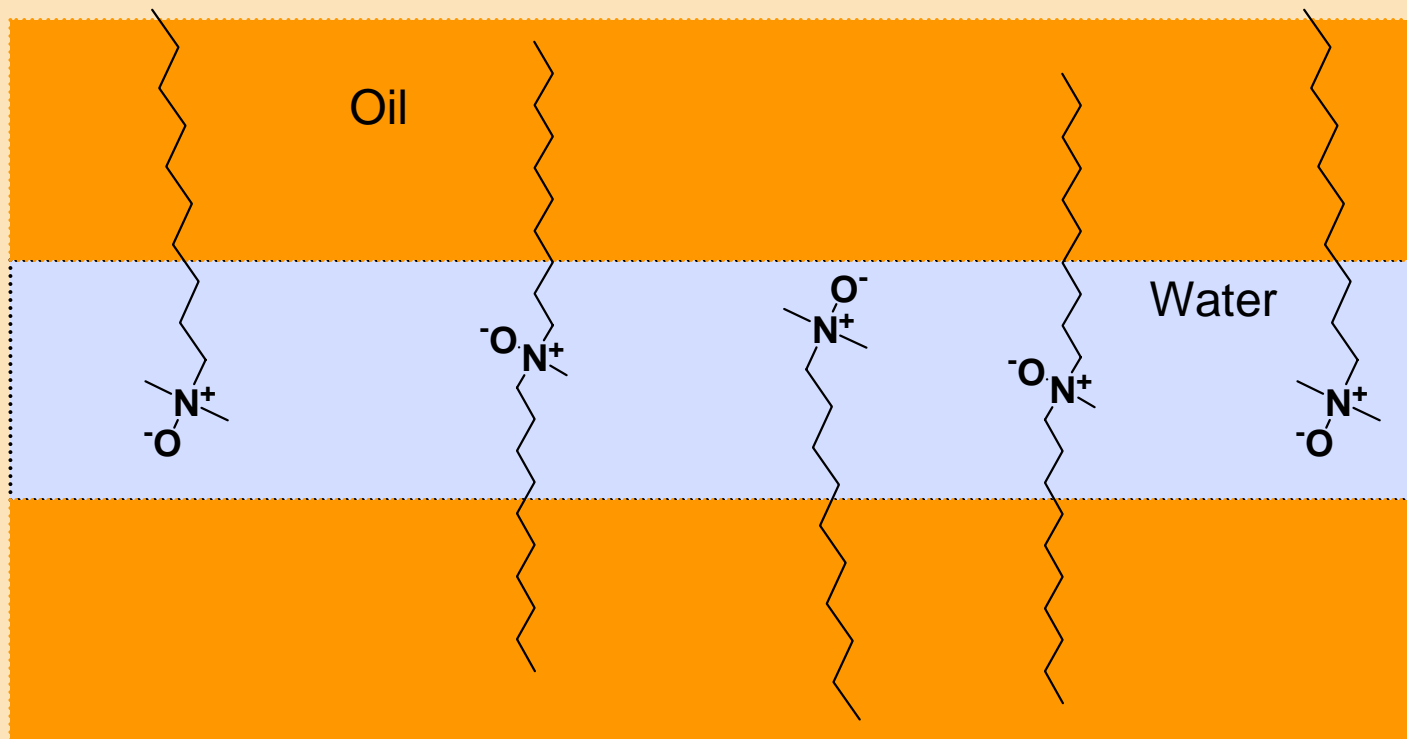
S1: C₁₀ Amine oxide

S2: di-C₁₀ Amine oxide

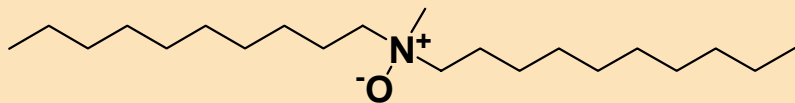


Decon Formulation Components - Surfactants

- **Current System - Mixed Amine Oxide Surfactant Microemulsion**



C₁₀ Amine oxide. Dimethyldecylamine oxide



Di-C₁₀ Amine oxide. Didecylmethylamine oxide



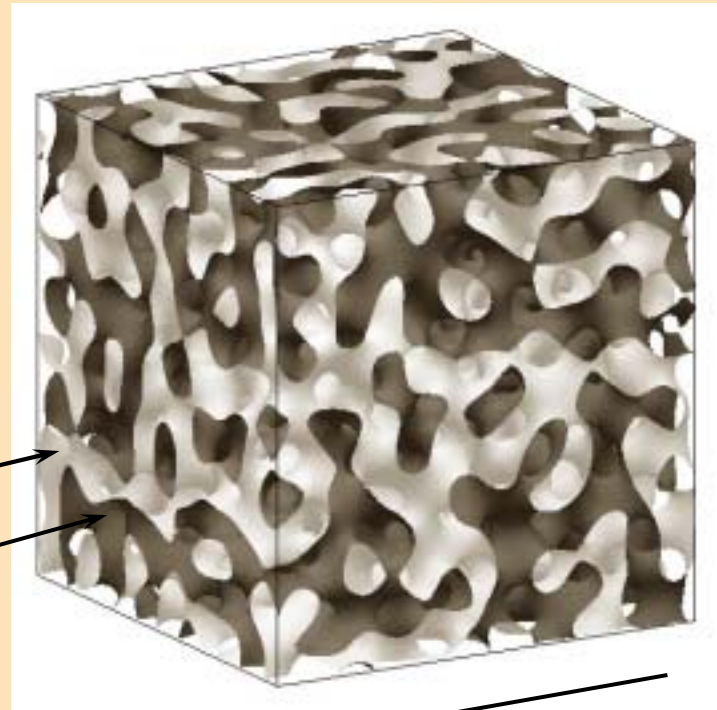
Decon Formulation Components - Surfactants

- **Why Microemulsion?**

- Increased Stability
- Increased Reactivity (Surface Area)

Lipophilic (Agent)

Hydrophilic (Oxidizer)



10 nm



Decon Formulation Components – Peroxygen Compound

- **Peroxygen Compounds**

- **Organic Peroxides (t-Butyl hydroperoxide)**

- Requires Catalyst to Meet Decon Reactivity Requirement
- Stable - Does Not Require Special Handling
- One of Two Organic Peroxides That Meet DOT Shipping Regs for Bulk Shipment

- **Peracids**

- Strong Oxidizers
- Broad Spectrum Disinfectants
- Use Neat or Generate In-Situ (Tide With Bleach)
- Stability (Handling / Storage) Can Be an Issue



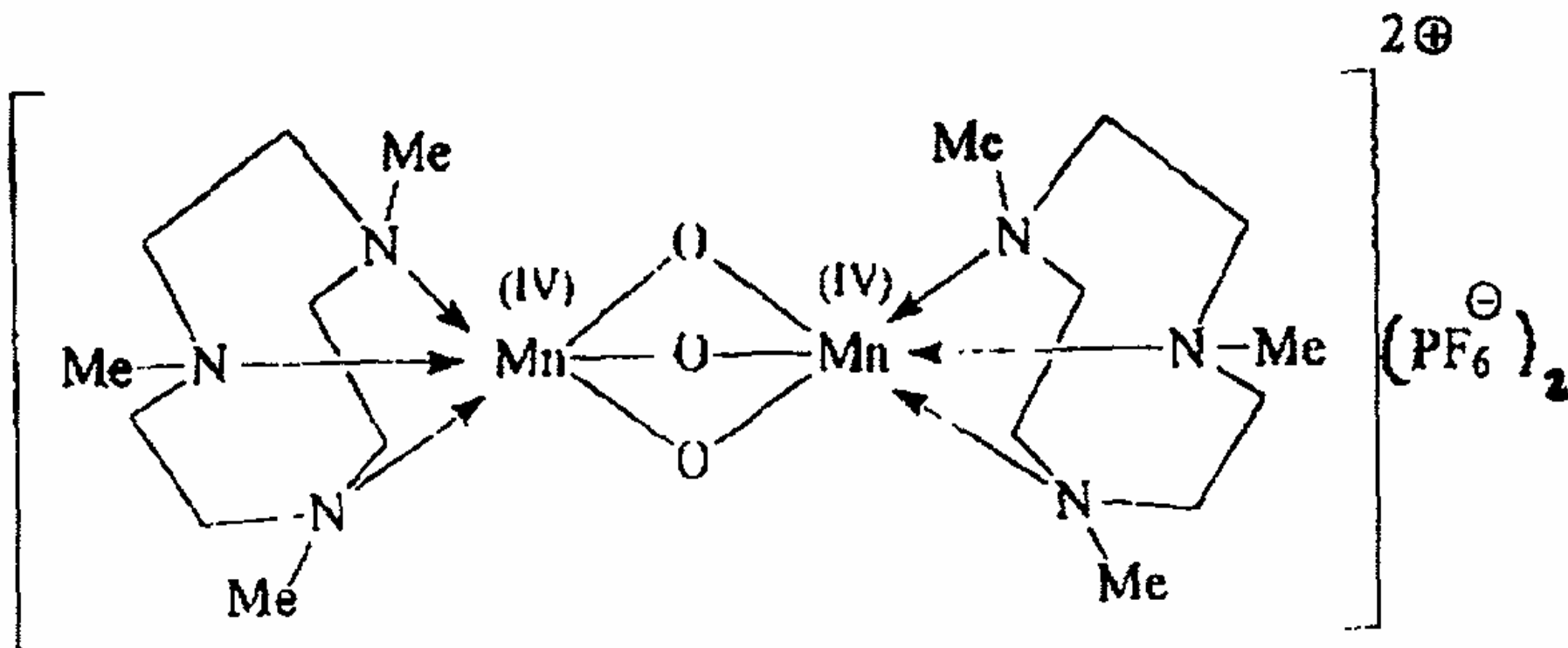
Decon Formulation Components – Catalyst

- **Dimanganese-Based Complex**
 - Developed for Laundry and Detergent Industry
 - Catalyst Activates Stain ($\text{Mn}^{4+} + \text{e}^{-} \rightarrow \text{Mn}^{3+}$)
 - Stain More Susceptible to Bleach (Oxidation)
- **Macrocyclic Tetradentate Liqand (TAML)**
 - Developed by Carnegie Mellon
 - Complexes the Peroxygen
 - Stable in Presence of Strong Oxidants
 - Defluoro Reported to Promote Hydrolysis and Oxidation at Lower pH



Decon Formulation Components – Catalyst

- **Dimanganese-Based Complex**
 - 1,4,7-Trimethyl-1,4,7-triazacyclononane ligands (Me₃TACN)

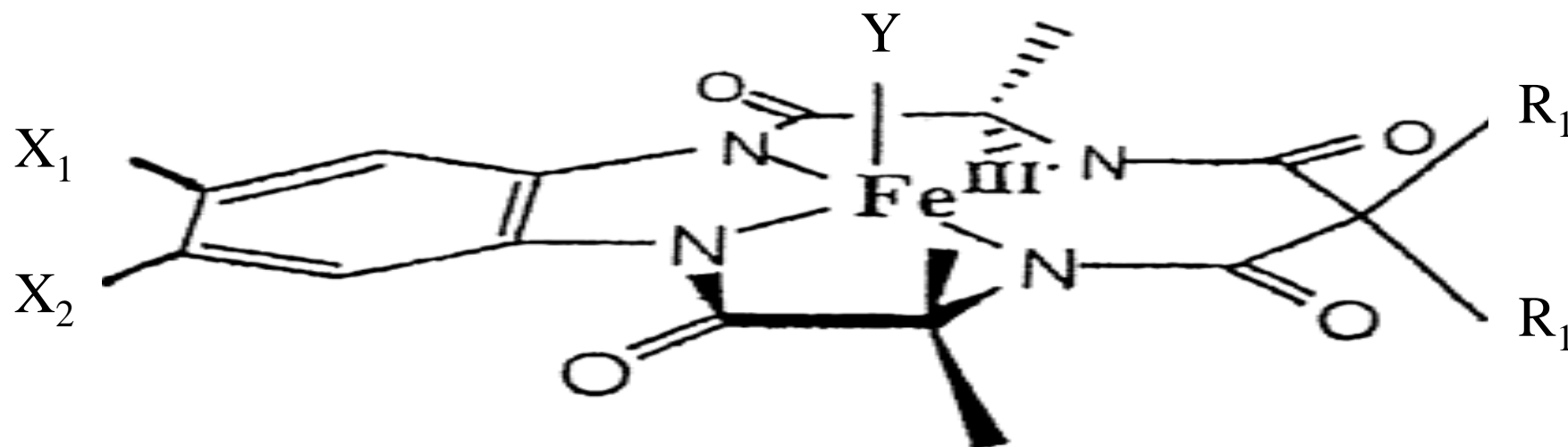




Decon Formulation Components – Catalyst

- **Macrocyclic Tetradentate Ligand**

	<u>X₁</u>	<u>X₂</u>	<u>R₁</u>
– FeMB	Me	H	Me
– FeB	H	H	Me
– FeF ₂ B	H	H	F
– FeF ₂ DCB	Cl	Cl	F

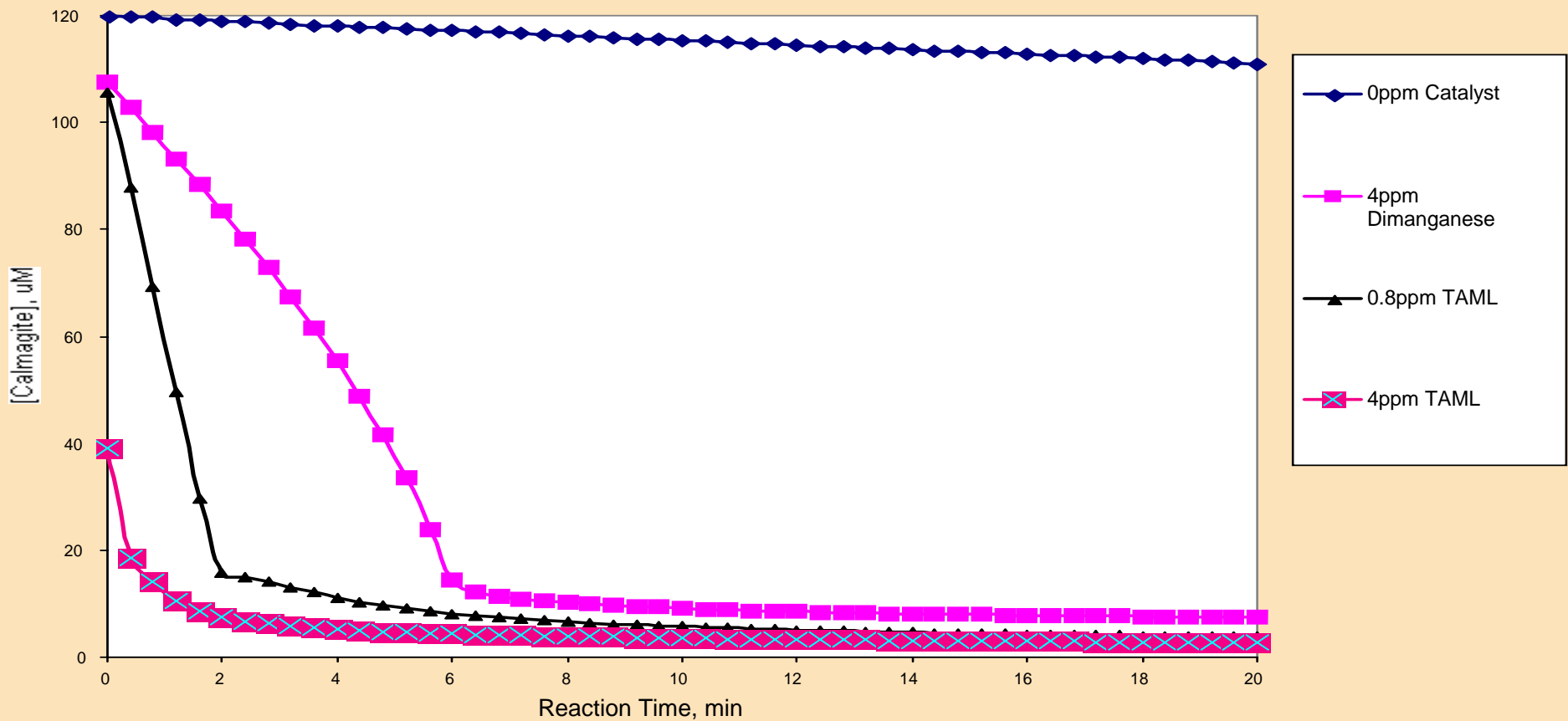




Decon Formulation Components – Peroxygen Compound and Catalyst

Oxidation of Calmagite Dye by t-Bu Hydroperoxide

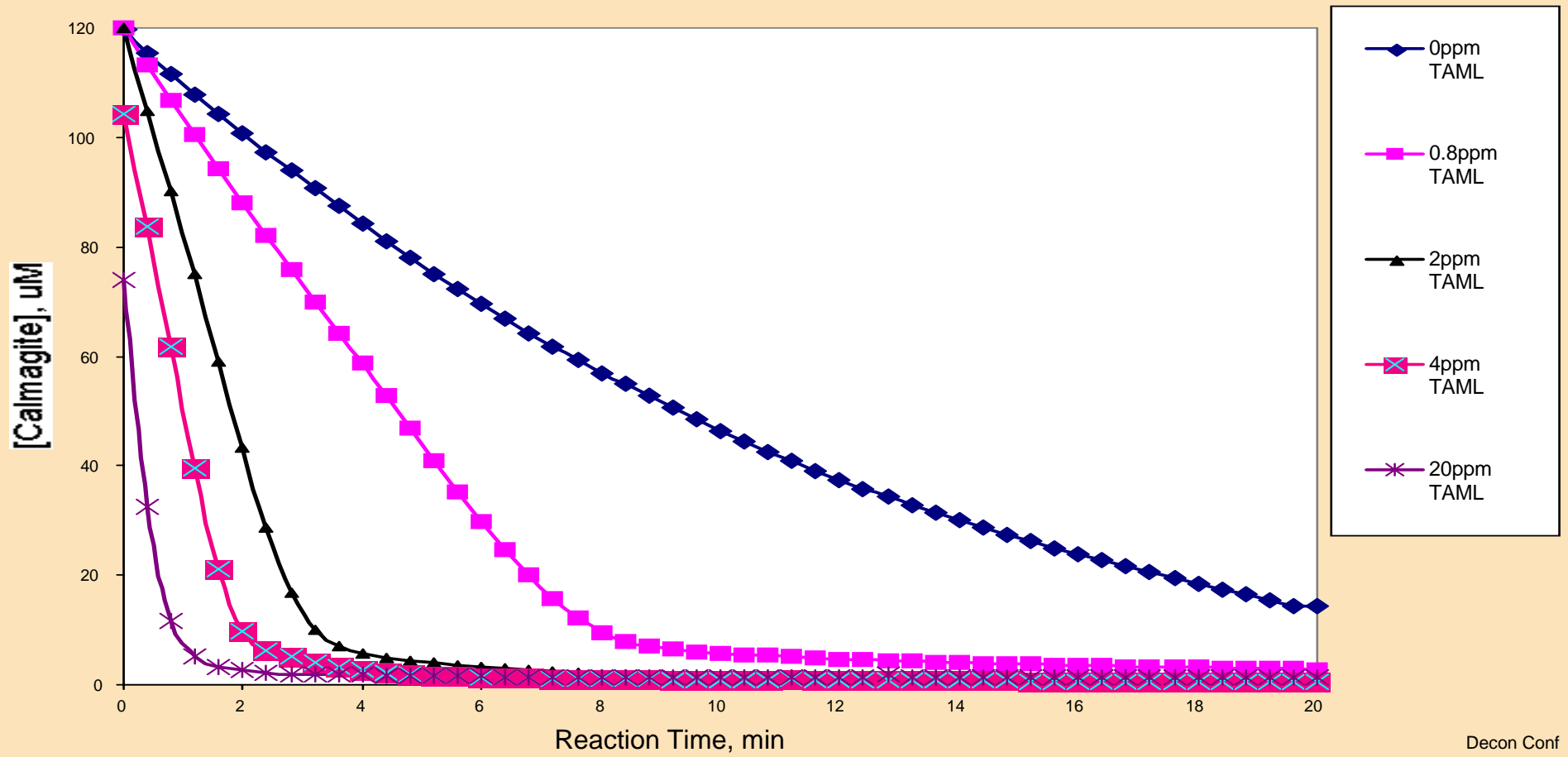
TAML FeMB and Dimanganese Catalysts





Decon Formulation Components – Peroxygen Compounds and Catalysts

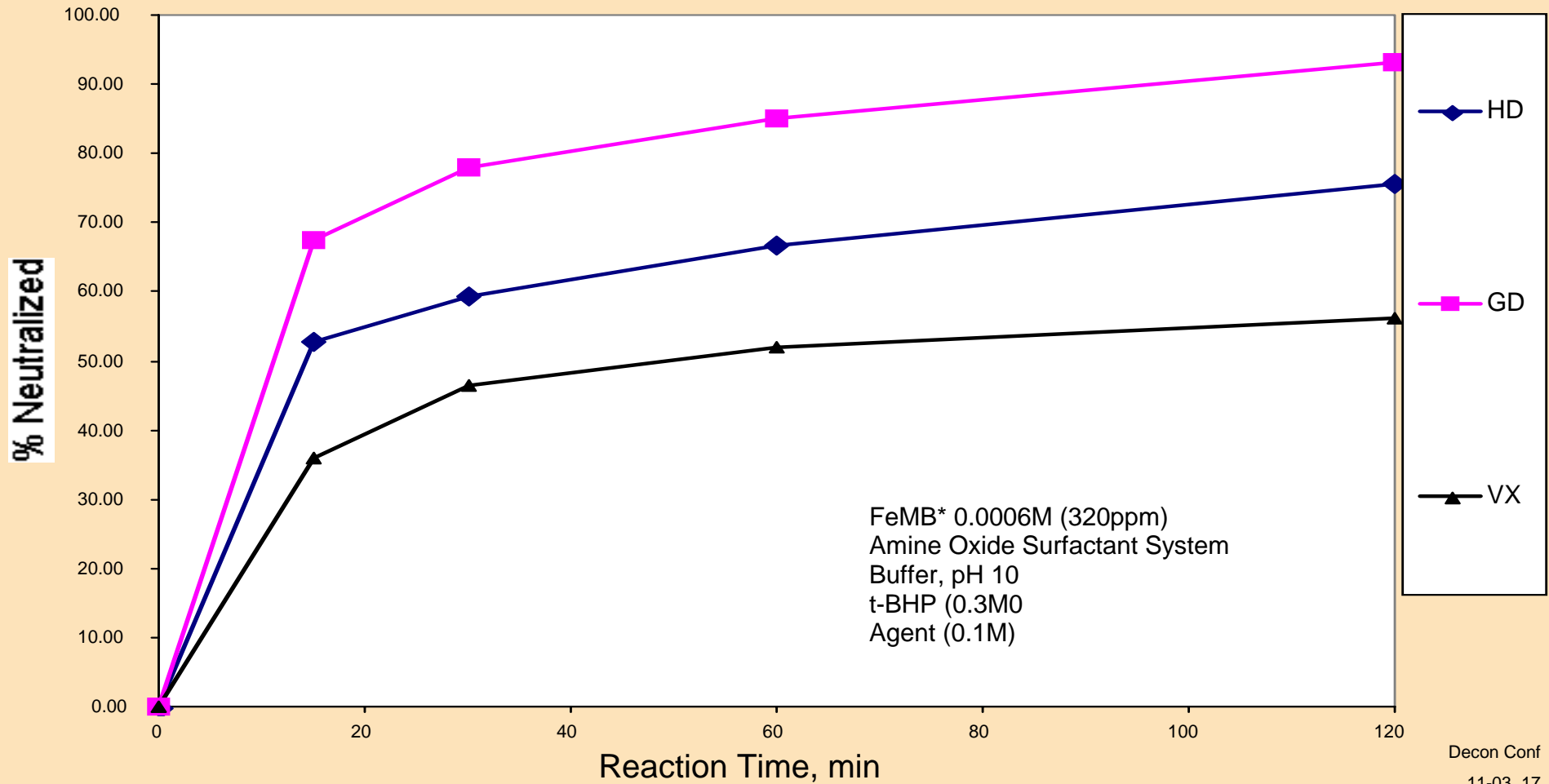
Oxidation of Calmagite Dye by Peracetic Acid TAML FeMB Catalyst





Decon Formulation Components – Peroxygen Compounds and Catalysts

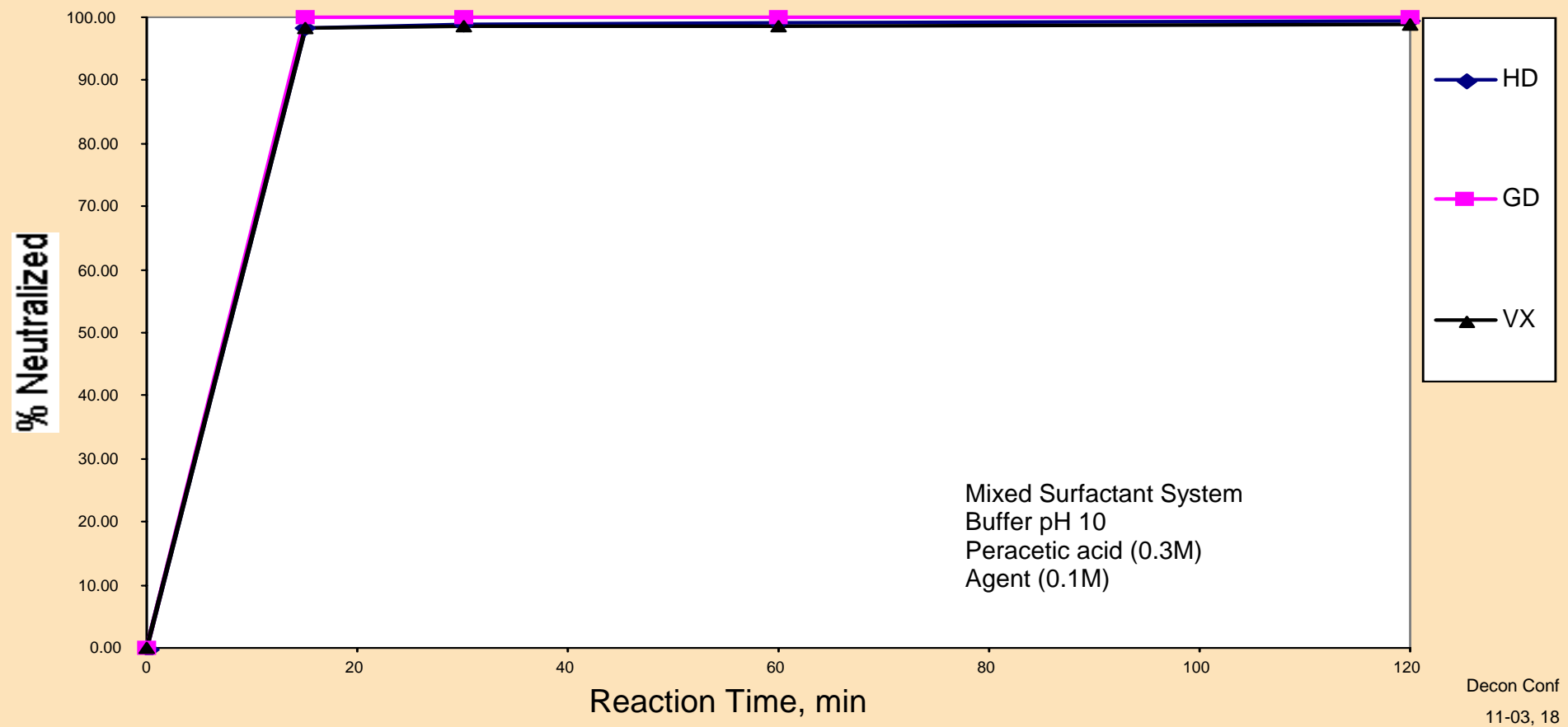
Agent Decontamination by t-Bu Hydroperoxide in Microemulsion TAML FeMB Catalyst





Decon Formulation Components – Peroxygen Compounds and Catalysts

Agent Decontamination by Peracetic Acid Uncatalyzed





Biological Decontamination

- **Peracetic Acid (PAA) Found to Be an Effective Disinfectant**

Reduction in *Bacillus globigii* after 15 minute Exposure to Candidate Solutions

	<u>BG Initial CFU/mL</u>	<u>Log Reduction, CFU/mL</u>
Damox 1010 (1)	10 ⁶	0 (30 min exposure)
Barlox 10S (2)	10 ⁶	0 (30 min exposure)
PAA, 5%	10 ⁸	4
PAA in uEm	10 ⁸	8

(1) C₁₀ Amine oxide

(2) di-C₁₀ Amine oxide



Summary

- **Microemulsions Developed From Oxidation and Hydrolysis Resistance Surfactants**
- **Peracids (Peracetic) Demonstrated Efficacy Against Chemical Agents (HD, VX, GD)**
 - Efficacy Achieved Without Catalyst
- **Peracids (Peacetic) Demonstrated Efficacy Against Biological Agent Simulant (BG)**