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LABORATORY EVALUATION OF THE CLEAN EARTH TECHNOLOGIES DECONTAMINATION SOLUTIONS FOR CHEMICAL AND BIOLOGICAL AGENTS

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14. ABSTRACT:
The purpose of this test was to evaluate two Clean Earth Technologies (CET) decontamination solutions against agent at the laboratory scale per a customer request. The Chemical Decontamination Solution (CDS) was evaluated against the chemical agents, HD, GD, and VX, using standard stirred reactor and panel tests. The results were compared against DF200. The Biological Decontamination Solution (BDS) was evaluated against *Bacillus anthracis* using standard methods and compared against bleach. The work was conducted for information purposes only; this test was not intended to certify any commercial product. The test procedures and results are detailed in this report. Test results show CET CDS to be superior or comparable to DF200 in the decontamination of HD, GD, and VX. Test results also show CET BDS to be superior or comparable to bleach in the destruction of *Bacillus anthracis* spores. The work discussed in this report was conducted from April to December 2006.

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PREFACE

The work described in this report was started in April 2006 and completed in December 2006.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

Manufacturer names and model numbers are provided for completeness.

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LABORATORY EVALUATION OF THE CLEAN EARTH TECHNOLOGIES DECONTAMINATION SOLUTIONS FOR CHEMICAL AND BIOLOGICAL AGENTS

1. INTRODUCTION

Clean Earth Technologies (CET), LLC, introduced into the marketplace an Electrostatic Decontamination System (EDS) technology for "the decontamination of biological and chemical warfare agents and toxic industrial chemicals (TICs) on a variety of hard and porous surfaces without adversely affecting materials."[1-5]

The Clean Earth Technologies decontaminant system consists of two solution products. One solution is specifically designed for biological agent. The second solution is specifically designed for chemical agent.

A Biological Decontamination Solution (BDS) and an ultraviolet (UV) light source are used for biological decontamination applications.[2] The BDS is a 24% hydrogen peroxide concentrate that is diluted down to approximately 4% or less for application. The BDS solution can be diluted according to the CET report with "city water supply, ocean or river and no difference in efficacy has been observed." Biological Decontamination Solution is applied as a photosensitizer and illuminated by UV. The BDS is reported to be an effective antimicrobial agent. The application of UV light is reported to increase the rate of kill on the order of 6-log kill from minutes to seconds.

A Chemical Decontamination Solution (CDS) is used for chemical agent applications.[3] The CDS is prepared from two separate solutions at time of use. Solution "A" contains hydrogen peroxide. Solution "B" contains sodium hydroxide. Both solutions contain other ingredients to "promote the solubilization of the agent, the oxidation reaction, catalysis, and surface interactions." The principal active ingredient in the CDS is the peroxy anion. The mixed solution has about a 6-hr shelf-life before simulant decontamination efficacy begins to decline.

The purpose of this test was to evaluate two CET decontamination solutions against agent at the laboratory scale per a customer request. The CDS was evaluated against chemical agents HD, GD, and VX using standard stirred reactor and panel tests. The results were compared against DF200. The BDS was evaluated against *Bacillus anthracis* using standard methods and compared against bleach. The UV light source was not used during this testing. The work was conducted for information purposes only; this test was not intended to certify any commercial product. The tests were performed between April and August 2006 at the Edgewood Chemical Biological Center (ECBC), Aberdeen Proving Ground, MD. The results for the chemical- and biological-agent studies are presented in this report.

The purpose of this test was to evaluate the CET CDS and BDS decontaminants against chemical- and biological-agents. The summary of conclusions is provided in the bulleted list.

- The stirred reactor tests showed CET CDS decontaminant to be superior to DF200 for HD and VX decontamination and equivalent to DF200 for GD decontamination.

- No HD was detected in the CDS-HD reaction mixture after 10 min.
- HD was detected by GC-AED in all samples of the DF200-HD reaction mixture with an average of 2.12 ± 1.38% of the initial HD concentration was found in each reactor at 60 min. During the sampling period, concentration of HD increased and decreased sporadically—indicating the presence of HD globules in the DF200 solution.
- The ^{1}H-NMR spectrum of the CDS-HD reaction mixture showed that no HD was present. Detected compounds appeared to be hydrolyzed versions of sulfoxide and sulfone: 2,2'-sulfinyl diethanol and 2,2'-sulfonyl diethanol.
- ^{1}H-NMR analysis of the DF200-HD reaction mixture revealed the presence of 2-chloroethylvinyl sulfone and divinyl sulfone
- No VX was detected in the CDS-VX reaction mixture after 3 min.; whereas, approximately 1% residual VX was observed in the DF200-VX reaction mixture at 20 min.
- NMR analysis of the CDS-VX and DF200-VX samples showed the VX product ethylmethylphosphonic acid (EMPA)
- No GD was detected in either CDS or DF200 reaction mixtures at 2 min.
- NMR analysis of the CDS and DF200 reaction mixtures confirmed the absence of GD and the presence of the GD hydrolysis product pinacolyl methylphosphonic acid (GD-acid).

- The contact hazard tests showed that the CET CDS decontaminant efficacy ranged from comparable-to-superior than DF200 for all agents on CARC and aluminum surfaces.
  - The contact hazard on HD and GD contaminated CARC was below the threshold limit within 15 min following a 10- or 20-min decontamination with CDS.
  - For the 20 min residence time CDS cleaned GD from aluminum 2.7 times more than DF200. This condition was below the JPID threshold ORD level.
  - For the 20 min residence time CDS cleaned GD from CARC 1.5 times better than DF200. This condition was below the JPID threshold ORD level.
  - For the 20 min residence time CDS cleaned HD from CARC 6.4 times better than DF200. This condition was below the JPID threshold ORD level.
  - Aluminum and CARC contaminated with VX was not consistently decontaminated below the contact hazard threshold level by either CDS or DF200.

- The vapor hazard tests showed that the CET CDS decontaminant efficacy was equivalent to significantly better than the corresponding DF200 treatment.

Overall, the CDS performed equivalent or better than DF200 on aluminum.

- Both CDS and DF200 reduced the HD to below detection limits; the decontaminants show equivalent performance.
- CDS reduced the VX contamination 2.8 times lower than DF200 after a 10-min exposure
- CDS reduced the VX contamination 10.1 times lower than DF200 after a 20-min exposure
- Both CDS and DF200 clean GD to below detection limits; the decontaminants show equivalent performance.

Overall, the CDS performed equivalent or lower than DF200 on CARC painted aluminum

- CDS reduced the HD contamination 7.56 times lower than DF200 after a 10-min exposure.
- CDS reduced the HD contamination 7.42 times lower than DF200 after a 20-min exposure.
- CDS reduced the VX contamination 1.6 times lower than DF200 after a 10-min exposure.
- CDS reduced the VX contamination 4.91 times lower than DF200 after a 20-min exposure.
- CDS reduced the GD contamination 9.52 times lower than DF200 after a 10-min exposure.
- CDS reduced the GD contamination 13.61 times lower than DF200 after a 20-min exposure.

In summary, CDS performed better than DF200 for CARC and aluminum based test.

- The biological tests show that BDS is comparable to bleach for decontamination of *Bacillus anthracis* on unpainted and CARC-painted metal coupons.
  - >6-log kill was observed for bleach and BDS
  - 2 colony-forming units (CFU) were observed for bleach while zero CFU were observed for BDS

2. METHODS AND PROCEDURES

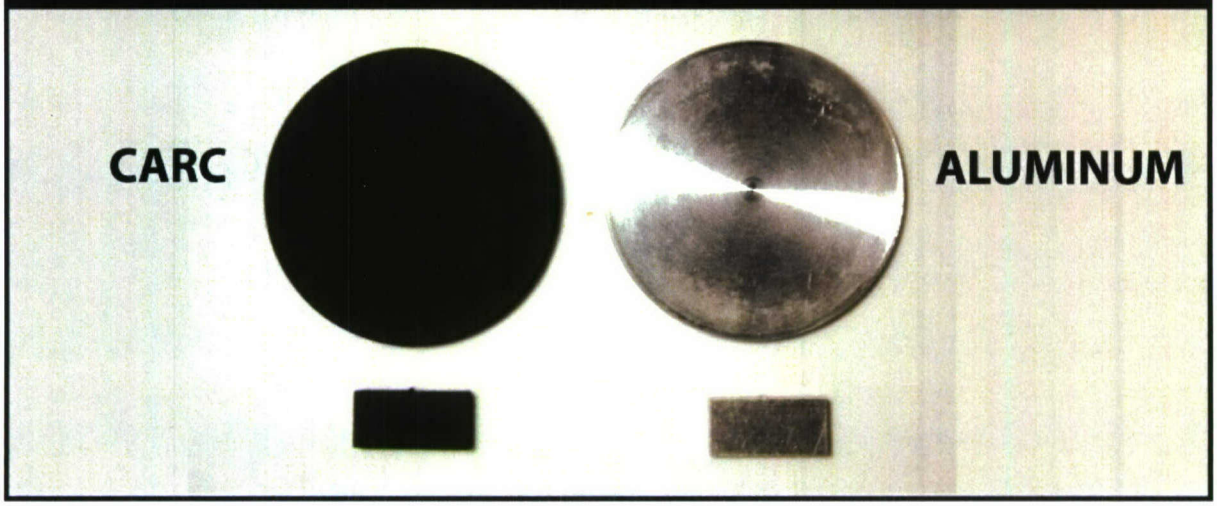
2.1 Test Solutions

The two decontaminants for evaluation are the Clean Earth Technologies (CET) Biological Decontamination Solution (BDS) and Chemical Decontamination Solution (CDS). The proposed testing utilized a disposable spray bottle application of the decontaminant. The solutions were provided by the customer and used according to manufacturer's directions. All testing was conducted with a side-by-side comparison to a reference decontaminant. The CET BDS evaluation will be compared to bleach. The CET CDS evaluation was compared to DF200. DF200 was selected since the customer currently uses this decontaminant.

2.2 Test Materials

The test materials selected represent two of the structural and functional materials used for vehicle and equipment construction. The test materials are bare aluminum 7075 and CARC-painted aluminum (Figure 2.1). The biological agent test coupon dimensions are 2-cm by 1-cm. The chemical agent test coupons are 2-in. circular disks with a surface area of 3.14 in^2 (20.27 cm^2). The chemical aluminum coupons were used as received. The biological aluminum coupons were rinsed with 70% ethanol to clean the surface and dried completely before autoclaving. The CARC-painted coupons were primed in accordance with (IAW) MIL-P-53022B and painted IAW MIL-C-53039B, color Green 383 to apply green CARC paint. The top and sides of each coupon were painted.

Figure 2.1: Photograph of Test Coupons



2.3 Chemical Agent

The chemical agents used included HD, VX and GD. Each agent was obtained from the ECBC Chemical Transfer Facility. High purity GD (Lot No. GD-U-2323-CTF-N) and VX (Lot No. VX-U-5055-CTF-N) were used. No impurities were detected by ^{31}P-NMR in the VX and GD used for this study. CASARM-grade HD (Lot No. HD-U-9040-CTF-N) was used. The certificate of analysis indicated that the purity of HD was 98.2 ± 0.01 mole percent as determined by freezing point depression.

2.4 Biological Agent

Bacillus anthracis spores will be used for the biological testing. The spores used for this study will be prepared from plasmid-free avirulent strain of *B. anthracis* (NNR1Δ1). The spores were prepared in-house by the BioDefense Team, Biosciences Business Area, R&T Directorate.

2.5 Stirred Reactor Test

Reactions and Sample Collection: The stirred reactor test station was equipped with reactor vessels, temperature control and monitoring devices, and mechanical stirrers. Reactions were performed in jacketed 100 mL glass vessels fitted with Heidolph RZR2051 mechanical stirrers. A Polyscience Model 9110 bath circulator maintained the reactor temperature. A Digisense Scanning Thermometer interfaced with a computer equipped with Windows 95 and Scanlink 2001 software (Barnant/Cole-Parmer Instrument Company) monitored and recorded temperature during the course of the reaction. The test station had three vessels enabling simultaneous testing in triplicate. An illustration of the test station is shown in Figure 2.2.

Figure 2.2: Stirred Reactor Test Set-Up

Reactions were initiated by adding 50 mL of decontaminant (CDS or DF200) to each of three reactor vessels. The decontaminant was then stirred at a speed of 350 rpm and allowed to thermally equilibrate at 25 °C. Afterwards, 1.00 mL of agent (GD, HD or VX) was pipetted into the first reactor, and a timer was started.

Fifty-microliter samples were removed from reactor vessels at selected intervals (Table 2.1) and prepared for residual agent analysis by GC-AED. After being removed from the reactor, samples were immediately transferred to vials containing the appropriate agent quench solution and chloroform. For GD and HD, the quench solution was 0.2 M sodium sulfite in water. For VX, the quench solution was 0.2 M sodium sulfite and 0.2 M sodium carbonate in water. Sodium sulfite was used to destroy residual oxidant. Sodium carbonate facilitated extraction of VX into chloroform by keeping the amine group of VX in the free base form.

The collected samples were vigorously agitated using a Vortex Genie mixer. Then the aqueous and non-aqueous phases were allowed to separate before a micropipette was used to transfer a sample of the non-aqueous (chloroform) layer to a GC vial.

The agent addition and sample collection times for the other two reactors were staggered 11 min apart from the first reactor enabling simultaneous testing of three replicates. Table 2.1 shows the sampling times for the three reactors.

Table 2.1: Agent Addition and Sampling Times for Reactors 1, 2, and 3

	Reactor 1	Reactor 2	Reactor 3
Start Time (min) (Addition of Agent)	0	11	22
Sampling Times (min)	2	13	24
	3	14	25
	4	15	26
	5	16	27
	10	21	32
	20	31	42
	30	41	52
	40	51	62
	50	61	72
	60	71	82

Samples were also collected from reactors for analysis by nuclear magnetic resonance (NMR) spectroscopy. At the end of the each reaction, a sample was collected from the first reactor and placed in a 5-mm NMR tube.

GC-AED Analysis: GC analysis was performed on a Hewlett Packard (HP) 6890 gas chromatograph equipped with a HP 6890 auto sampler and G2350A Atomic Emission Detector (GC-AED). An RTx-1 column (30m X 0.32 mm X 1 μm df) with ultra high purity helium as the carrier gas was employed. The carbon channel was used in the analysis of all three agents. Phosphorus was used only in the analysis of GD and VX samples. Sulfur was used only for HD and VX analysis, and fluorine was used only for GD analysis. The wavelength (nm) for each channel and other GC-AED parameters are listed in Table 2.2.

Table 2.2: GC-AED Parameters for Analysis of Reactor Samples

PARAMETERS	AGENT ANALYZED		
	VX	HD	GD
Initial Oven Temperature (°C)	45	45	45
Initial Time (min)	3	3	3
Rate 1(°C/min)	20	20	20
Final Oven Temperature 1 (°C)	180	150	150
Final Time 1 (min)	0	0	0
Rate2 (°C/min)	40		
Final Oven Temperature 2 (°C)	260		
Final Time 2	0		
Injection Port Temp	250	250	250
Injection Port Pressure	20	20	20
Column	HP-1; 25 X 0.32 X 0.52 mm		
AED Specific Parameters:			
Element Groups	Carbon 193 Sulfur 181 Phosphorus 178	Carbon 193 Sulfur 181 Chlorine 479	Carbon 193 Phosphorus 178 Fluorine 690
Transfer Line Temperature (°C)	250	250	250
Cavity Temperature	250	250	250

NMR Analysis: One-dimensional NMR analysis of agent feedstock and reactor samples was performed using a Varian 400-MHz narrow bore spectrometer equipped with a 5-mm broad band liquids probe. Agent feedstock samples were prepared by adding 10 µL of agent to 750 µL of deuterated chloroform ($CDCl_3$), then doubly containing the sample within two NMR tubes. Reactor samples were prepared by adding 690 µL of sample from the first reactor to a 5-mm NMR tube containing 77 µL of D_2O, which was used to facilitate shimming. Spectra were referenced externally using H_3PO_4 for ^{31}P-NMR, TMS for ^{1}H-NMR, and 1, 4-dioxane for ^{13}C-NMR. Shifts for the references are listed in Table 2.3. All peak identifications were based on reference to previous experimental data.

Table 2.3: Chemical Shifts of External Reference Standards

NUCLEUS	EXTERNAL REFERENCE	CHEMICAL SHIFT (PPM)
^{1}H	TMS	0.00
^{31}P	H_3PO_4	0.00
^{13}C	1,4- Dioxane	69.3

2.6 Chemical Agent Panel Test: Preparation, Contamination, Decontamination

Conditioning of Panels: The panel test coupons were either 2-in. diameter aluminum or CARC-painted aluminum disks. Tests were conducted in triplicate. Test coupons were placed on clean aluminum foil and thermally equilibrated to approximately 20 °C.

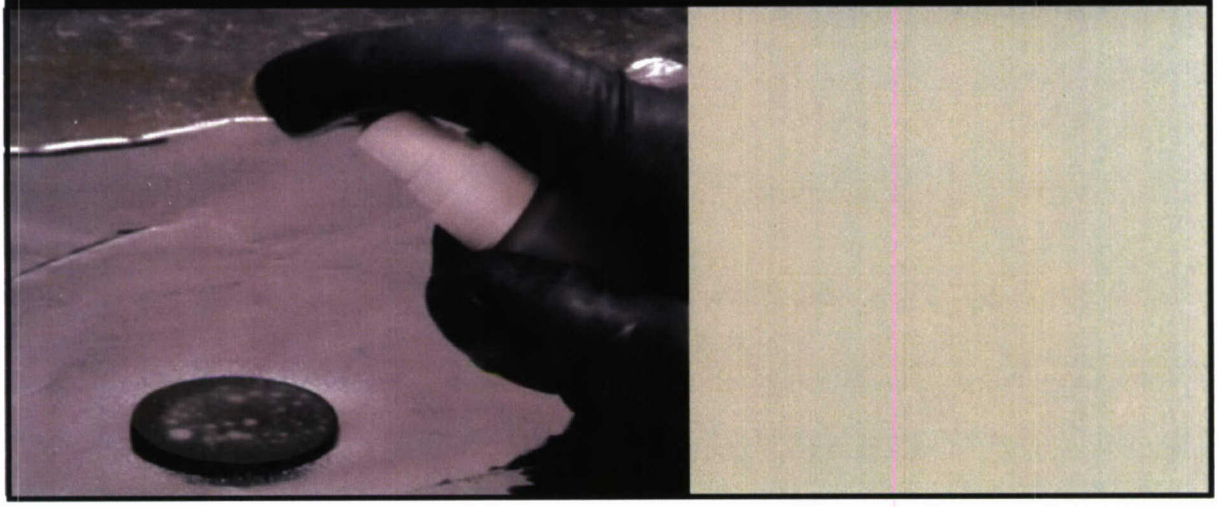
Contamination of Panels: The chemical agent challenge was approximately 1 g/m^2. The agent was applied to the coupons as two 1-µL drops approximately .5 in. apart in the center of the coupon. Drops were applied with an Eppendorf repeater pipette model # 4780 set at 1 and fitted with 0.05-mL Combitips and 10 µL ultra microtips. Droplets were not artificially spread. Contaminated panels were covered with small Petri dish covers to minimize agent evaporation. Coupons were aged for 60 min prior to application of decontaminant.

Preparation of Decontaminant Solutions: Decontaminant solutions were prepared 15 min prior to being used. Additionally, the pH of CDS solution was measured to confirm pH was within the 9.3 to 9.9 range.

Decontamination with CDS: CDS solution was mist-sprayed onto the contaminated surface using a 30 mL finger pump sprayer provided by Clean Earth Technologies. The sprayer was pumped several times with CDS decon before each decon application to ensure sprayer was primed with liquid and bubbles in the line were eliminated.

After the priming process, the cover was removed from the test coupon. The sprayer was held at a 45° angle with orifice approximately 3 in. above surface. The solution was sprayed using 5 pumps of the finger-pump spray bottle. After 5 min, an additional 4 pumps of solution were applied. An illustration of the spray application is shown in Figure 2.3. Finally, the sample was covered with a Petri dish cover and allowed to stand for a total of 10 or 20 min depending on desired decontamination contact period.

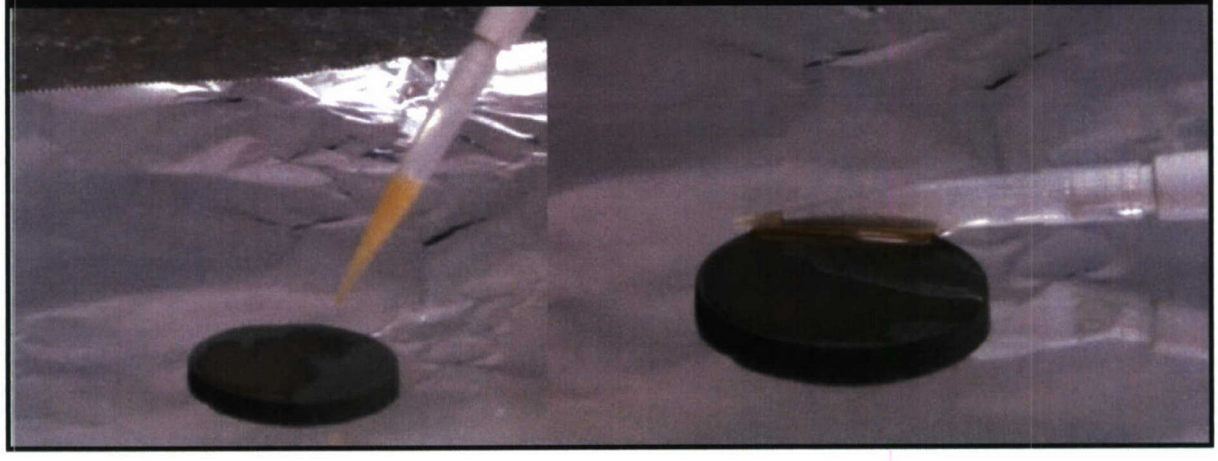
Figure 2.3: Mist Finger Pump Spray Application of CDS on Coupon



After decon application was completed, the spray bottle reservoir was rinsed with water several times. Water was sprayed through the sprayer to remove residual CDS decon. The sprayer was pumped several times to expel all water from the pump assembly before storage.

Decontamination with DF200: 1 mL of DF200 decontaminant was pipetted onto the contaminated surface using an Eppendorf 1000 reference pipettor. The pipettor was oriented parallel to the surface, and the edge of the pipette tip was used to coax the decon solution (using surface tension) to completely cover the contaminated area including the coupon edges as needed. An illustration of the application and spreading is shown in Figure 2.4. Panels were covered with Petri dishes and allowed to stand for 10 or 20 min depending on desired decontamination contact period

Figure 2.4: Pipette Application and Spreading of DF200 on Coupon



Post-Decontamination Treatment: Following the prescribed decon period, the excess decon on the surface was poured off and then rinsed with 40 mL of water (2 rinses of 20 mL each) using Dispensette III. The back of each panel was rinsed with 20 mL of water also. Excess water was forced off the panel by sharply tapping the panel once or twice in a vertical orientation. Each panel was oriented in a near vertical position for 2 min to allow remaining water to run off and the panel to air dry.

The residual water remaining on CARC panels formed a thin film which enabled drying within two minutes.

The residual water remaining on aluminum panels did not spread out evenly over the surface as on CARC and therefore impeded quick drying. To facilitate quicker water evaporation from aluminum panels, the water remaining on the aluminum panels was very lightly blotted with clean lab tissues to remove excess water and to allow water to completely evaporate before testing. Clean tissues were used for each panel.

2.7 Chemical Agent Panel Test: Contact Hazard Assessment

Contact Hazard Assessment: Following the contamination, decontamination and drying procedures, the panels were assessed by either the contact hazard or vapor hazard procedures to determine decontamination efficacies.[6] The contact hazard was measured by placing panels on 30 °C ± 3 °C temperature-regulated surface. A latex disk, aluminum foil (to prevent contamination of the contact weight) and 1-kg weight (with foam bottom) were placed on dried panel for 15 min to mimic a hand touching the surface. The latex and aluminum foil were removed after 15 min and extracted in 20 mL of chloroform (with 1 mL thiolane/liter to quench any remaining peroxide) for one hour. The panel was covered with an inverted Petri dish during the 15-45 min time interval when panel was not being sampled. The contact hazard test procedure was repeated for the 45-60 min time interval.

Following contact hazard tests, each panel was extracted in a dish with 20-mL chloroform (and 1-mL thiolane/liter to quench any remaining peroxide) for 65 min to remove any residual agent.

Non-contaminated panels, latex with aluminum, and 20 µL of each decon were individually extracted to check for possible interferences that may co-elute with the agents. All samples were analyzed undiluted using GC/FID and compared to external standards. Standards containing 0.05 to 500 g of agent per mL were prepared in chloroform.

GC analysis of extracts was performed on an Agilent 6890 gas chromatograph equipped with an Agilent 7683 series auto sampler and Flame Ionization Detector (GC-FID). An HP-5 column (30m X 0.53 mm X 1.5 µm film) with ultra high purity helium as the carrier gas was employed. The other GC-FID parameters are listed in Table 2.4.

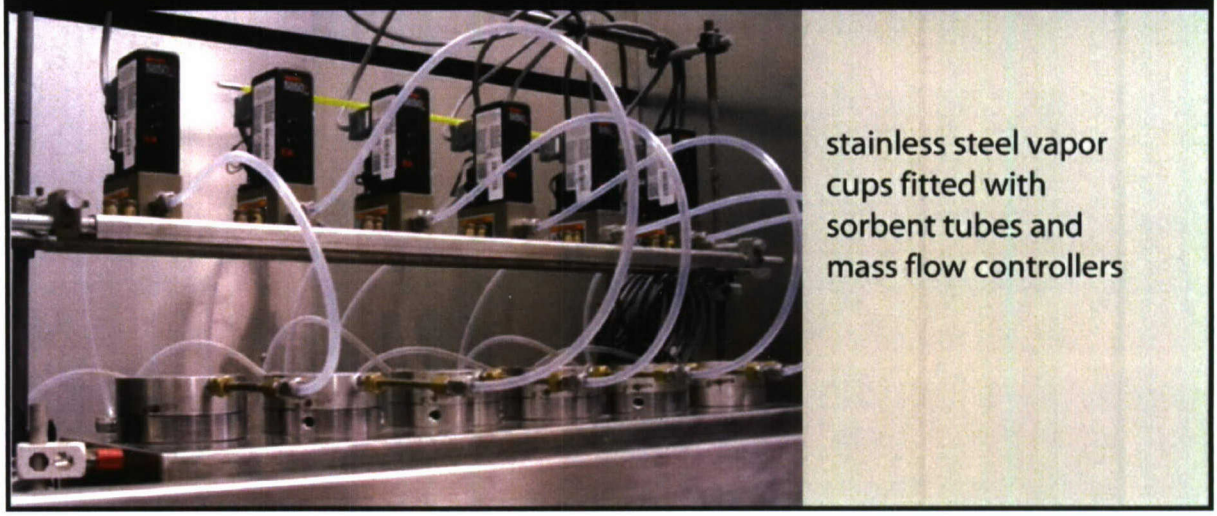
Table 2.4: GC-FID Parameters for Analysis of Contact Hazard Samples

PARAMETERS	AGENT ANALYZED		
	VX	HD	GD
Syringe Washes with Sample	2	2	2
Syringe Pumps with Sample	3	3	3
Injection Volume (µL)	1	1	1
Post Injection Syringe Washes with acetonitrile	2	2	2
Injection Port Temperature (°C)	220	220	250
Injection Mode	splitless	splitless	splitless
Column	HP-5; 30m X 0.53mm dia. X 1.50 µm film		
Carrier Gas Type	He	He	He
Gas Flow (mL/min)	2.9	2.9	2.9
Flow Mode	Constant	Constant	Constant
Gas Velocity (cm/sec)	23	23	23
Initial Oven Temperature (°C)	60	60	60
Initial Time (min)	1	1	1
Rate (°C/min)	20	20	20
Final Oven Temperature (°C)	240	240	240
Final Time (min)	10	3	5
Run Time (min)	20	13	15
Detector	FID	FID	FID
Detector Temperature (°C)	250	250	250

2.8 Chemical Agent Panel Test: Vapor Hazard Assessment

Vapor Sample Collection: A vapor manifold, illustrated in Figure 2.5, is used to collect the agent off gas after a coupon is decontaminated, rinsed, and dried. The vapor manifold is comprised of 6 vapor cups that hold 1 coupon each. A DAAMS tube is attached to each of the cups. Air is drawn across the coupon and through the tube at a set flow rate (300 mL/min) as regulated by a vacuum backed by a Brooks 5850E Mass Flow Controller (MFC). Tubes are manually changed at specified intervals; blank tubes (tubes that are not analyzed) are used between sampling intervals to maintain a continuous air flow. At least six tubes will be pulled over a six hour period for each cup. As soon as a tube is removed from the system, it is sealed with diff-lok end caps and stored for analysis within 24 hr.

Figure 2.5: Vapor Sampling Test Set-Up



For the analysis of GD and VX, the agent vapor was passed through and trapped in a Supelco ATD stainless steel tube, which was packed with Chromasorb 106 and placed immediately down flow from the vapor cup. A 3/8 in. diameter V-to-G conversion pad containing AgF from CMS Field Products was placed in line immediately before the Chromasorb tube to convert the VX vapor to its G-analog, ethyl methylphosphonofluoridate, for analysis.

For the analysis of GD and VX, tubes were changed manually at 0.5-, 1-, 2-, 4-, and 6-hr to cover a six hour period. Both ends of sorbent tubes were sealed with friction fit end caps immediately after removal from vapor cup. Tubes were then placed on the thermal desorber carousel. Agent trapped on sorbent tubes was thermally desorbed from sorbent by a Perkin Elmer Automated Thermal Desorber (ATD) TurboMatrix and quantitated using an Agilent 6890N GC-FID. The GC-FID parameters are listed in Table 2.5.

Table 2.5: ATD/GC-FID Parameters for Analysis of Vapor Samples

PARAMETERS	AGENT ANALYZED	
	VX	GD
VAPOR CAPTURE		
Air Flow (sccm)	300	300
V-to-G Conversion Pad used?	yes	no
Sorbent Type	Chromasorb 106	Chromasorb 106
THERMAL DESORBER		
Desorb Time (min)	5	5
Desorb Flow (mL/min)	30	30
Inlet Split (mL/min)	40	40
Outlet Split (mL/min)	50	50
Trap Temp (°C)	300	300
Trap Desorb Temp (°C)	254	254
Trap Desorb Time (min)	1	1
Valve Temp (°C)	200	200
Transfer Line Temp (°C)	200	200
GAS CHROMATOGRAPH		
Inlet Temperature (°C)	180	180
Injection Mode	split	split
Column		
Carrier Gas Type	He	He
Gas Flow (mL/min)	0.2	0.2
Initial Oven Temperature (°C)	30	60
Initial Time (min)	1	1
Rate (°C/min)	20	20
Final Oven Temperature (°C)	240	240
Final Time (min)	5	5
Run Time (min)	16.5	15
Detector	FID	FID
Detector Temperature (°C)	250	250

For the GD and VX analysis, peak areas were compared to areas obtained from external standards applied to similar sorbent tubes. Sorbent tubes for GD calibration were prepared by pipetting 10 µL of standards prepared in acetonitrile into the sorbent tube. Air flowing at 3 sccm for 3 min was used to pull the agent into the sorbent. Sorbent tubes for VX calibration were prepared similarly, but the standard solution was pipetted onto a V-to-G conversion pad positioned immediately before the sorbent tube, and a 20 min flow time instead of a 3 min flow time was used.

The analysis of HD was conducted using tubes of 6 mm diameter stainless steel DAAMs tubes packed with a solid sorbent, Tenax TA. The DAAMs tubes are analyzed using a Marks UNITY/ULTRA thermal desorption system coupled to an Agilent 6890N gas chromatography-mass selective detector (GC-MSD). The instrument reports the mass of agent on the tube in nanograms (ng).

Data Analysis: Vapor data are presented in units of mg/m^3. The vapor concentration is calculated using the mass of agent collected on the vapor tube and the volume of air that passed through the tube during collection. The mass of agent on a tube is derived from the GC data. The volume of air is calculated by multiplying the air flow rate set by the MFCs by the amount of time the tube is attached to the vapor cup. Depending on the agent, material properties and experimental time tubes are attached to the vapor cup for various amounts of time (e.g. shorter times for high concentrations, longer times for lower concentration).

2.9 Biological Agent Test Procedures

Bacterial Strains and Media: Plasmid-free avirulent strain of *B. anthracis* (NNR1Δ1) was used for this study. The cells were grown at 37 °C in tryptic-soy broth (TSB) or tryptic-soy agar (TSA). Frozen stock of bacterial cells, stored at -80 °C in TSB supplemented with glycerol to a final concentration of 20% (v/v), were streaked for isolated single colonies onto TSA plates and incubated over-night at 37 °C. Following the appearance of distinct colonies, a single colony was inoculated into 50 mL of TSB and grown to early stationary phase (at 37 °C for 36-48 hr). The spores of this strain were prepared using this broth culture.

Spore Preparation: An aliquot of 1.0 mL bacterial culture was spread onto the surface of Lemko Sporulation Medium (LSM; large plates) and incubated at 37 °C. A set of 4 plates was wrapped together with parafilm. The plates were inverted and incubated at 37 °C for a period of 72-144 hr (depending on the progression of sporulation on each plate, which is assessed by wet mount of microbial growth on each of the plates). If the sporulation was assessed to be >85%, the plate was pulled out for harvest. Plates with >85% sporulation were removed from the incubator and placed at 4 °C for 1.5 - 2 h.

A 25-mL aliquot of sterile distilled H_2O was transferred onto the plate. Using a sterile cell spreader, the microbial culture was dislodged off the surface. The suspended culture from all plates was pooled in the same centrifuge bottle. The spore suspension was mixed thoroughly after closing the lid tightly. The spores were pelleted by centrifugation and washed once in 100 mL of sterile distilled H_2O. The pellet was suspended in 100 mL 70% ethanol and allowed to stand at room temperature for 1 hr. The spore pellet was resuspended in 100 mL sterile distilled H_2O and heat-treated at 65 °C for 30 min.

The spore suspension was enumerated and diluted to 4×10^8/mL. Equal volumes of spore suspension and 1% serum protein were mixed to generate working stock of 2×10^8/mL containing 0.5% serum protein. An aliquot of 50-µL spore suspension containing ~1×10^7 spores was inoculated on each coupon.

Coupon Handling & Inoculation: Metal coupons in 1-cm by 2-cm size, unpainted and CARC-painted, were washed in 70% ethanol before use. In addition, glass coupons were used in

some preliminary experiments to address the recovery from hard surface and the responsiveness of spores to different dose of bleach. The coupons were autoclaved in glass Petri-plates with painted surface on top. The top surface was inoculated with 50-µL spore suspension as prepared in section 5.1.2. The inoculated coupons were dried in a BSL-2 cabinet with lid open over-night before disinfection experiments.

Test Chemical Preparation: Two disinfectants, Ultra Clorox bleach (~6%) and Clean Earth Technology (CET, ~24%), were compared in this study. The active component in bleach is hypochlorite (OCl^-), and the active component in CET is hydrogen peroxide. The working stock was a 4% concentration for Peridox™ and a 6000-ppm concentration for bleach. Prior to initiating disinfection experiments, a quantitative method to confirm the concentration of active ingredient in both test chemicals was used. The quantitative methods used for measuring the available chlorine in bleach and hydrogen peroxide in Peridox™ are briefly described below:

A) Hydrogen Peroxide Concentration Determination in Peridox™

Solutions and Chemicals:

1) Acid solution - 0.18 g ammonium molybdate was dissolved in 750 mL of distilled water, followed by slow addition of 300 mL of conc. sulfuric acid. Store in a sealed beaker
2) Peridox™ stock - Dilute (1:10) CET in water
3) Titrant - 0.1 N sodium thiosulfate
4) Potassium iodide crystals

Procedure:

To 50 mL of water in a beaker, add 1 mL of dilute CET followed by 2.5 mL of acid solution, and then 0.6 g of potassium iodide crystals. Titrate this mix with 0.1 N sodium thiosulfate until the solution turns colorless. Record the volume of titrant consumed.

Calculation:

H_2O_2 (g/L) = $(A \times N \times D \times 17.007)/V$

A= titrant volume consumed
N= normality of titrant
D= dilution factor of Peridox™ stock
V= Peridox™ stock volume used
17.007= Equivalent weight of H_2O_2 (2 moles of thiosulfate reacts with 1 mol of peroxide)

B) Hypochlorite Determination in Bleach

Solutions and Chemicals:

1) Acid - 6 N HCl
2) Bleach stock - Dilute (1:10) bleach in water
3) Titrant - 0.1-N sodium thiosulfate
4) Potassium iodide crystals

Procedure:
To 50 mL of water in a beaker, add 5 mL of dilute bleach solution, followed by 10 mL of acid, and then 1 g of potassium iodide. Titrate this mix with 0.1 N sodium thiosulfate until the solution turns colorless. Record the volume of titrant consumed.

Calculations:
Available OCl^- (g/L) = $(A \times N \times D \times 34.23)/V$

A=	titrant volume consumed
N=	normality of sodium thiosulfate
D=	dilution factor of Bleach stock
V=	Bleach stock volume used
34.23=	Equivalent weight of NaOCl (2 moles of thiosulfate reacts with 1 mol of hypochlorite)

Disinfectant Neutralization Confirmation: A solution of 1 M sodium thiosulfate was prepared in 1.0% buffered peptone water containing 0.5% tween-80 surfactant and used as the neutralizer. Equal volumes (5 mL each) of Peridox™ stock or bleach stock were mixed with the neutralizer. The presence of the oxidizing moiety was confirmed by addition of acid solution as described in section above and addition of potassium iodide crystals. No change in color (appearance of yellow-brown color) was observed, confirming complete absence of oxidizing active ingredient, i.e. peroxide in Peridox™ and hypochlorite in bleach. Additionally, the residual peroxide presence was also tested using peroxide test strip (EM Quant, Germany; catalog # 10011-1)

Decontamination Testing: A Single Tube Method (STM) for bio-decontamination efficacy was used for this study. Two types of controls were used in this study. A negative control (uninoculated coupons) and a positive control (inoculated but not treated with either disinfectant) were set up in addition to test samples treated with 6000-ppm of bleach or 4% Peridox™ for a period of 15 min. Inoculated coupons were placed in sterile 50 mL blue-cap tubes. Five milliliters of sterile water (controls) or same volume of disinfectant was added to the tubes containing the test coupons. After 15-min incubation at room temperature (23 $\pm$ 1 °C), 5 mL of neutralizer, i.e. 1M sodium thiosulfate (prepared in 0.5% buffered peptone water containing 0.5% tween-80 surfactant). The tubes containing the coupons were sonicated for 10 min (Branson sonicator) at 25 °C and vortexed for 2 min @1500 rpm for dislodging the spores off the coupons.

The spores released off the coupons were enumerated by dilution plating (ten-fold serial dilution up to 10^{-4}). For control samples, an aliquot of 100-µL from 10^{-3} and 10^{-4} dilution tubes was transferred on three replicate plates and spread-plated. The plates were incubated at 37 °C over-night. For treated samples, in addition to spread plates from dilution tubes between 10^{-1} and 10^{-3}, pour-plating from 10^{-1} was also performed. For pour-plating, an aliquot of 1 mL sample was pipetted to each of the three plates and ~20-23 mL of liquid TSA media (maintained at 55°C) was added. The plates were swirled to mix and let set for 3 hr at room temperature before incubation at 37 °C for up to a period of 6 days.

The colony-forming units (CFU) on spread plates were counted by QCount™ (>30 CFU) or manually (<30 CFU). The CFU on pour-plates were observed first after 24 hr and then final counts were recorded after 6-day incubation.

In treated samples, since a low number of viable spores was expected, an aliquot of 3 mL out of a total of 10 mL sample was analyzed. If no colony was observed in $1/3^{rd}$ of the fraction analyzed, the limit of detection (LOD) in this study, therefore, is construed to be 1-5 viable spores. If no colonies were observed in a sample, a fixed value of 2 was substituted for that sample.

Data Handling and Reduction: The CFU observed on triplicate plates for control and treated samples were averaged and multiplied by a volume factor, 10 (since 100 µL was plated out of a 1 mL diluted sample), dilution factor, 10^3 or 10^4 (for controls), and appropriate dilution factor for treated sample. The $\log_{10}$ of the total CFU were computed for each replicate control and treated sample. Standard deviation was computed for all five replicate samples. Kill efficacy was estimated in terms of Log Reduction (LR) by subtracting the log $CFU^{treated}$ from log CFU^{contol} values.

3. STIRRED REACTOR TEST RESULTS AND DISCUSSION

3.1 GD Test Results

GD was rapidly destroyed by CDS and DF200. No GD was detected by GC-AED in either the first sample of CDS-GD reaction mixture or the first sample of the DF200-GD reaction mixture. The first samples of both mixtures were collected at 2 min.

Because DF200 and CDS destroyed GD very quickly, no half-life values for GD in the presence of either decontaminant could be calculated. The amount of GD detected according to GC-AED channel and time for CDS and DF200 samples is included in Appendices A1-A6.

NMR analysis of both reaction mixtures confirmed the absence of GD and the presence of the GD hydrolysis product pinacolyl methylphosphonic acid (GD-acid). ^{31}P-NMR spectra of the CDS-GD reaction mixture showed a singlet representing GD-acid at δ17 ppm (Fig. 3.1), while spectra of DF200-GD reaction mixture showed this singlet at δ25 ppm (Fig. 3.2). The component detected at δ12 ppm in the CDS-GD reaction mixture (Fig. 3.2) was originally present in the CDS. ^{1}H-NMR spectra of the CDS-GD and DF200-GD reaction mixtures are located in Appendices B1 and B2.

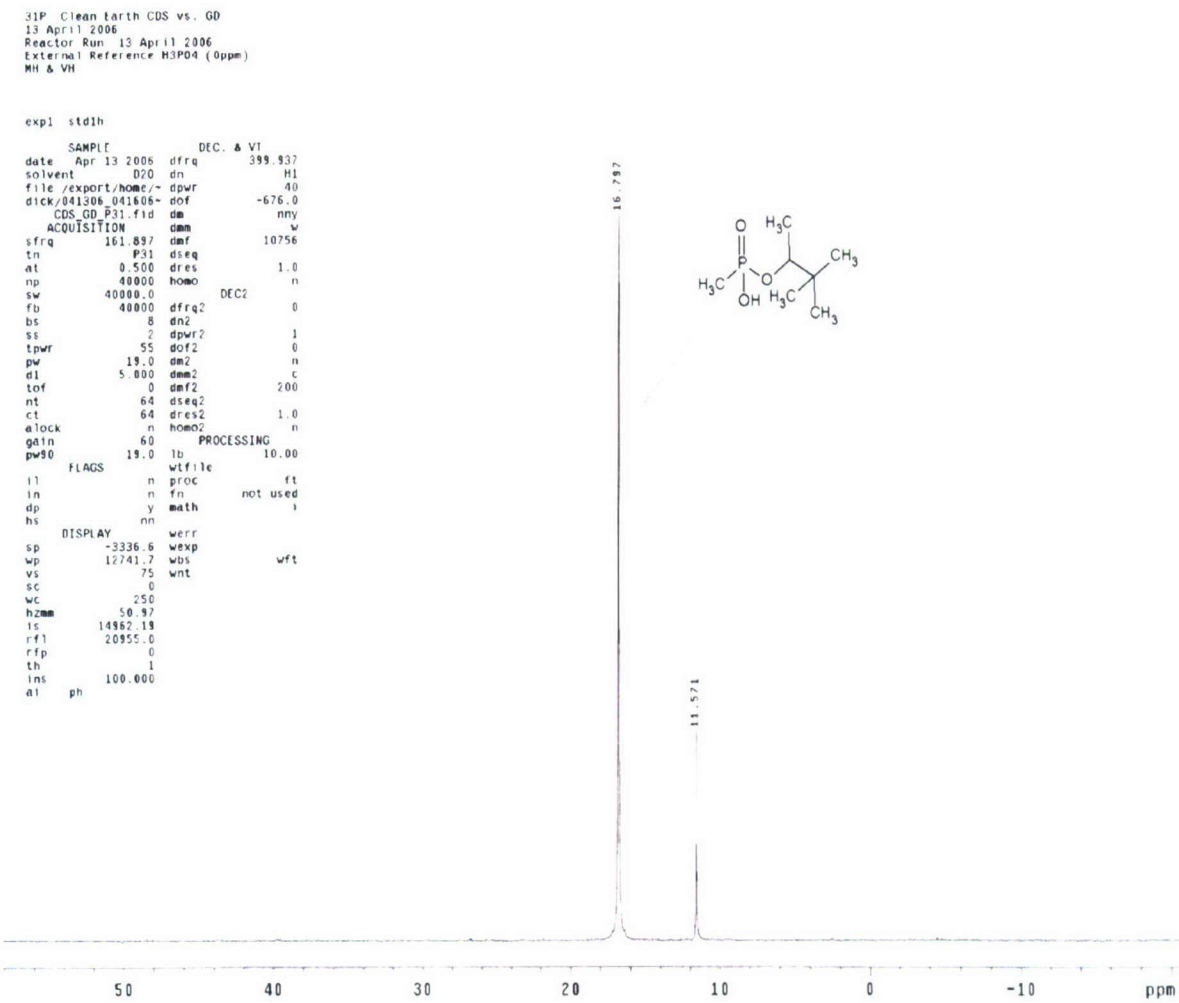


Figure 3.1: ^{31}P-NMR Spectra Showing Pinacolyl Methylphosphonic Acid as the Major Product of GD Decontamination with CDS

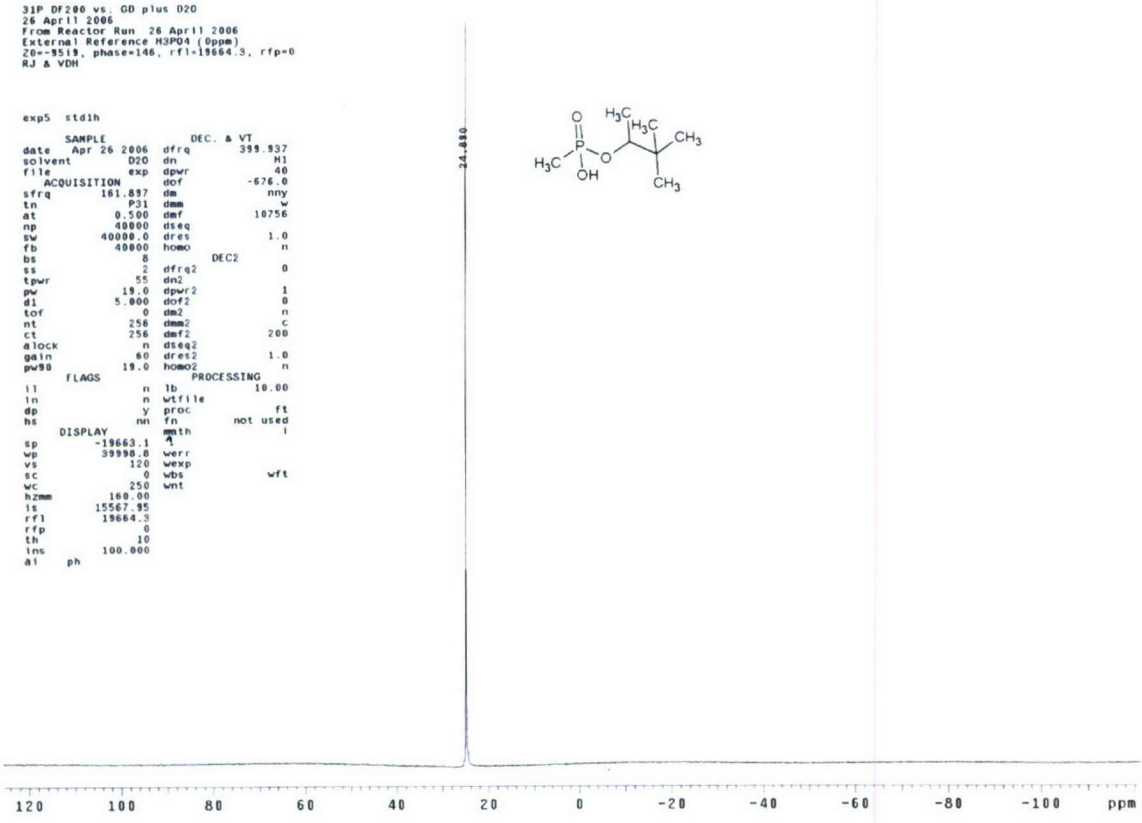


Figure 3.2: ^{31}P-NMR Spectra Showing Pinacolyl Methylphosphonic Acid as the Major Product of GD Decontamination with DF200

3.2 HD Test Results

CDS: No HD was detected by GC-AED in the CDS-HD reaction mixture after 10 min. (At 10 min, approximately 1% of the initial HD concentration was detected in each reactor; at 20 min, none was detected.) The amount of HD detected in each CDS sample versus time and according to GC-AED channel is included in Appendices A7-A9.

Considering data collected on carbon, sulfur, and chlorine AED channels, the average half-life for HD in CDS at 25 °C was 1.42 ± 0.10 min. Calculated half-life values for each reactor and channel are listed in Table 3.1. The calculated half-life values varied among the channels for each reactor—the sulfur channel tended to yield the highest values. Calculated half-life values based on carbon and chlorine channels data were approximately 1.40 min while values based on sulfur channel data were approximately 1.50 min.

Table 3.1: Calculated Half-Life Values by Reactor and Channel for HD Treated with CDS

AED CHANNEL	CALCULATED HALF-LIFE (MIN)			AVERAGE HALF-LIFE (MIN)
	Reactor 1	Reactor 2	Reactor 3	
Carbon (193 nm)	1.39	1.34	1.40	1.38 ± 0.03
Sulfur (181 nm)	1.48	1.59	1.55	1.54 ± 0.06
Chlorine (479 nm)	1.30	1.35	1.40	1.35 ± 0.05

The ^{1}H-NMR spectrum of the CDS-HD reaction mixture showed that no HD was present. Detected compounds appeared to be hydrolyzed versions of sulfoxide and sulfone: 2,2'-sulfinyl diethanol and 2,2'-sulfonyl diethanol. Since the sample was not analyzed immediately, the detected products may not be the initial reaction products (Figure3.3). A full ^{1}H-NMR spectrum of the CDS-HD reaction mixture is located in Appendix B3.

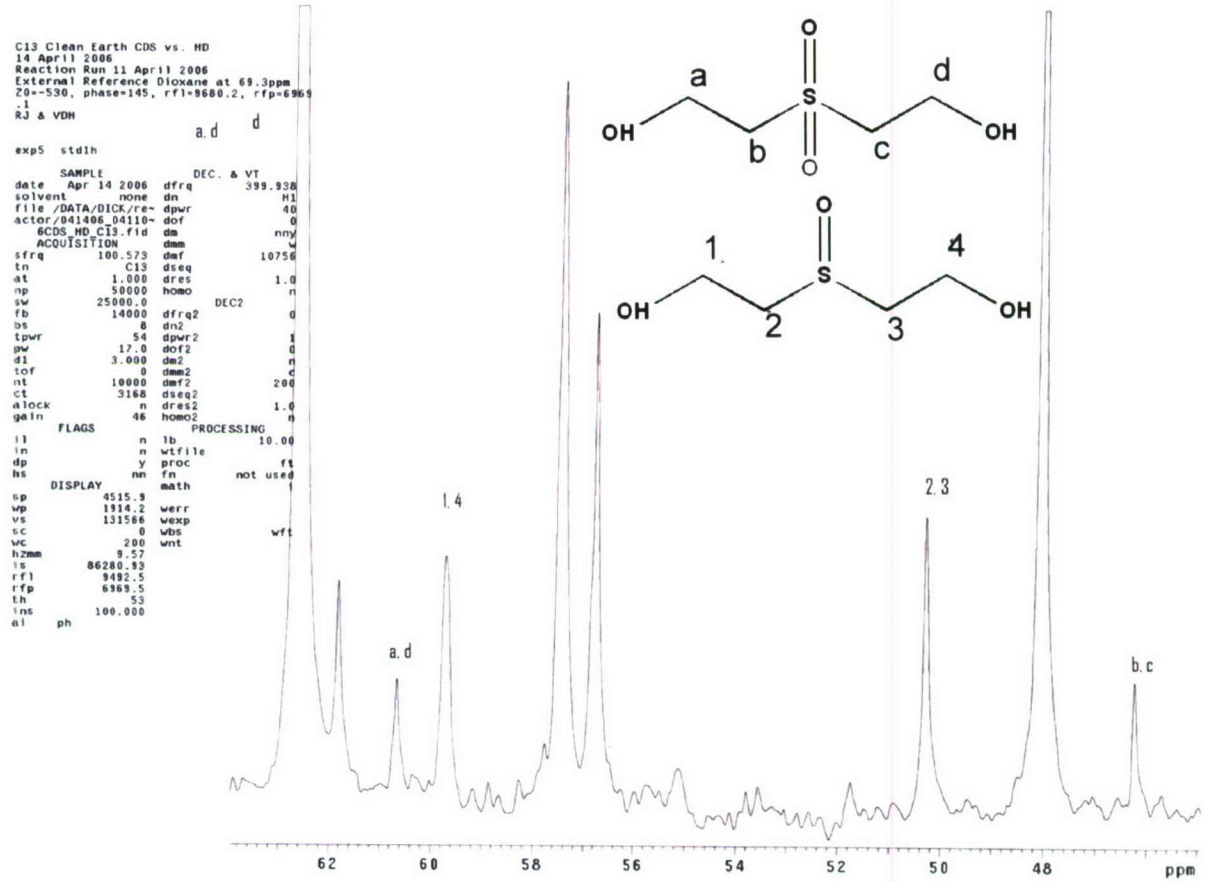


Figure 3.3: ^{13}C-NMR Spectra Showing 2,2'-Sulfinyl Diethanol and 2,2'-Sulfonyl Diethanol as Products of HD Decontamination with CDS.

DF200: HD was detected by GC-AED in all samples of the DF200-HD reaction mixture. During the sampling period, concentration of HD increased and decreased sporadically—indicating the presence of HD globules in the DF200 solution. At 1 h, an average of 2.12 ± 1.38% of the initial HD concentration was found in each reactor. The amount of HD detected in each DF200 sample versus time and according to GC-AED channel is included in Appendices A10-A12.

Due to the data scatter for the other two reactors, half-life could only be calculated for reactor 3 (Table 3.2). The average calculated half-life at 25 °C based on data from the chlorine, carbon and sulfur channels was 28.08 ± 2.39 min.

Table 3.2: Calculated Half-Life Values by Channel for HD Treated with DF200 in Reactor 3

AED CHANNEL	HALF-LIFE (MIN)
Carbon (193 nm)	25.9
Sulfur (181 nm)	30.6
Chlorine (479 nm)	27.6

A globule of an unidentified substance was found in one of the reactors at the end of the test period. The substance was later analyzed by GC-MS. Results showed that the substance contained a very small amount of HD and an unknown component or components.

^{1}H-NMR analysis of the DF200-HD reaction mixture revealed the presence of 2-chloroethylvinyl sulfone and divinyl sulfone (Figure 3.4). Full ^{1}H-NMR and ^{13}C-NMR spectra of the DF200-HD reaction mixture are located in Appendices B4 and B5.

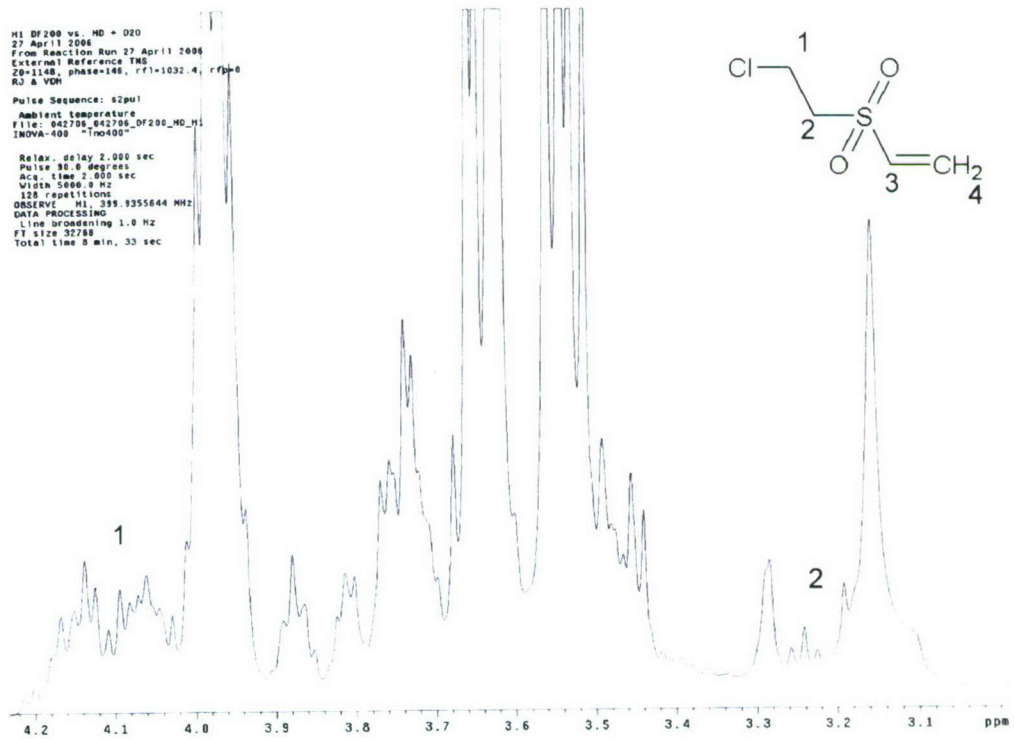


Figure 3.4: ^{1}H-NMR Spectra Showing 2-Chloroethylvinylsulfone as a Product of HD Decontamination with DF200

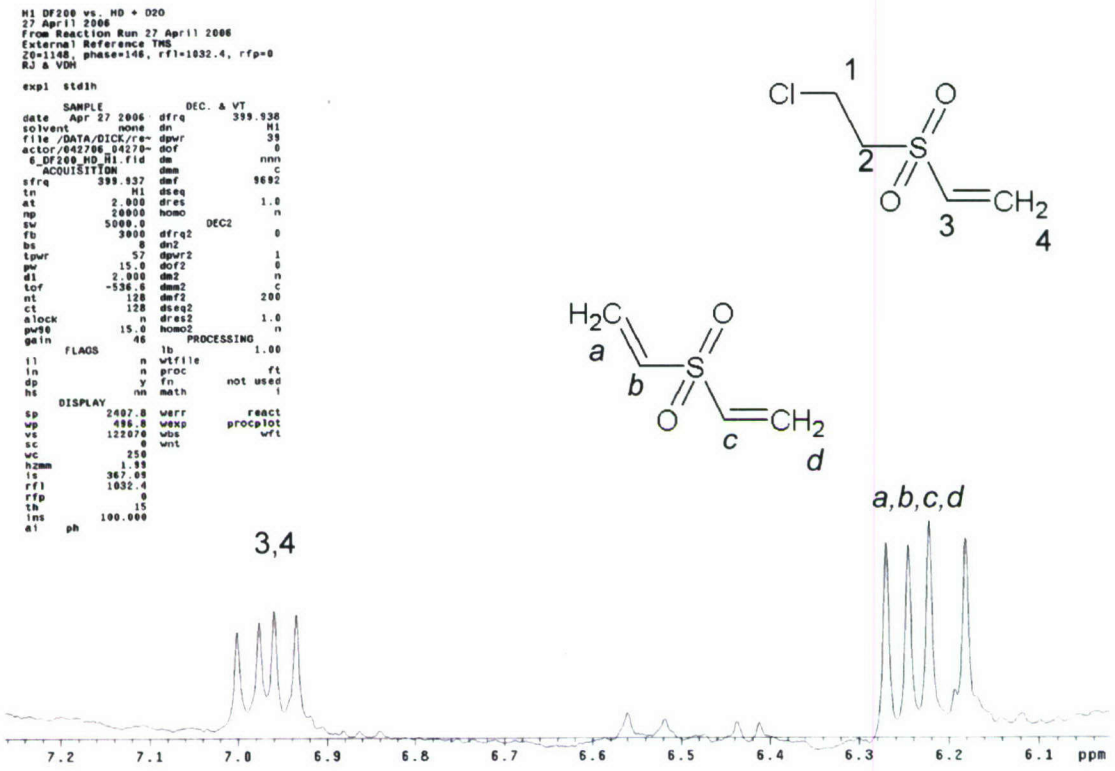


Figure 3.5: ^{1}H-NMR Spectra Showing 2-Chloroethylvinylsulfone and Divinyl Sulfone as Products of HD Decontamination with DF200

3.3 VX Test Results

CDS: No VX was detected by GC-AED in the CDS-VX reaction mixtures after 3 min. (An average of 0.04 % of initial VX concentration was detected in the reactors at 3 min; none was detected at 4 min.) The amount of VX detected versus time according to GC-AED channel is included in Appendices A13-A15.

Because of the rapid reaction between VX and CDS, only data at 2 min and 3 min could be used for the half-life calculation. Half-life values for each reactor and channel of analysis are shown in Table 3.3. Considering the data collected on the carbon, sulfur and phosphorus channels, the overall average half-life for VX in CDS at 25 °C was 0.40 ± 0.05 min.

Table 3.3: Calculated Half-Life Values by Reactor and Channel for VX Treated with CDS

AED CHANNEL	CALCULATED HALF-LIFE (MIN)			AVERAGE HALF-LIFE (MIN)
	Reactor 1	Reactor 2	Reactor 3	
Carbon (193 nm)	0.33	0.54	0.45	0.44 ± 0.11
Phosphorus (178 nm)	0.36	0.46	0.42	0.41 ± 0.05
Sulfur (181nm)	0.35	0.35	0.36	0.35 ± 0.01

No VX was detected by NMR in the reactor sample. A peak representing ethylmethylphosphonic acid (EMPA) was observed at approximately δ27 ppm in ^{31}P-NMR spectra of CDS reaction mixtures (Fig. 3.6). ^{1}H-NMR spectra for the CDS-VX reaction mixtures are located in Appendix B6.

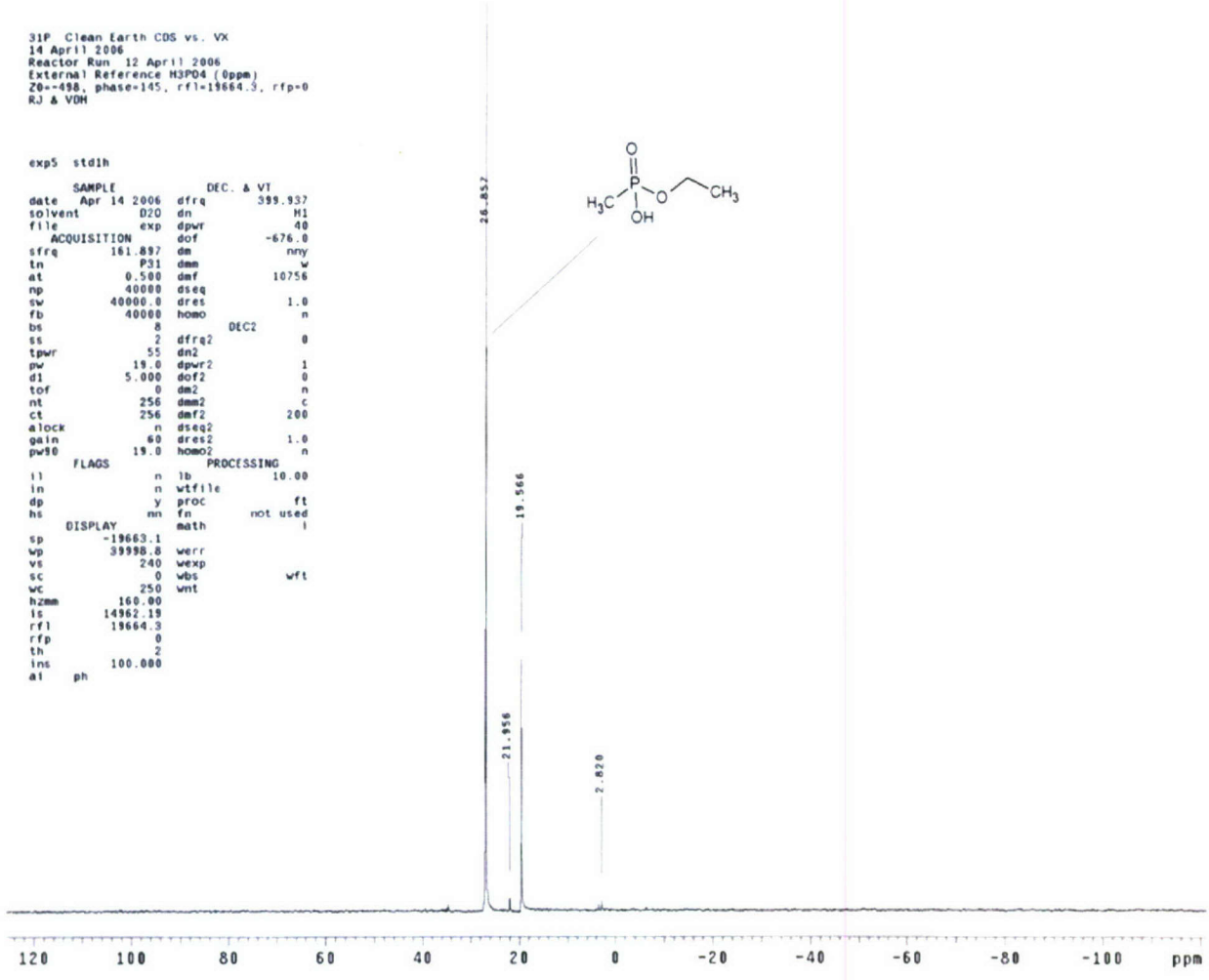


Figure 3.6: ^{31}P-NMR Spectra Showing Ethylmethylphosphonic Acid as the Major Product of VX Decontamination with CDS

DF200: VX was detected by GC-AED in the DF200-VX reaction mixture throughout the reaction period. The average amount of VX detected in the reactor samples at 30 min based on data from the sulfur and phosphorus channels was 0.70 ± 0.01% of the original concentration. The amount of VX detected in each DF200 sample according to time and GC-AED channel is included in Appendices A16-A18.

Considering the data collected on the sulfur and phosphorus channels, the overall average half-life for VX in DF200 at 25 °C was 5.17 ± 0.91 min. Data for each reactor and channel of analysis are shown in Table 3.4. Data from the carbon channel after 10 min of reaction time were not used in the calculation of the average overall half-life because of interference from a co-eluting peak.

Table 3.4: Calculated Half-Life Values by Reactor and Channel for VX Treated with DF200

AED CHANNEL	CALCULATED HALF-LIFE (MIN)			AVERAGE HALF-LIFE (MIN)
	Reactor 1	Reactor 2	Reactor 3	
Carbon (193 nm)*	2.93	3.10	2.49	2.84 ± 0.31
Phosphorus (178 nm)	6.28	4.31	4.94	5.18 ± 1.01
Sulfur (181nm)	6.34	4.41	4.75	5.17 ± 1.03

*Only the first five data points (data up to and including 10 min) used in half-life calculation.

No VX was detected by NMR in the DF200-VX reaction mixture. The NMR analysis was completed at least 4 hr after the start of the reaction. A peak representing ethylmethylphosphonic acid (EMPA) was observed at approximately δ27 ppm in the ^{31}P-NMR spectrum of DF200 reaction mixture (Fig. 3.6). A ^{1}H-NMR spectrum for the DF200-VX reaction mixture is located in Appendix B7.

3.4 Discussion

The purpose of this study was to evaluate the efficacy of CDS against HD, GD, and VX and to compare CDS to DF200. CDS destroyed all agent challenges in less than 20 min. The efficacy of CDS was comparable to DF200 for GD decontamination. CDS was more effective than DF200 in VX decontamination.

Judging by reaction rate, CDS was also more successful than DF200 in HD destruction. The reason for this success may be that CDS solubilizes mustard more effectively than DF200. The immediate reaction products of HD decontamination by CDS could not be identified with certainty. It is possible that CDS quickly produces some sulfone, which is a vesicant and therefore an undesired reaction product. Characterization of the CDS-HD reaction mixture at an earlier stage is necessary to identify initial HD reaction products.

4. PANEL CONTACT HAZARD TEST RESULTS

4.1 GD Contact Hazard Test Results

GD was recovered from the panels during the two 15 min contact periods following decontamination. The agent quantity recovered is expressed as milligrams GD per square meter of surface. The JPID threshold contact exposure level for GD is < 1.7 mg/m^2 (This value does not apply to surface residual measurements). Values above the threshold contact exposure level during the contact periods are shown in bold (Tables 4.1 and 4.2). The GD still associated with the panel following the two contact periods was also quantified and presented as the surface residual.

Table 4.1: GD Contact Hazards and Surface Residual (mg /m^2) on CARC and Aluminum after 10-Min Decontamination with CDS and DF200

Material/Decon	**0-15 min**	**45-60 min**	**Surface Residual**
CARC			
CDS	1.19 ± 0.93	1.35 ± 0.13	33.89 ± 10.58
DF200	**8.87 ± 1.54**	1.63 ± 0.09	52.95 ± 4.37
Aluminum			
CDS	0.30 ± 0.26	1.15 ± 0.07	0.80 ± 0.11
DF200	1.02 ± 0.04	1.36 ± 0.06	0.07 ± 0.08

Average of three replicates ± standard deviation

Table 4.2: GD Contact Hazards and Surface Residual (mg /m^2) on CARC and Aluminum after 20-Min Decontamination with CDS and DF200

Material/Decon	**0-15 min**	**45-60 min**	**Surface Residual**
CARC			
CDS	1.39 ± 1.01	1.13 ± 0.27	39.91 ± 17.50
DF200	**2.06 ± 0.65**	1.48 ± 0.08	61.49 ± 20.42
Aluminum			
CDS	0.29 ± 0.26	1.01 ± 0.12	0.76 ± 0.23
DF200	0.78 ± 0.11	1.28 ± 0.04	0.10 ± 0.10

Average of three replicates ± standard deviation

4.2 HD Contact Hazard Test Results

HD was recovered from the panels during the two 15 min contact periods following decontamination. The agent quantity recovered is expressed as milligrams HD per square meter of surface. The JPID threshold contact exposure level for HD is < 3.0 mg/m^2 (this value does not apply to surface residual measurements). Values above the threshold contact exposure level during the contact periods are shown in bold (Tables 4.3 and 4.4). The HD still associated with the panel following the two contact periods was also quantified and presented as the surface residual.

Table 4.3: HD Contact Hazards and Surface Residual (mg /m^2) on CARC and Aluminum after 10-Min Decontamination with CDS and DF200

Material/Decon	**0-15 min**	**45-60 min**	**Surface Residual**
CARC			
CDS	**5.60 ± 1.47**	0.87 ± 0.10	26.19 ± 2.66
DF200	**12.65 ± 3.17**	2.12 ± 0.42	34.33 ± 6.01
Aluminum			
CDS	0.00 ± 0.00	0.00 ± 0.00	0.99 ± 0.19
DF200	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00

Average of three replicates ± standard deviation

Table 4.4: HD Contact Hazards and Surface Residual (mg /m^2) on CARC and Aluminum after 20-Min Decontamination with CDS and DF200

Material/Decon	**0-15 min**	**45-60 min**	**Surface Residual**
CARC			
CDS	1.67 ± 0.60	0.22 ± 0.20	29.68 ± 4.48
DF200	**10.65 ± 4.55**	1.48 ± 0.17	28.33 ± 3.14
Aluminum			
CDS	0.00 ± 0.00	0.00 ± 0.00	0.60 ± 0.58
DF200	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00

Average of three replicates ± standard deviation

4.3 VX Contact Hazard Test Results

VX was recovered from the panels during the two 15 min contact periods following decontamination. The agent quantity recovered is expressed as milligrams VX per square meter of surface. The JPID threshold contact exposure level for VX is < 0.04 mg/m^2 (this value does not apply to surface residual measurements). Values above the threshold contact exposure level during the contact periods are shown in bold (Tables 4.5 and 4.6). The VX still associated with the panel following the two contact periods was also quantified and presented as the surface residual.

Table 4.5: VX Contact Hazards and Surface Residual (mg /m^2) on CARC and Aluminum after 10-Min Decontamination with CDS and DF200

Material/Decon	**0-15 min**	**45-60 min**	**Surface Residual**
CARC			
CDS	**1.21 ± 1.04**	**0.14 ± 0.24**	7.87 ± 3.59
DF200	**7.86 ± 1.71**	**1.90 ± 0.17**	20.34 ± 3.42
Aluminum			
CDS	0.00 ± 0.00	**0.12 ± 0.21**	0.00 ± 0.00
DF200	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00

Average of three replicates ± standard deviation

Table 4.6: VX Contact Hazards and Surface Residual (mg /m^2) on CARC and Aluminum after 20-Min Decontamination with CDS and DF200

Material/Decon	**0-15 min**	**45-60 min**	**Surface Residual**
CARC			
CDS	**0.42 ± 0.17**	0.00 ± 0.00	3.33 ± 1.43
DF200	**3.51 ± 1.64**	**0.92 ± 0.48**	12.52 ± 5.83
Aluminum			
CDS	0.00 ± 0.00	**0.18 ± 0.16**	0.00 ± 0.00
DF200	**0.18 ± 0.16**	**0.19 ± 0.33**	0.00 ± 0.00

Average of three replicates ± standard deviation

4.4 Contact Hazard Discussion

The purpose of the study was to evaluate and compare the ability of the decontaminants CDS and DF200 to reduce the contact hazard of surfaces contaminated with GD, HD and VX at 1 g/m^2. The contact hazard was assessed during two 15 min periods, 0-15 min following decontamination and again at 45-60 min following decontamination. Overall, CDS provided superior or comparable decontamination efficacy to DF200 for all agents on CARC and aluminum surfaces. A 10 min CDS treatment provided superior or comparable decontamination efficacy to a 20 min DF200 treatment for all agents on CARC and aluminum surfaces.

For CARC surfaces CDS-decontaminated GD to below JPID threshold levels by the first contact period with a 10 min decontaminant residence time. For HD, the CDS required a 20 min decontaminant residence time to clean the surface to concentrations below the JPID threshold level by the first contact period. On the other hand, DF200-decontaminated CARC surfaces were not cleaned below the JPID threshold level for GD and HD by the first contact period with either 10 or 20 min decontaminant exposure times but were below the threshold level by the second contact period with 10 and 20 min exposure times.

VX was not consistently decontaminated below the threshold level by either decontaminant during the two contact periods on aluminum or CARC. CDS treated CARC had approximately 1/3 the residual VX associated with the surface following the contact tests as DF200-treated CARC surfaces. The surface residual agent shows that a significant quantity of agent remains trapped in the CARC surface one hour following surface decontamination with either CDS or DF200.

Aluminum surfaces contaminated with GD and HD were decontaminated by CDS and DF200 to below threshold levels by the first contact period with only 10 min decontaminant residence time. Very little GD or HD, < 1 mg/m^2, remained on the aluminum surface following the contact tests. VX was not consistently decontaminated below the threshold level by either decontaminant during the two contact periods on aluminum, but no VX was detected on the surface following the contact tests. The decontaminants performed fairly well at decontaminating VX, but since the threshold level of VX is almost two orders of magnitude lower than HD due to its toxicity, the threshold level is more easily exceeded.

Table 4.7 shows a summary of the contact hazard decontamination data for the 0-15 min contact test (note that there are some inconsistencies with the 45-60 min contact test for VX) in the format of JPID threshold ORD Factors and the ratio of the DF200 results to CET. Table 4.8 is a similar presentation of the 45-60 min results. An ORD Factor is the contact hazard result divided by the ORD value (e.g. 1.7 mg/m^2 for GD), values less than one pass the ORD, values greater than 1 indicate how many times greater the result is than the ORD. The ratio of the results for DF200 to CDS illustrates how many times less hazard is presented by CDS than DF200, for example a result of 4.0 indicates that CDS decontaminated coupons present 4 times less hazard as DF200 did for the same residence time. These data show that CDS will reduce the contact hazard to below JPID threshold ORD levels for Aluminum and CARC contaminated with GD, HD, and VX with the exception of VX on CARC which was only decontaminated to 10.5 times the JPID threshold level.

These data show that CDS will reduce the contact hazard for GD, HD, and VX to between 1.5 and 8.4 times less than the equivalent treatment with DF200.

Table 4.7: Summary of Results for the 0-15 Min Contact Hazard Test

Material	Residence Time (min)	Agent	CDS JPID T Factor	DF200 JPID T Factor	Ratio DF200/CDS
Aluminum	10	GD	0.18	0.71	4.0
Aluminum	20	GD	0.17	0.46	2.7
Aluminum	10	HD	0.00	0.00	N/A
Aluminum	20	HD	0.00	0.00	N/A
Aluminum	10	VX	0.00	0.00	N/A
Aluminum	20	VX	0.00	4.50	N/A
CARC	10	GD	0.70	5.22	7.5
CARC	20	GD	0.82	1.21	1.5
CARC	10	HD	1.87	4.22	2.3
CARC	20	HD	0.56	3.55	6.4
CARC	10	VX	30.25	196.50	6.5
CARC	20	VX	10.50	87.75	8.4

Table 4.8: Summary of Results for the 45-60 Min Contact Hazard Test

Material	Residence Time (min)	Agent	CDS JPID T Factor	DF200 JPID T Factor	Ratio DF200/CDS
Aluminum	10	GD	0.68	0.80	1.18
Aluminum	20	GD	0.59	0.75	1.27
Aluminum	10	HD	0.00	0.00	N/A
Aluminum	20	HD	0.00	0.00	N/A
Aluminum	10	VX	3.00	0.00	0.00
Aluminum	20	VX	4.50	4.75	1.06
CARC	10	GD	0.79	0.96	1.21
CARC	20	GD	0.66	0.87	1.31
CARC	10	HD	0.29	0.71	2.44
CARC	20	HD	0.07	0.49	6.73
CARC	10	VX	3.50	47.50	13.57
CARC	20	VX	0.00	23.00	N/A

5. PANEL VAPOR HAZARD TEST RESULTS

5.1 HD Vapor Hazard Test Results

Using the methods described in Section 2.8, six panels were analyzed during a 6 hr period. In some experiments the agent droplets spread on the surface, resulting in significant data skew. For this analysis only sessile drops (droplets that do not spread) are analyzed, drops that spread are rejected. Figure 5.1 depicts the HD vapor concentration for each of the CARC panels treated with CDS for 20 min. Figure 5.2 depicts the HD vapor concentration for each of the CARC panels treated with DF200 for 20 min.

Figure 5.1: Vapor Concentration for HD on CARC after 20 Min Treatment with CDS

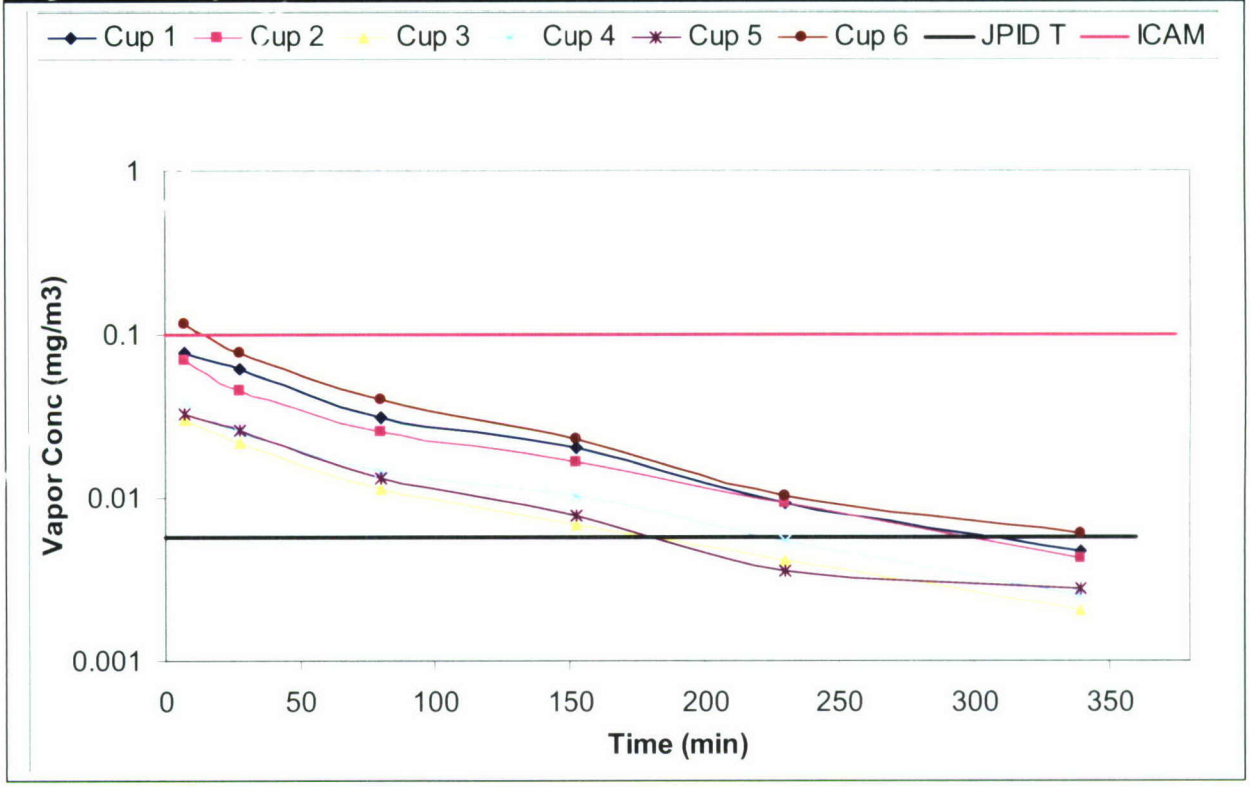
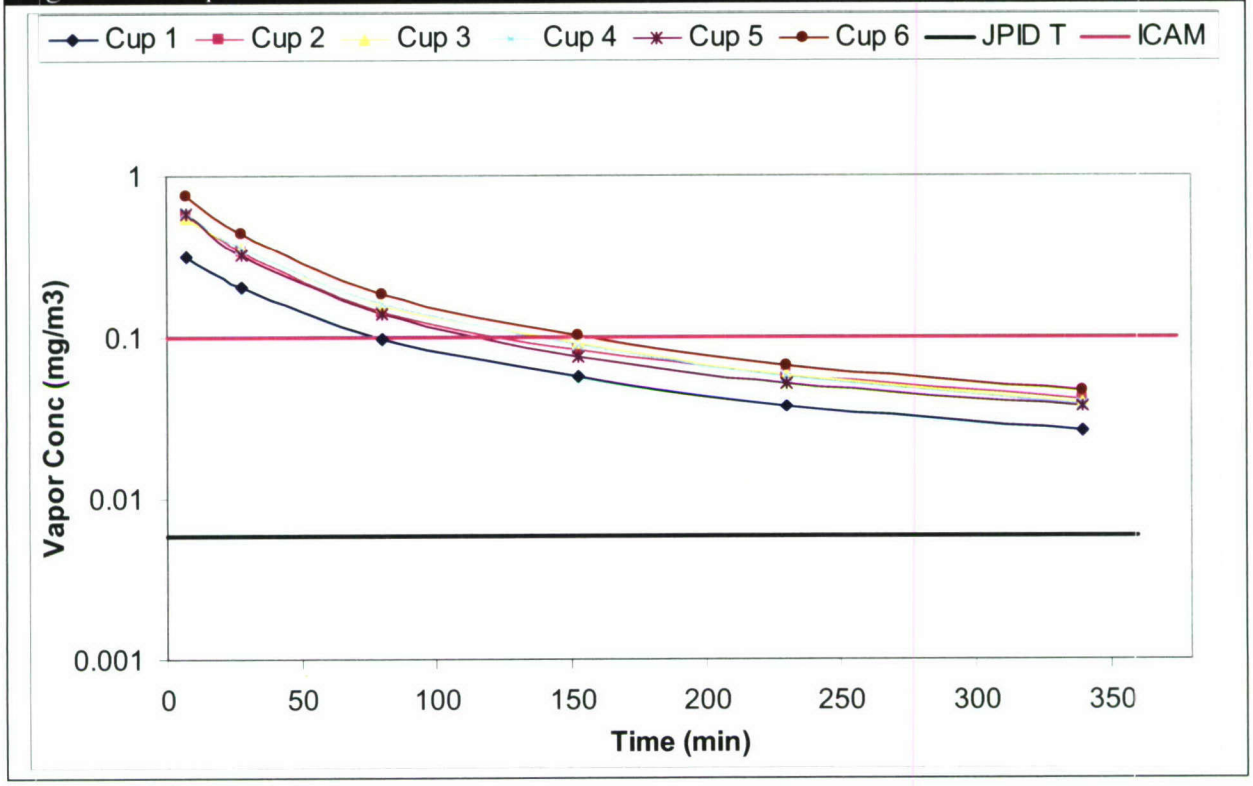
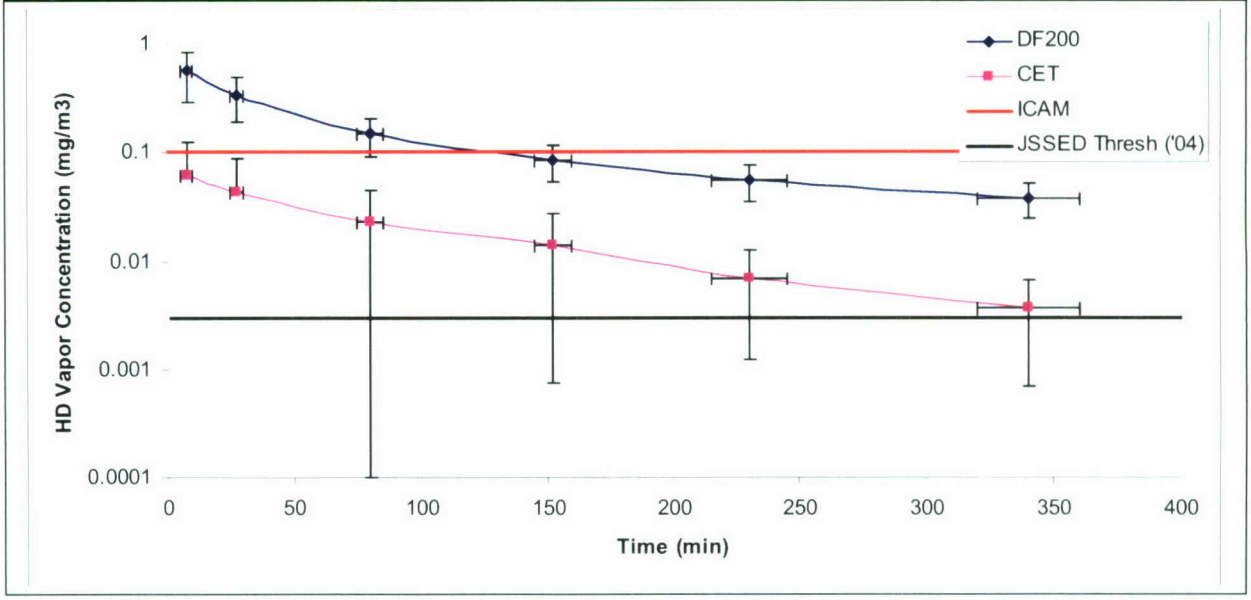


Figure 5.2: Vapor Concentration for HD on CARC after 20 Min-Treatment with DF200



A comparison of the results between CDS and DF200 is presented in Figure 5.3. The x-axis error bars indicate sampling time for the DAAMs tube, y-axis error bars correspond to the 95% confidence interval. These results show that a 20 min treatment with CDS produces a vapor hazard 9.25 times **LESS** than the equivalent 20 min treatment with DF200. Further, treatment of CARC with CDS for 20 min would produce vapor hazards that are below the detection limit of an ICAM; however, these levels are above the JSSED threshold of 0.003 mg/m^3.

Figure 5.3: Average HD Vapor Concentration after 20-Min Treatment.



The equivalent test was executed for the material Aluminum. The treatment with CDS and DF200 sufficiently decontaminated all samples such that all vapor concentrations were below detection limits; the decontaminants show equivalent performance.

5.2 GD & VX Vapor Hazard Test Results

The vapor testing was executed in accordance with TOP 8-2-061. During initial testing the timing used to start and stop vapor collection on the DAAMs tubes resulted in anomalous data for the later time-point vapor samples collected. The timing issue was corrected and the analysis of HD was repeated (data presented in Section 5.1) to confirm the usability of the first tube. To enable comparison between the decontaminants, the results of the initial testing and the repeated HD analysis were analyzed. It was determined that the results for the first vapor time-point sample of the initial tests can be used to provide a comparison between the CDS and DF200 performance.

The following results correspond to the first vapor sample acquired after decontamination. The sample was collected for 30 min at a flow rate of 300 mL/min. The comparison is presented as the ratio of the vapor concentration for the DF200 samples divided by the vapor concentration of the CDS samples. The result indicates how many times better CDS performed compared to DF200 in this test.

The comparison of the decontaminants show:

Overall, the CDS performed equivalent or better than DF200 on aluminum.

- Both CDS and DF200 reduced the HD to below detection limits; the decontaminants show equivalent performance.
- CDS reduced the VX contamination 2.8 times lower than DF200 after a 10 min exposure
- CDS reduced the VX contamination 10.1 times lower than DF200 after a 20 min exposure
- Both CDS and DF200 clean GD to below detection limits; the decontaminants show equivalent performance.

Overall, the CDS performed equivalent or lower than DF200 on CARC painted aluminum

- CDS reduced the HD contamination 7.56 times lower than DF200 after a 10 min exposure.
- CDS reduced the HD contamination 7.42 times lower than DF200 after a 20 min exposure.
- CDS reduced the VX contamination 1.6 times lower than DF200 after a 10 min exposure.
- CDS reduced the VX contamination 4.91 times lower than DF200 after a 20 min exposure.
- CDS reduced the GD contamination 9.52 times lower than DF200 after a 10 min exposure.
- CDS reduced the GD contamination 13.61 times lower than DF200 after a 20 min exposure.

In summary, CDS performed better than DF200 for CARC and aluminum based test.

6. BIOLOGICAL AGENT TEST RESULTS AND DISCUSSION

6.1 Spore Preparation

High-quality spores of plasmid-free avirulent *B. anthracis* were used in this study (see Figure 6.1). The preparation contained only a small fraction of vegetative/proto-spore components. Because the spore preparation was heat and ethanol-treated, no live vegetative cells were present in the sample used in this study. The spore architecture and biochemistry of the avirulent strain are identical to the virulent strain (abs, 2003; def, 2004). Since the spores used in this study are a realistic model for the virulent spores, the results obtained with this strain can be directly extrapolated to the spores from virulent strain of *B. anthracis*.

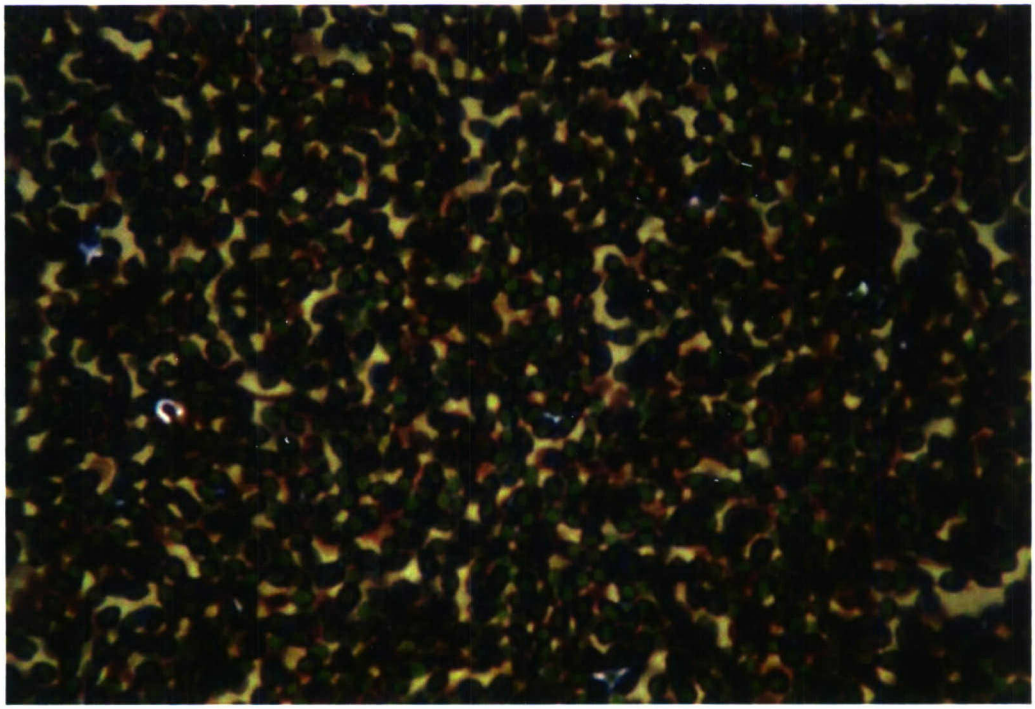


Figure 6.1: Spores of *B. anthracis* (NNR1Δ1) Used in this Study

Spores of *B. anthracis* (NNR1d1) were prepared, heat-treated and ethanol-treated before staining with Malachite Green and viewed under oil immersion lens. The green to dark blue colored oval structures are spores and few long strands of vegetative cells are also visible in the background.

6.2 Baseline Study with Bleach using Glass & Metal Coupons

In the baseline experiments, recovery of spores from three common hard surfaces, glass, unpainted metal and CARC-painted coupons was investigated. Figure 6.2 shows the Log (CFU) recovered using five replicate coupons. On all the coupons, 7-logs spores were loaded in a 50-mL volume. As seen in the figure, comparable recoveries >6.7-logs were recovered from all three test coupons.

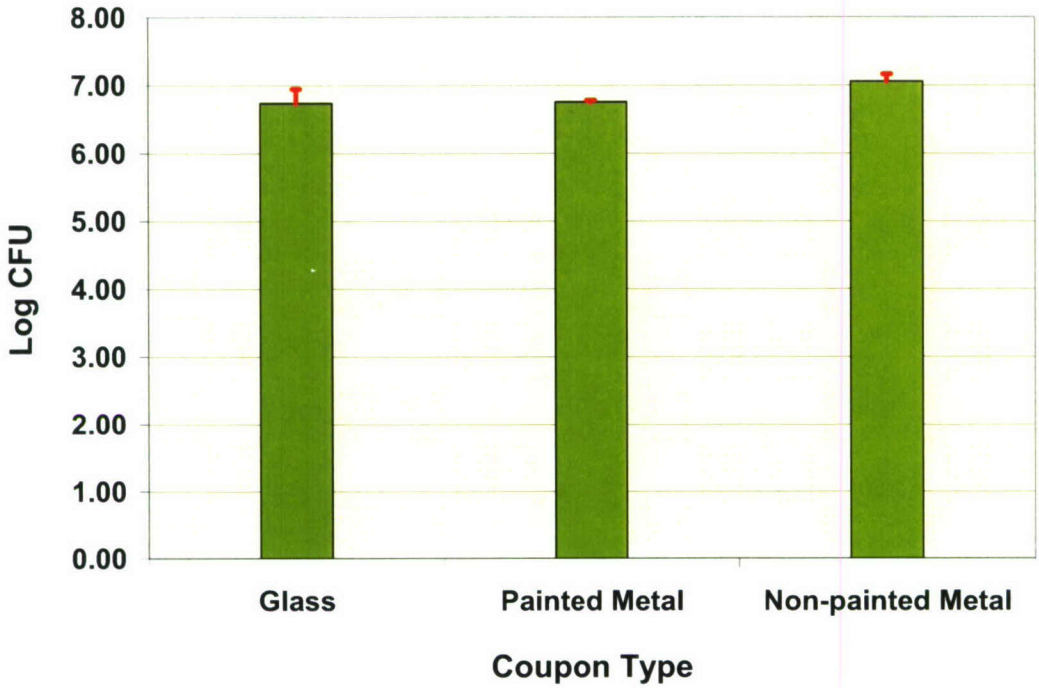


Figure 6.2: Recovery of Spores from Three Coupon Types

Approximately 7-logs spores were dried on the sterile coupons. The spores off the coupons were extracted by 10-min sonication and 2-min vortexing, before dilution plating on TSA plates. The standard deviation from three coupons is shown.

Published data and the previous work in our laboratory (BioDefense Team) have established that while pH unadjusted bleach (<3,000-ppm for 10-min) is a weak sporicidal agent, pH adjusted bleach (3,000-ppm for 15-min and 6,000-ppm for 10-min) is a strong sporicidal agent. The robustness of our STM protocol was tested in these preliminary experiments to see if this protocol will distinguish between different types of treatments, i.e. weak vs. strong sporicidal agent. Figure 6.3 summarizes the results obtained with glass coupons treated with 1,500-ppm pH-unadjusted bleach for 5 and 10-min (a weak treatment) relative to a glass coupon treated with 3,000-ppm or 6,000-ppm pH-adjusted bleach for 30-min (strong treatment). As seen in the Figure 6.3, the STM method can differentiate between a weak and a strong disinfectant treatment.

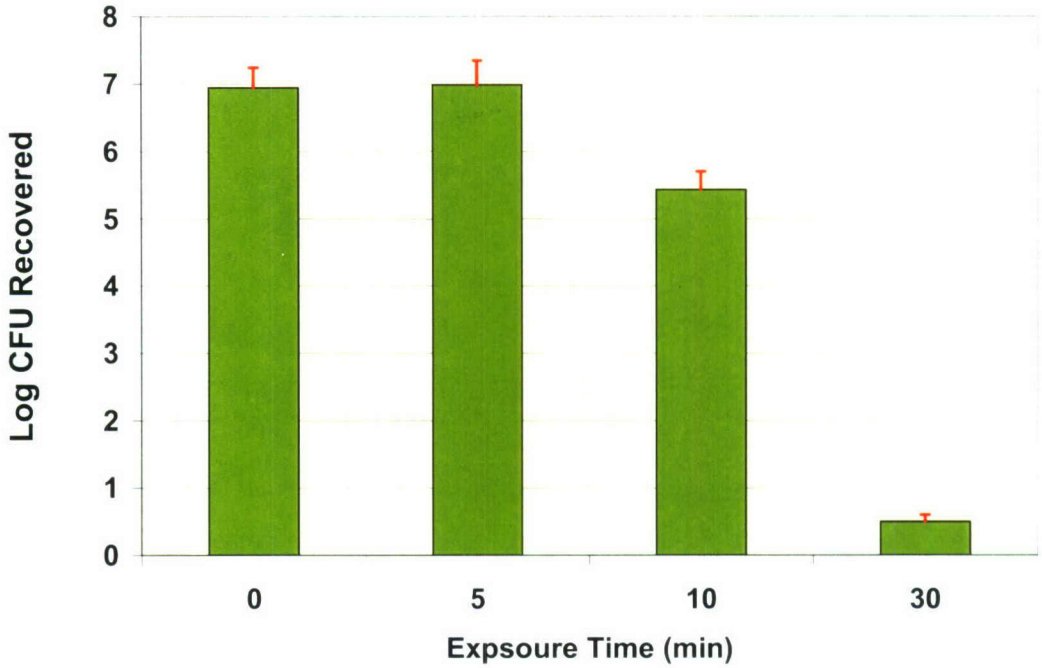


Figure 6.3: Efficacy of Weak Disinfectant (3000-ppm Bleach for 5 or 10-min on Glass Coupons) and Strong Disinfectant (6000-ppm Bleach for 30-min on Metal Coupons)

Approximately 7-logs spores were dried on the sterile coupons. The inoculated coupons were treated with bleach for the times indicated above and then neutralized with 0.5-M sodium thiosulfate (final). The spores were dislodged off the coupons by sonication and vortexing. The viable spores were enumerated after dilution plating. The standard deviation from five coupons is shown.

6.3 Efficacy Study with Bleach and CET BDS

The unpainted and CARC-painted metal coupons were inoculated with ~7-logs spores and dried over-night. The test samples were treated with 15-min exposure to 6000-ppm bleach (pH ~7.0) and 4% CET Peridox™. Control samples were treated with water. As seen in Figure 6.4, >6-log kill was observed for both test chemicals. In bleach-treated sample, only 2 CFU were observed while zero CFU were observed for Peridox™ treatment. Since the limit of detection (LOD) is 2-5, presence of <4 viable spores in test samples cannot be ruled out.

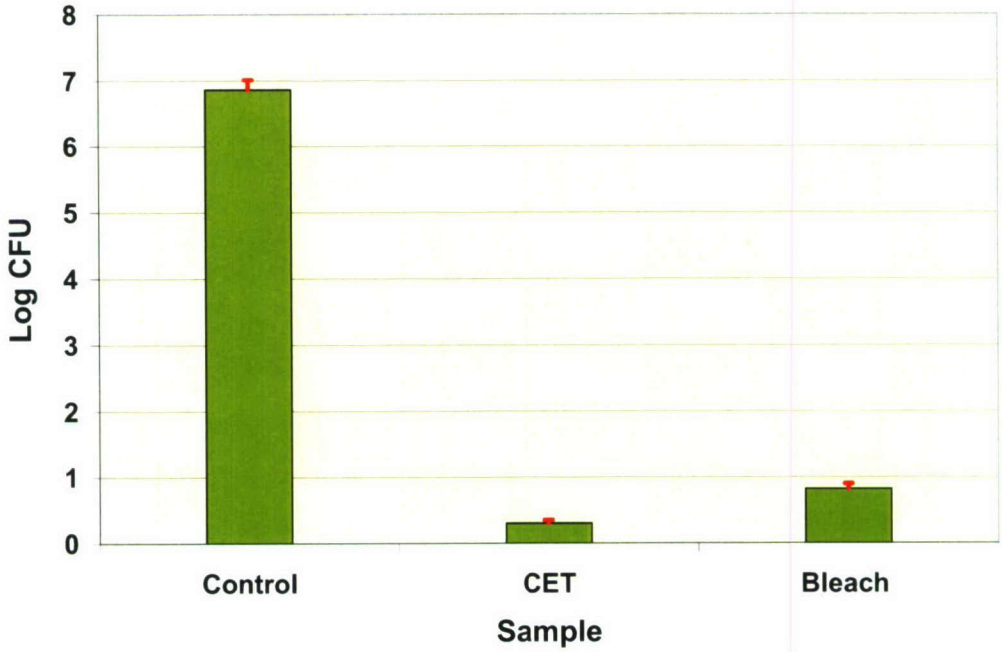


Figure 6.4: Sporicidal Efficacy of Bleach and Peridox™

Approximately 7-logs spores were dried on five sterile replicate coupons. The inoculated coupons were treated with 5 mL of either 6000-ppm bleach (pH ~7.0) or 4% Peridox for 15-min. The active oxidizer component was then immediately neutralized with 5 mL of 1-M sodium thiosulfate. The spores were dislodged off the coupons by sonication and vortexing, and plated after appropriate dilution. The CFU appearing on TSA plates were counted after incubation of plates at 37 °C for over-night. The standard deviation from five replicate coupons is shown.

6.4 Discussion

The purpose of this study was to compare sporicidal efficacy of two oxidizing test chemicals, Clorox bleach (NaOCl) with CET, LLC Peridox™ (containing peroxyacetic acid and H_2O_2). Spores prepared from avirulent strain of *B. anthracis* represent a 'realistic' anthrax surrogate for these results to be valid for the pathogenic strain of anthrax-causing agent. Use of a high-quality spore test material is clear from Figure 6.1, since the preparation contained >90% heat-resistant spores and the dead vegetative cell component was very minor.

In this study, a newly-developed STM protocol was used to show the kill efficacy of the test chemicals. The robustness of this protocol is evident from Figure 6.4, since the method can delineate a weak sporicidal treatment from a strong sporicidal treatment.

Based on the results summarized in this report, the Peridox™ component of the CET is a very effective sporicidal agent.

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LITERATURE CITED

(1) Clean Earth Technologies, L. "Protoype Field Test Report Electrostatic Decontamination for CB Counter-Terrorism," 2003.

(2) Clean Earth Technologies, L. "Test Results for Microbicidal Efficacy, Material Compatibility, and Operability of the Electrostatic Decontamination System Technology and the Biological Decontamination Solution," 2005.

(3) Clean Earth Technologies, L. "Decontamination Effectiveness of Clean Earth Technologies, LLC's Chemical Decontamination Solution, CDS," 2005.

(4) Curry, R.; Golden, J. Clean Earth Technologies, LLC: USA, 1999.

(5) Golden, J.; Stader, J.; Morgan, R. "Electrostatic Decontamination System (EDS): Effective, Fast, Easy Destruction of Chemical and Biological Agents," 2005.

(6) "TOP 8-2-061, Test Operations Procedure for Chemical and Biological Decontaminant Testing," 2002.

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APPENDIX A: STIRRED REACTORS GC-AED DATA

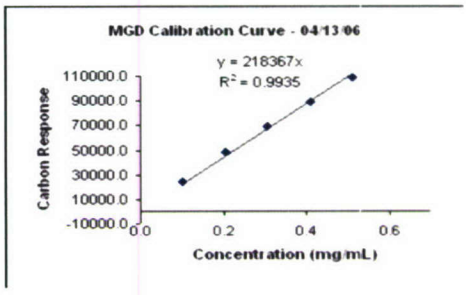
Appendix A1: Carbon Channel GC-AED Data for CDS-GD Samples

Run 041306

GD vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method GD, monitoring Carbon 193
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

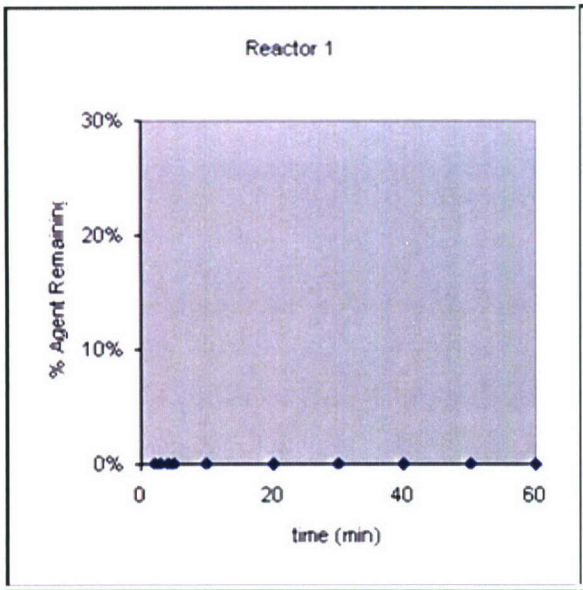
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Carbon)
STD GD-1	1000	0	0.5111	108683
STD GD-2	800	200	0.4089	89250.5
STD GD-3	600	400	0.3067	68882.1
STD GD-4	400	600	0.2044	48126.5
STD GD-5	200	800	0.1022	24362



Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	1	2	2	0	0	0.00%		half life	#DIV/0!	min
	2	3	3	0	0	0.00%				
	3	4	4	0	0	0.00%				
	4	5	5	0	0	0.00%				
	5	10	10	0	0	0.00%				
	6	20	20	0	0	0.00%				
	7	30	30	0	0	0.00%				
	8	40	40	0	0	0.00%				
	9	50	50	0	0	0.00%				
	10	60	60	0	0	0.00%				
	QCC-2 (0.2044mg/mL)			47063.9	0.2145	105.00%				

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	11						half life	#DIV/0!	min
	1	13	2	0	0	0.00%				
	2	14	3	0	0	0.00%				
	3	15	4	0	0	0.00%				
	4	16	5	0	0	0.00%				
	5	21	10	0	0	0.00%				
	6	31	20	0	0	0.00%				
	7	41	30	0	0	0.00%				
	8	51	40	0	0	0.00%				
	9	61	50	0	0	0.00%				
	10	71	60	0	0	0.00%				
	QCC-3 (0.3067mg/mL)			65195.8	0.2972	96.90%				

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	22						half life	#DIV/0!	min
	1	24	2	0	0	0.00%				
	2	25	3	0	0	0.00%				
	3	26	4	0	0	0.00%				
	4	27	5	0	0	0.00%				
	5	32	10	0	0	0.00%				
	6	42	20	0	0	0.00%				
	7	52	30	0	0	0.00%				
	8	62	40	0	0	0.00%				
	9	72	50	0	0	0.00%				
	10	82	60	0	0	0.00%				
	QCC-4 (0.4089mg/mL)			91245.8	0.416	101.70%				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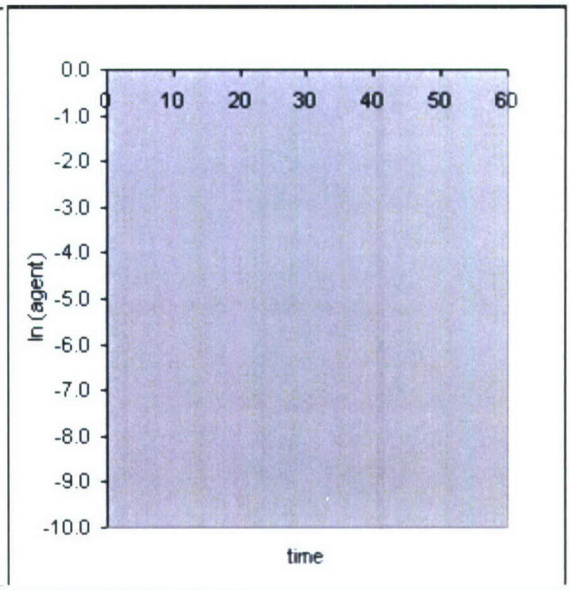
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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-2.0
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-4.0
-5.0
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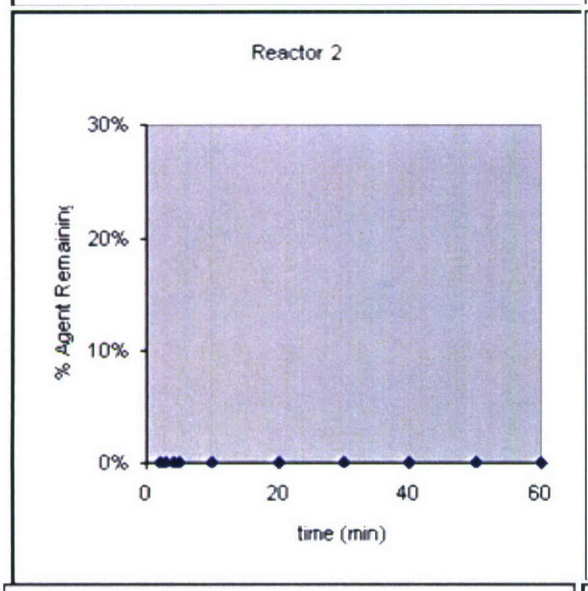
-10.0
0
10
20
30
40
50
60
ln (agent)
time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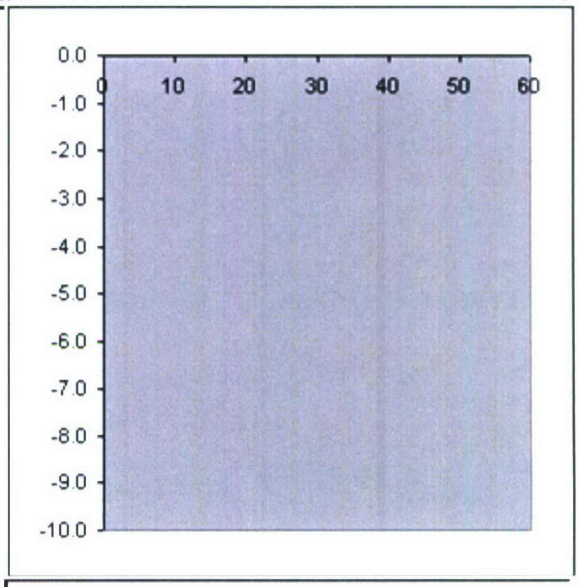
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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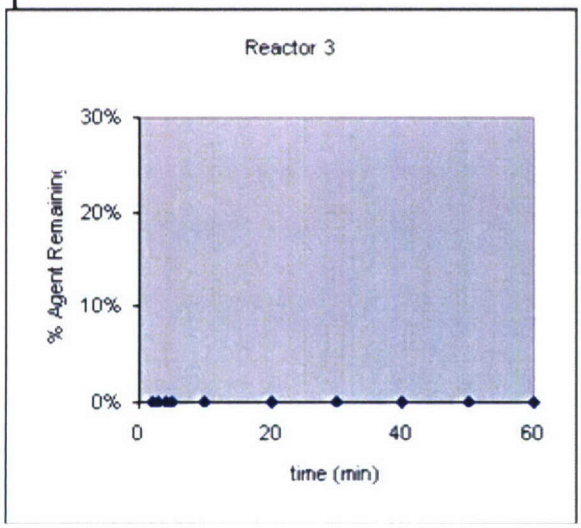
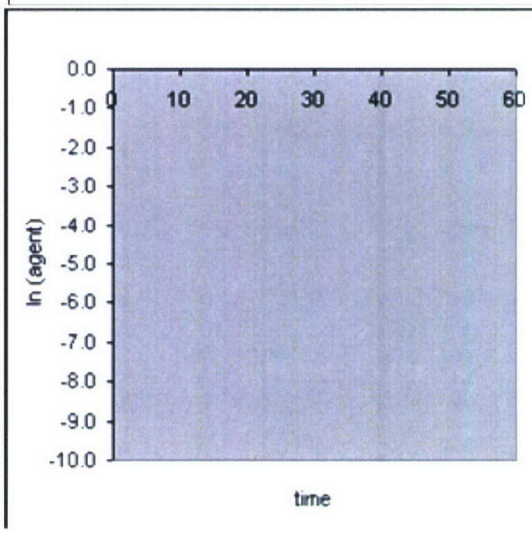
20
30
40
50
60


Reactor 3
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
ln (agent)
time

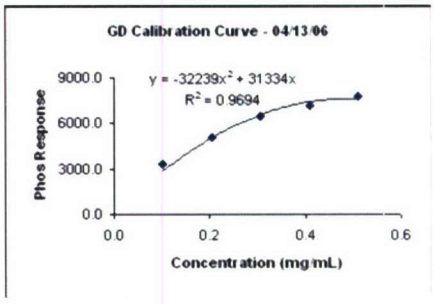
Appendix A2: Phosphorus Channel GC-AED Data for CDS-GD Samples

Run 041306

GD vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method GD, monitoring Phosphorus 178
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

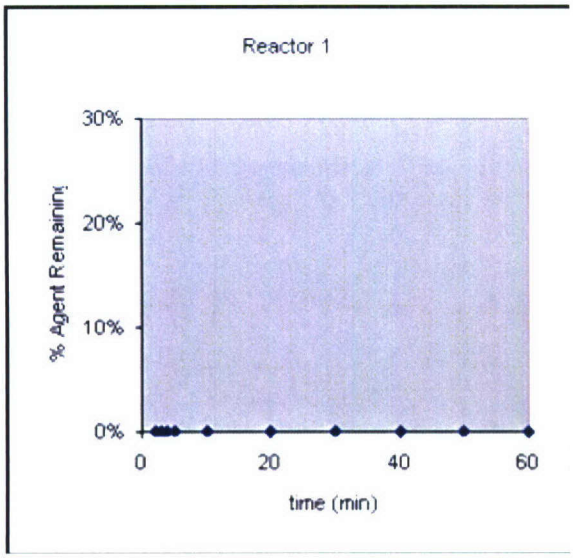
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Phos)
STD GD-1	1000	0	0.5111	7802.2
STD GD-2	800	200	0.4089	7135.4
STD GD-3	600	400	0.3067	6460.7
STD GD-4	400	600	0.2044	5034.8
STD GD-5	200	800	0.1022	3362.4

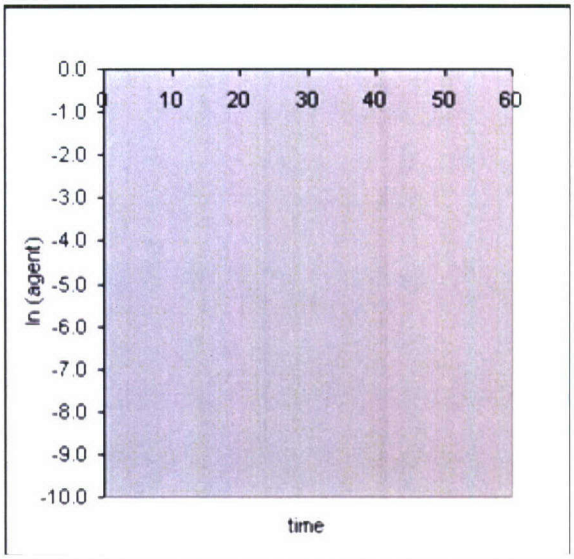


Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	1	2	2	0	0	0.00%		half life	#DIV/0!	min
	2	3	3	0	0	0.00%				
	3	4	4	0	0	0.00%				
	4	5	5	0	0	0.00%				
	5	10	10	0	0	0.00%				
	6	20	20	0	0	0.00%				
	7	30	30	0	0	0.00%				
	8	40	40	0	0	0.00%				
	9	50	50	0	0	0.00%				
	10	60	60	0	0	0.00%				
	QCC-2 (0.2044mg/mL)			5145.4	0.2093	102.40%				

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	11						half life	#DIV/0!	min
	1	13	2	0	0	0.00%				
	2	14	3	0	0	0.00%				
	3	15	4	0	0	0.00%				
	4	16	5	0	0	0.00%				
	5	21	10	0	0	0.00%				
	6	31	20	0	0	0.00%				
	7	41	30	0	0	0.00%				
	8	51	40	0	0	0.00%				
	9	61	50	0	0	0.00%				
	10	71	60	0	0	0.00%				
	QCC-3 (0.3067mg/mL)			6320	0.2857	93.10%				

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	22						half life	#DIV/0!	min
	1	24	2	0	0	0.00%				
	2	25	3	0	0	0.00%				
	3	26	4	0	0	0.00%				
	4	27	5	0	0	0.00%				
	5	32	10	0	0	0.00%				
	6	42	20	0	0	0.00%				
	7	52	30	0	0	0.00%				
	8	62	40	0	0	0.00%				
	9	72	50	0	0	0.00%				
	10	82	60	0	0	0.00%				
	QCC-4 (0.4089mg/mL)			7499.3	0.4264	104.30%				


Reactor 1
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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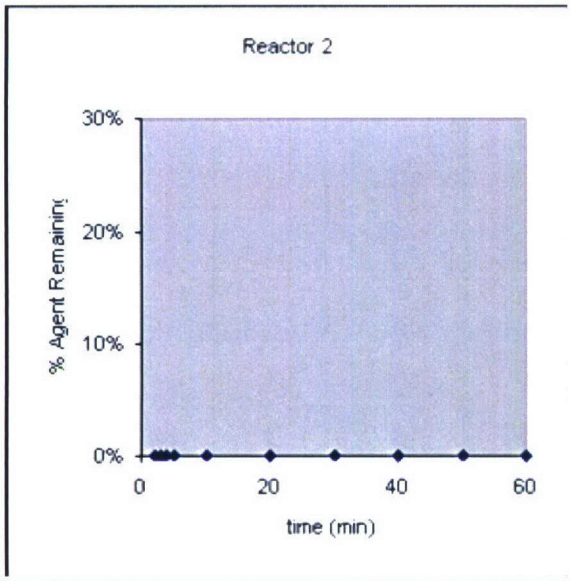
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
0
10
20
30
40
50
60
time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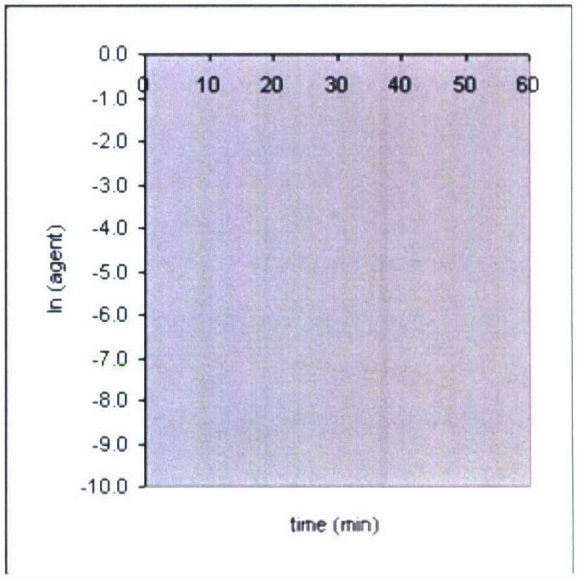
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
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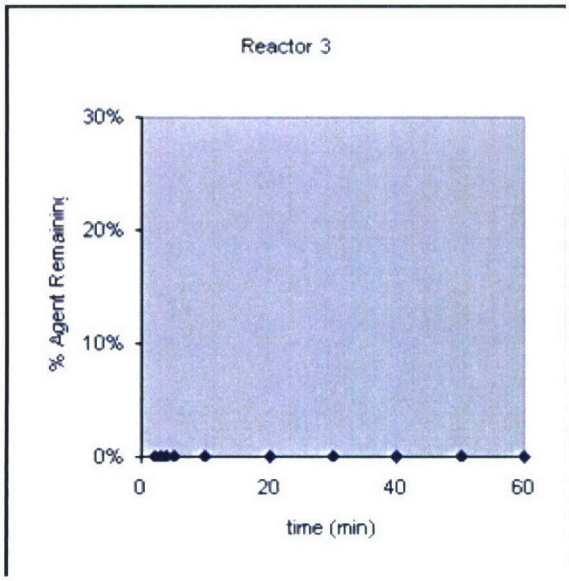
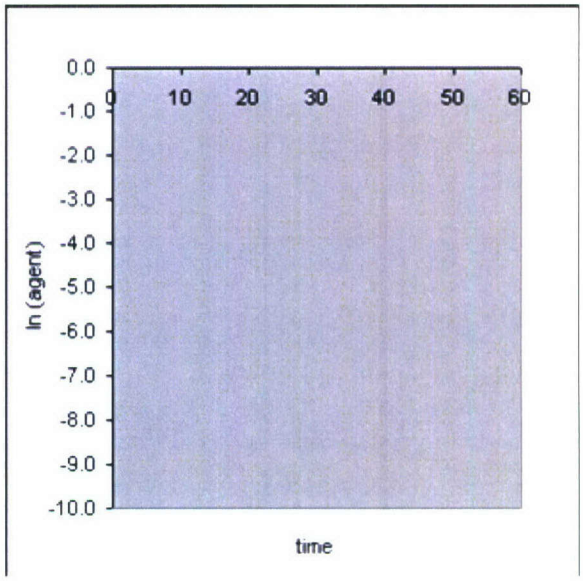
-9.0
-10.0
ln (agent)
0
10
20
30
40
50
60
time (min)


Reactor 3
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
0
10
20
30
40
50
60
time

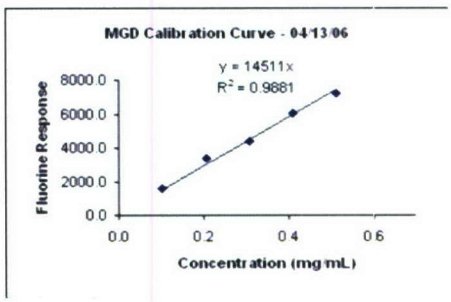
Appendix A3: Fluorine Channel GC-AED Data for CDS-GD Samples

Run 041306

GD vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method GD, monitoring Fluorine 690
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Fluorine)
STD GD-1	1000	0	0.5111	7213.4
STD GD-2	800	200	0.4089	6026.3
STD GD-3	600	400	0.3067	4359.6
STD GD-4	400	600	0.2044	3368.5
STD GD-5	200	800	0.1022	1592.5



Reactor 1

Sample	Time (min)	Corr.Time (min)	Area (Fluorine)	[Agent] µg	% agent remaining	ln (agent)
1	2	2	0	0	0.00%	
2	3	3	0	0	0.00%	
3	4	4	0	0	0.00%	
4	5	5	0	0	0.00%	
5	10	10	0	0	0.00%	
6	20	20	0	0	0.00%	
7	30	30	0	0	0.00%	
8	40	40	0	0	0.00%	
9	50	50	0	0	0.00%	
10	60	60	0	0	0.00%	
QCC-2 (0.2044mg/mL)			3075.5	0.2119	103.70%	

rate -0.2025 min-1
half life 3.42 min

Reactor 2

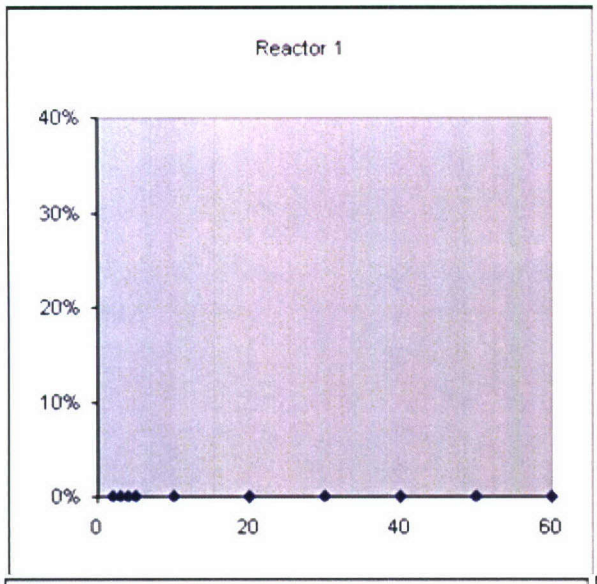
Sample	Time (min)	Corr.Time (min)	Area (Fluorine)	[Agent] µg	% agent remaining	ln (agent)
Start	11					
1	13	2	0	0	0.00%	
2	14	3	0	0	0.00%	
3	15	4	0	0	0.00%	
4	16	5	0	0	0.00%	
5	21	10	0	0	0.00%	
6	31	20	0	0	0.00%	
7	41	30	0	0	0.00%	
8	51	40	0	0	0.00%	
9	61	50	0	0	0.00%	
10	71	60	0	0	0.00%	
QCC-3 (0.3067mg/mL)			4036.2	0.2781	90.70%	

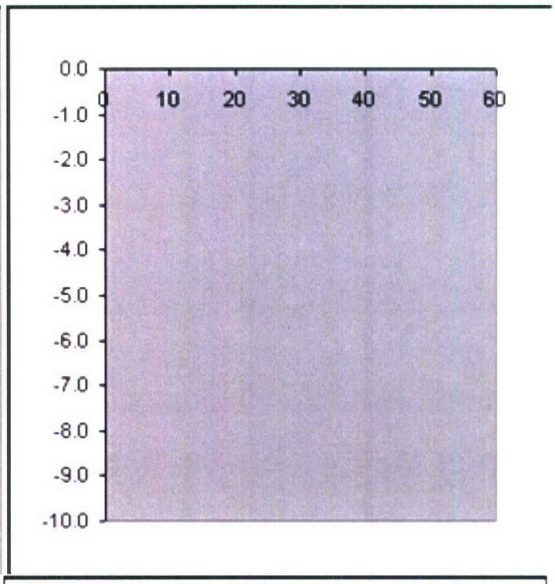
rate -0.3543 min-1
half life 1.96 min

Reactor 3

Sample	Time (min)	Corr.Time (min)	Area (Fluorine)	[Agent] µg	% agent remaining	ln (agent)
Start	22					
1	24	2	0	0	0.00%	
2	25	3	0	0	0.00%	
3	26	4	0	0	0.00%	
4	27	5	0	0	0.00%	
5	32	10	0	0	0.00%	
6	42	20	0	0	0.00%	
7	52	30	0	0	0.00%	
8	62	40	0	0	0.00%	
9	72	50	0	0	0.00%	
10	82	60	0	0	0.00%	
QCC-4 (0.4089mg/mL)			5954.3	0.4103	100.30%	

rate -0.1696 min-1
half life 4.09 min


Reactor 1
40%
30%
20%
10%
0%
0
20
40
60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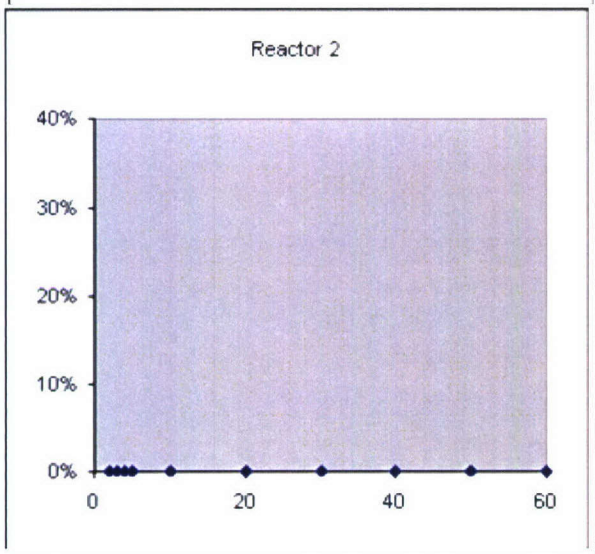
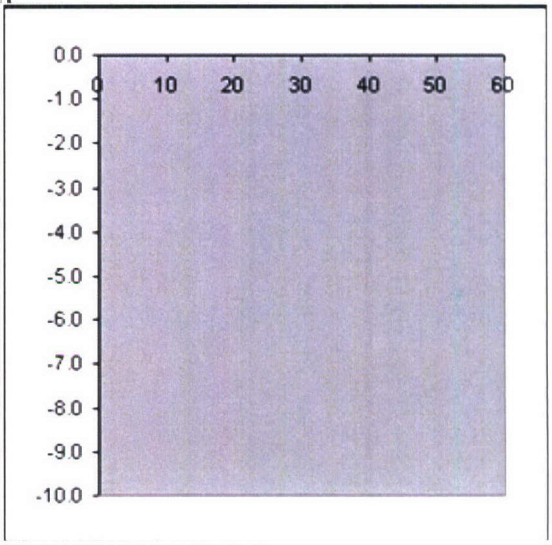
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60


Reactor 2
40%
30%
20%
10%
0%
0
20
40
60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-2.0
-3.0
-4.0
-5.0
-6.0
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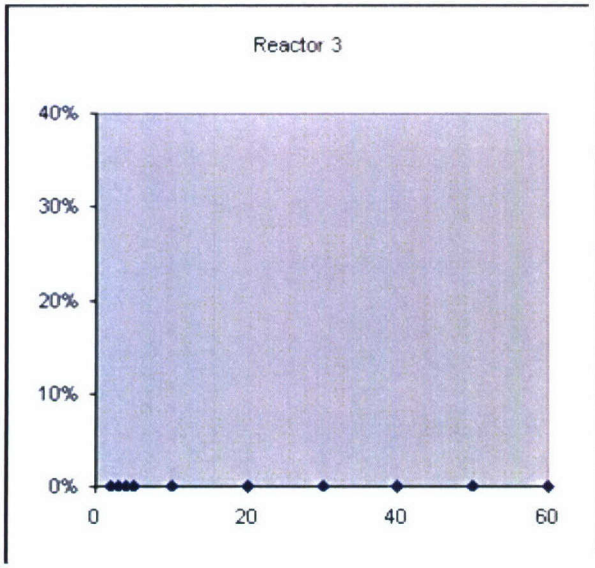
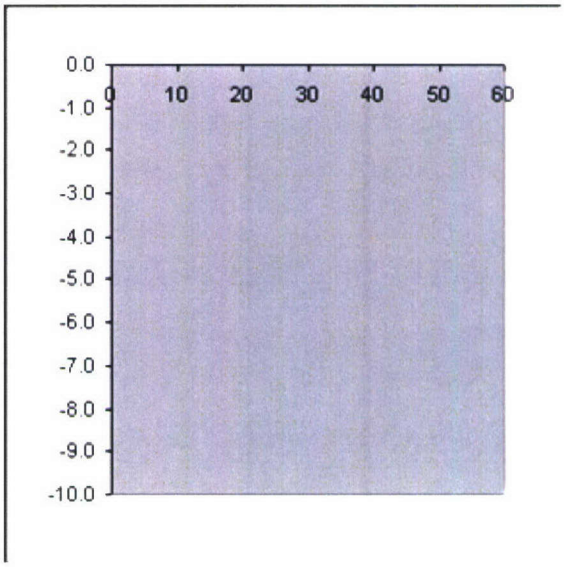
-8.0
-9.0
-10.0
0
10
20
30
40
50
60


Reactor 3
40%
30%
20%
10%
0%
0
20
40
60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-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60

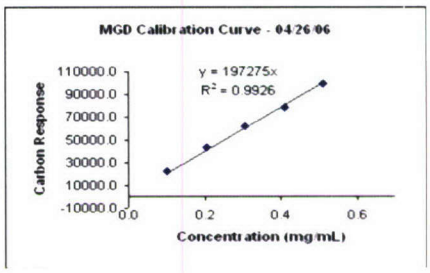
Appendix A4: Carbon Channel GC-AED Data for DF200-GD Samples

Run 042606

GD vs. DF200 @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method GD, monitoring Carbon 193
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Carbon)
STD GD-1	1000	0	0.5111	99860.3
STD GD-2	800	200	0.4089	78310.3
STD GD-3	600	400	0.3067	62232.4
STD GD-4	400	600	0.2044	43541.9
STD GD-5	200	800	0.1022	22775.4



Reactor 1

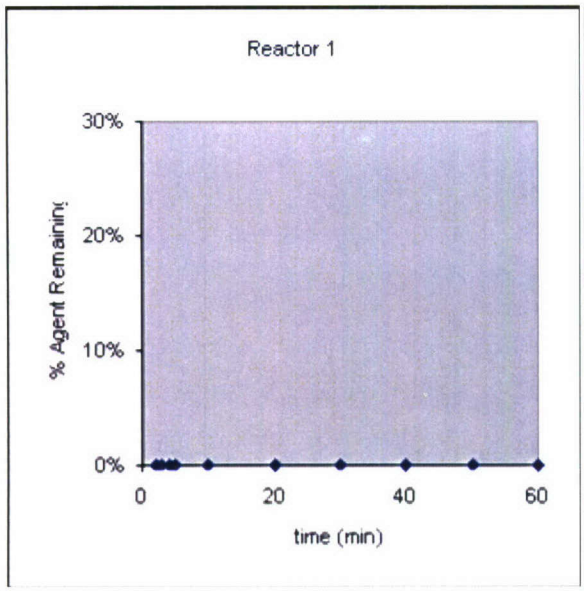
Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
							rate		min-1
1	2	2	0	0	0.00%		half life	#DIV/0!	min
2	3	3	0	0	0.00%				
3	4	4	0	0	0.00%				
4	5	5	0	0	0.00%				
5	10	10	0	0	0.00%				
6	20	20	0	0	0.00%				
7	30	30	0	0	0.00%				
8	40	40	0	0	0.00%				
9	50	50	0	0	0.00%				
10	60	60	0	0	0.00%				
QCC-2 (0.2044mg/mL)			42591.2	0.2159	105.60%				

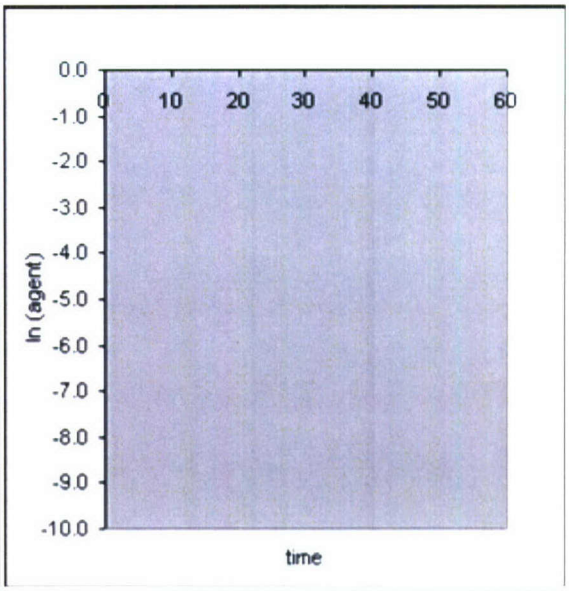
Reactor 2

Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
							rate		min-1
Start	11						half life	#DIV/0!	min
1	13	2	0	0	0.00%				
2	14	3	0	0	0.00%				
3	15	4	0	0	0.00%				
4	16	5	0	0	0.00%				
5	21	10	0	0	0.00%				
6	31	20	0	0	0.00%				
7	41	30	0	0	0.00%				
8	51	40	0	0	0.00%				
9	61	50	0	0	0.00%				
10	71	60	0	0	0.00%				
QCC-3 (0.3067mg/mL)			58451.7	0.2963	96.60%				

Reactor 3

Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
							rate		min-1
Start	22						half life	#DIV/0!	min
1	24	2	0	0	0.00%				
2	25	3	0	0	0.00%				
3	26	4	0	0	0.00%				
4	27	5	0	0	0.00%				
5	32	10	0	0	0.00%				
6	42	20	0	0	0.00%				
7	52	30	0	0	0.00%				
8	62	40	0	0	0.00%				
9	72	50	0	0	0.00%				
10	82	60	0	0	0.00%				
QCC-4 (0.4089mg/mL)			82489.1	0.4181	102.30%				


Reactor 1
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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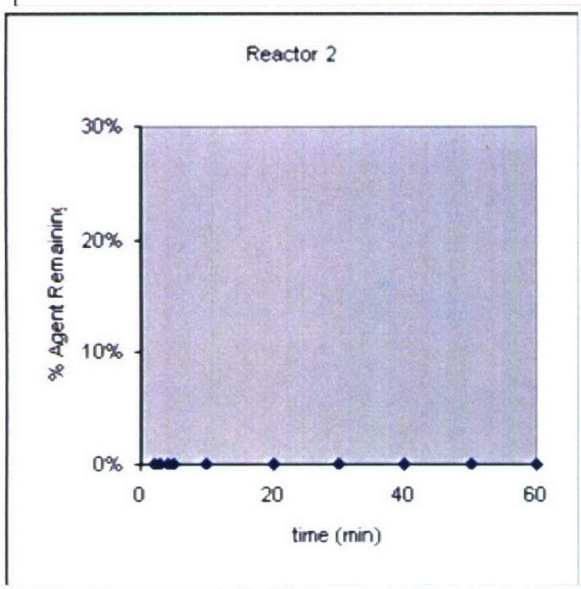
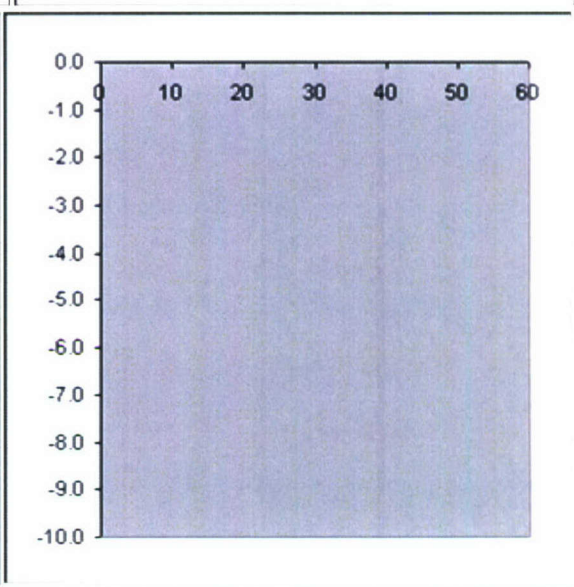
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
ln (agent)
time


Reactor 2
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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-2.0
-3.0
-4.0
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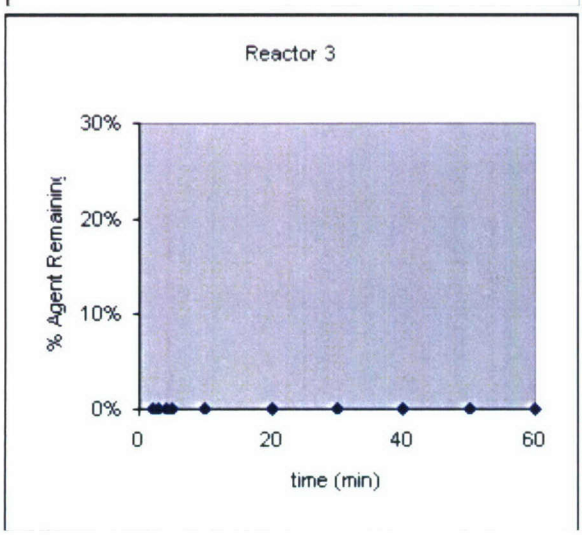
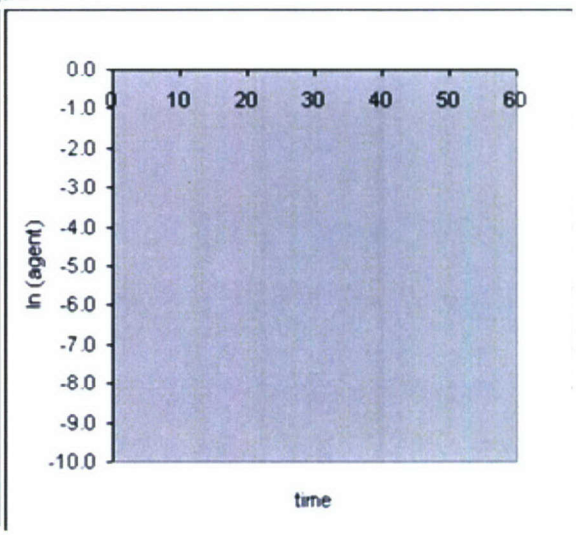
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60


Reactor 3
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
ln (agent)
time

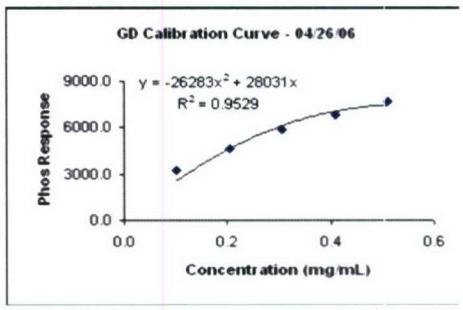
Appendix A5: Phosphorus Channel GC-AED Data for DF200-GD Samples

Run 042606

GD vs. DF200 @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method GD, monitoring Phosphorus 178
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

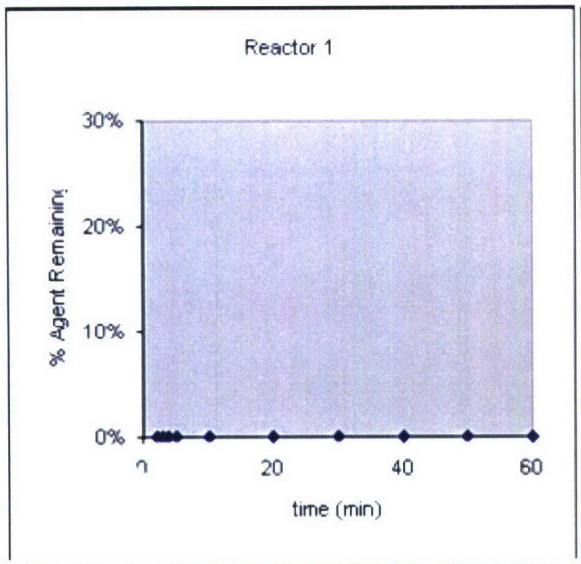
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Phos)
STD GD-1	1000	0	0.5111	7695.2
STD GD-2	800	200	0.4089	6809.1
STD GD-3	600	400	0.3067	5848.4
STD GD-4	400	600	0.2044	4664.2
STD GD-5	200	800	0.1022	3214.3

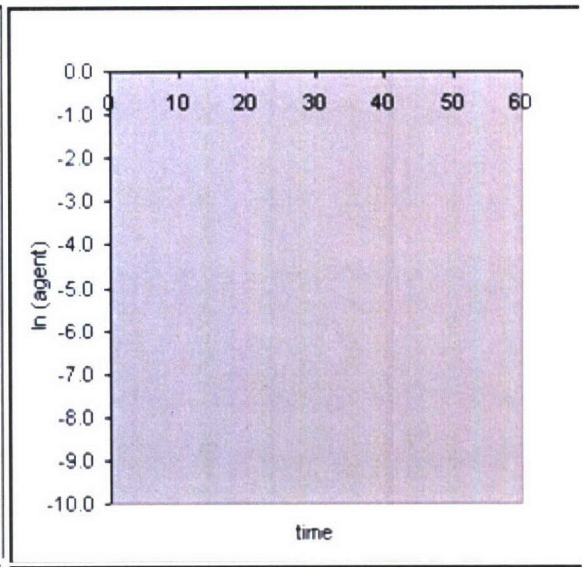


Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	1	2	2	0	0	0.00%		half life	#DIV/0!	min
	2	3	3	0	0	0.00%				
	3	4	4	0	0	0.00%				
	4	5	5	0	0	0.00%				
	5	10	10	0	0	0.00%				
	6	20	20	0	0	0.00%				
	7	30	30	0	0	0.00%				
	8	40	40	0	0	0.00%				
	9	50	50	0	0	0.00%				
	10	60	60	0	0	0.00%				
	QCC-2 (0.2044mg/mL)			4785.9	0.2135	104.40%				

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	11						half life	#DIV/0!	min
	1	13	2	0	0	0.00%				
	2	14	3	0	0	0.00%				
	3	15	4	0	0	0.00%				
	4	16	5	0	0	0.00%				
	5	21	10	0	0	0.00%				
	6	31	20	0	0	0.00%				
	7	41	30	0	0	0.00%				
	8	51	40	0	0	0.00%				
	9	61	50	0	0	0.00%				
	10	71	60	0	0	0.00%				
	QCC-3 (0.3067mg/mL)			6034.3	0.2992	97.60%				

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	22						half life	#DIV/0!	min
	1	24	2	0	0	0.00%				
	2	25	3	0	0	0.00%				
	3	26	4	0	0	0.00%				
	4	27	5	0	0	0.00%				
	5	32	10	0	0	0.00%				
	6	42	20	0	0	0.00%				
	7	52	30	0	0	0.00%				
	8	62	40	0	0	0.00%				
	9	72	50	0	0	0.00%				
	10	82	60	0	0	0.00%				
	QCC-4 (0.4089mg/mL)			6979.2	0.3961	96.90%				


Reactor 1
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)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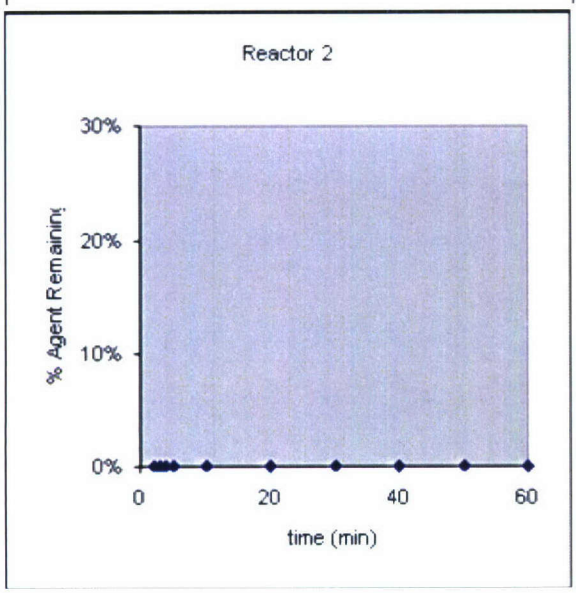
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
0
10
20
30
40
50
60
time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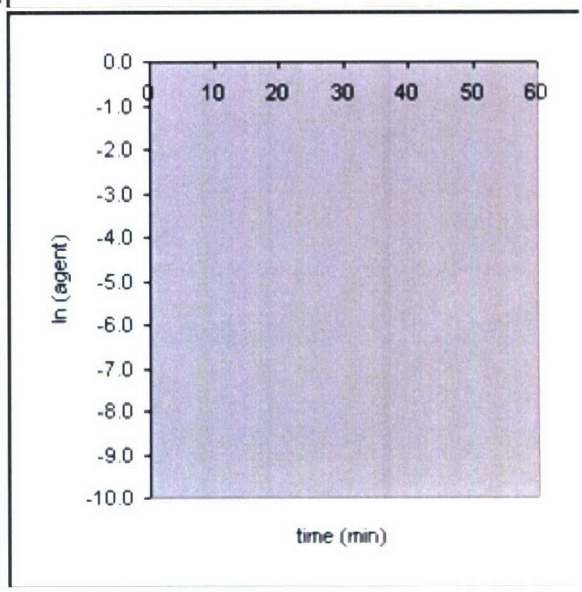
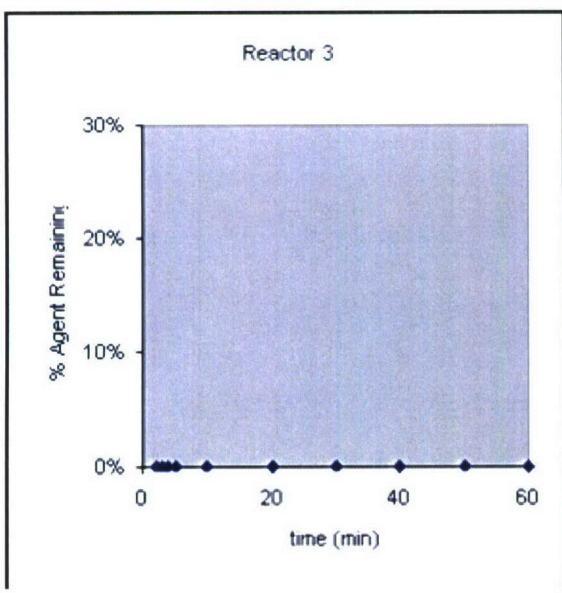
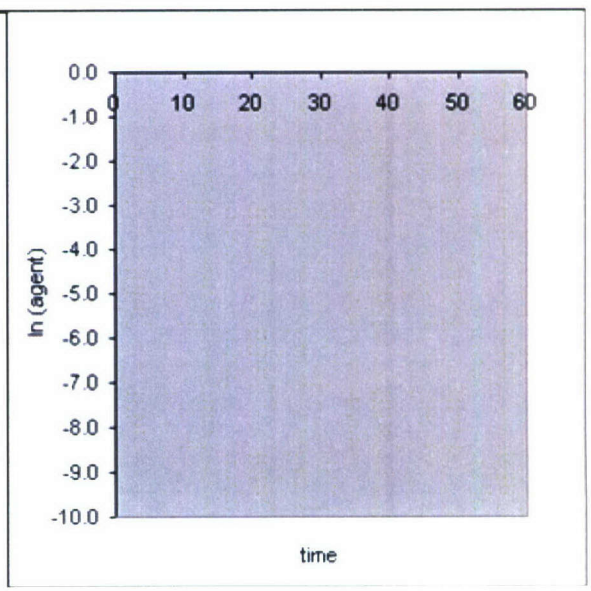
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
0
10
20
30
40
50
60
time (min)


Reactor 3
30%
20%
10%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
0
10
20
30
40
50
60
time

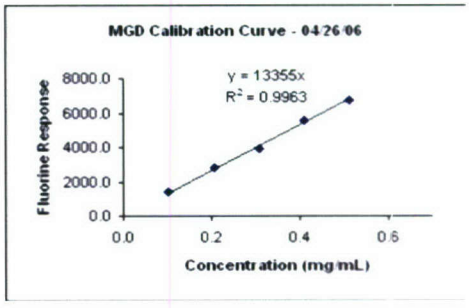
Appendix A6: Fluorine Channel GC-AED Data for DF200-GD Samples

Run 042606

GD vs. DF200 @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method GD, monitoring Fluorine 690
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

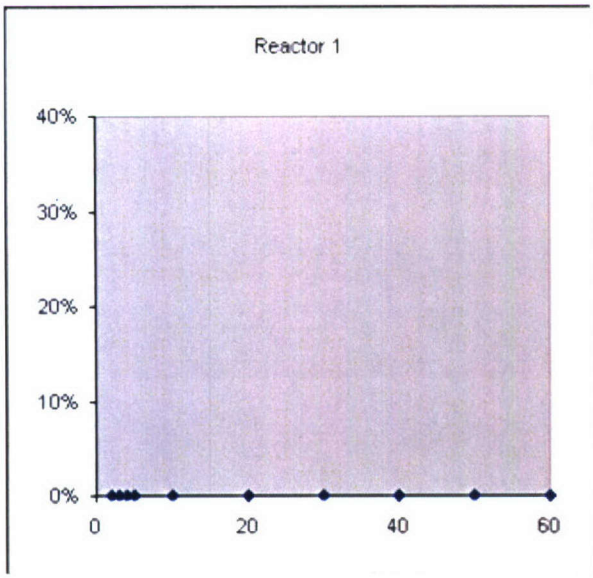
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Fluorine)
STD GD-1	1000	0	0.5111	6774.8
STD GD-2	800	200	0.4089	5583.5
STD GD-3	600	400	0.3067	3918.9
STD GD-4	400	600	0.2044	2851.4
STD GD-5	200	800	0.1022	1417.9

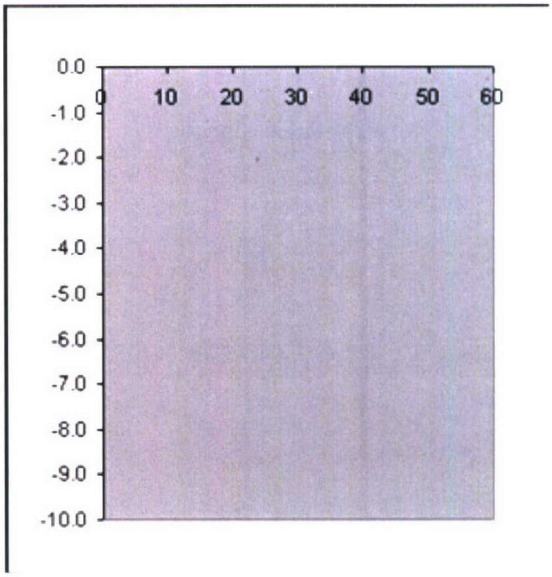


Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Fluorine)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
								half life	#DIV/0!	min
	1	2	2	0	0	0.00%				
	2	3	3	0	0	0.00%				
	3	4	4	0	0	0.00%				
	4	5	5	0	0	0.00%				
	5	10	10	0	0	0.00%				
	6	20	20	0	0	0.00%				
	7	30	30	0	0	0.00%				
	8	40	40	0	0	0.00%				
	9	50	50	0	0	0.00%				
	10	60	60	0	0	0.00%				
	QCC-2 (0.2044mg/mL)			3059.9	0.2291	112.10%				

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Fluorine)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	11						half life	#DIV/0!	min
	1	13	2	0	0	0.00%				
	2	14	3	0	0	0.00%				
	3	15	4	0	0	0.00%				
	4	16	5	0	0	0.00%				
	5	21	10	0	0	0.00%				
	6	31	20	0	0	0.00%				
	7	41	30	0	0	0.00%				
	8	51	40	0	0	0.00%				
	9	61	50	0	0	0.00%				
	10	71	60	0	0	0.00%				
	QCC-3 (0.3067mg/mL)			4552.1	0.3409	111.10%				

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Fluorine)	[Agent] µg	% agent remaining	ln (agent)			
								rate		min-1
	Start	22						half life	#DIV/0!	min
	1	24	2	0	0	0.00%				
	2	25	3	0	0	0.00%				
	3	26	4	0	0	0.00%				
	4	27	5	0	0	0.00%				
	5	32	10	0	0	0.00%				
	6	42	20	0	0	0.00%				
	7	52	30	0	0	0.00%				
	8	62	40	0	0	0.00%				
	9	72	50	0	0	0.00%				
	10	82	60	0	0	0.00%				
	QCC-4 (0.4089mg/mL)			5437	0.4071	99.60%				


Reactor 1
40%
30%
20%
10%
0%
0
20
40
60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-1.0
-2.0
-3.0
-4.0
-5.0
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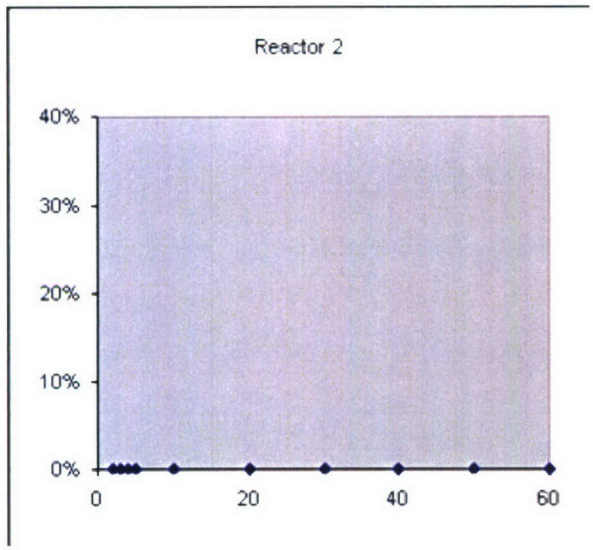
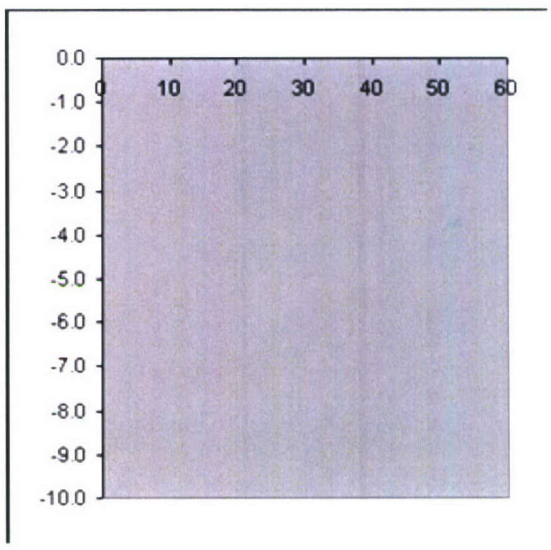
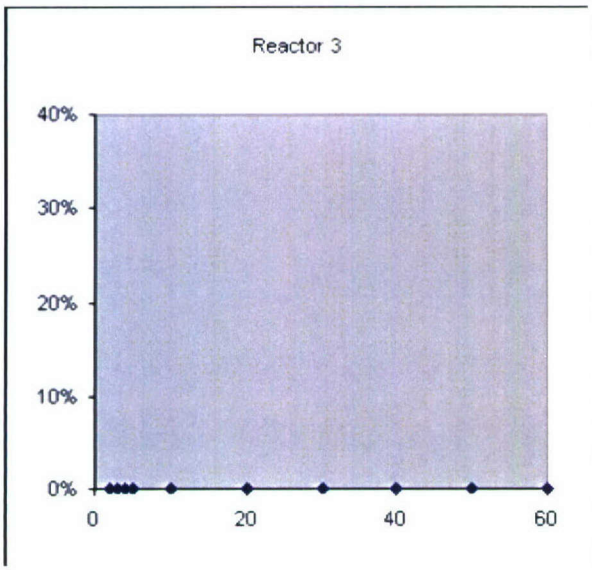
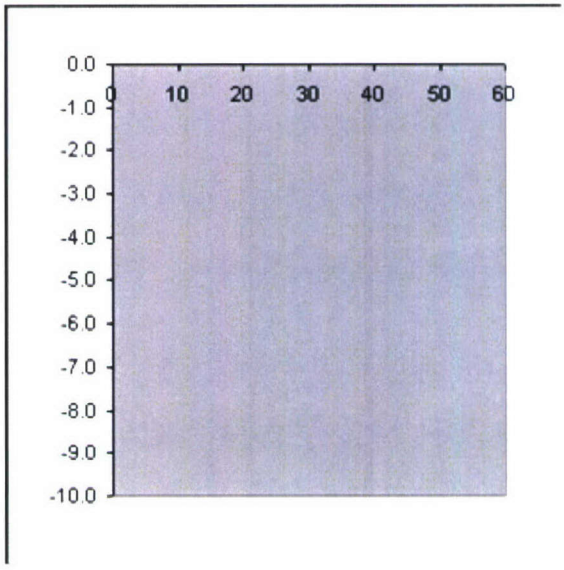
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60


Reactor 2
40%
30%
20%
10%
0%
0
20
40
60


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60


Reactor 3
40%
30%
20%
10%
0%
0
20
40
60


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60

Appendix A7: Carbon Channel GC-AED Data for CDS-HD Samples

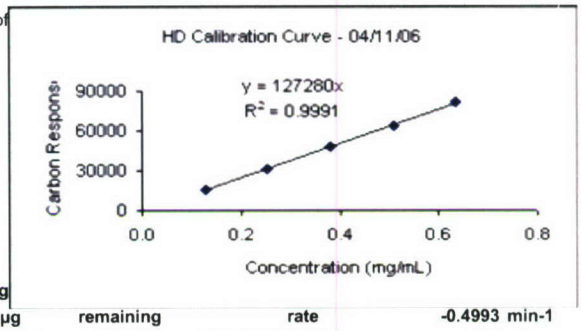
Run: 041106

HD vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.27g/mL
Initial agent concentration: 2% v/v
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite/0.2 M Sodium Carbonate and 2.0 mL chloroform
GC: GC-AED, method HD, monitoring Carbon 193
Standards: Stock solution: 5.0 µL agent into 10.0 mL chlorof

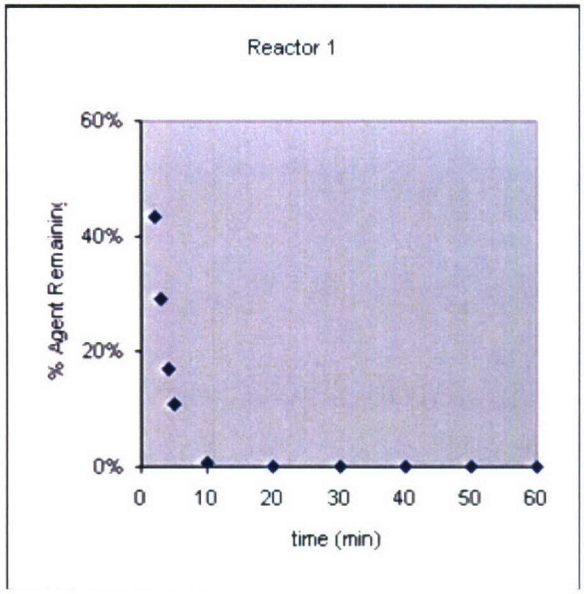
Solution	µL stock	µL CHCl3
STD HD-1	1000	0
STD HD-2	800	200
STD HD-3	600	400
STD HD-4	400	600
STD HD-5	200	800

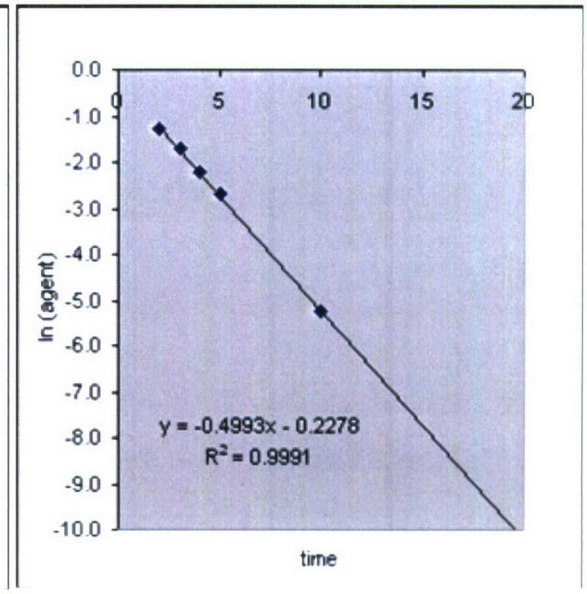


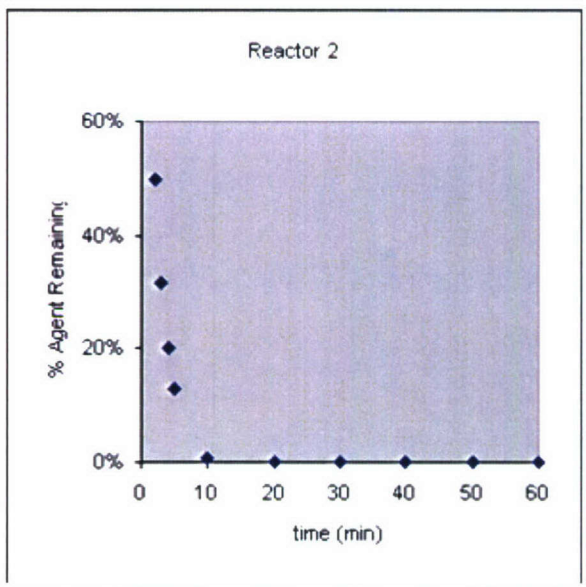
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Ag µg	remaining		rate	-0.4993 min-1
	1	2	2	35032.7	0.2752	43.30%	-1.2901075	half life	1.39 min
	2	3	3	23436.5	0.1841	29.00%	-1.6920947		
	3	4	4	13756	0.1081	17.00%	-2.2249143		
	4	5	5	8822.8	0.0693	10.90%	-2.6690501		
	5	10	10	671	0.0053	0.80%	-5.2453755		
	6	20	20	0	0	0.00%			
	7	30	30	0	0	0.00%			
	8	40	40	0	0	0.00%			
	9	50	50	0	0	0.00%			
	10	60	60	0	0	0.00%			
	QCC-2 (0.254mg/mL)			33537	0.2635	103.70%			

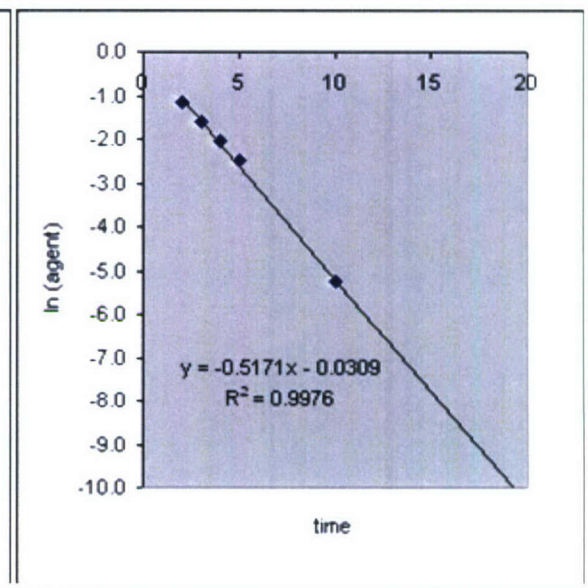
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.5171 min-1
	Start	11						half life	1.34 min
	1	13	2	40220.9	0.316	49.80%	-1.1520026		
	2	14	3	25760.6	0.2024	31.90%	-1.5975432		
	3	15	4	16191	0.1272	20.00%	-2.0619339		
	4	16	5	10432.7	0.082	12.90%	-2.5014443		
	5	21	10	665.9	0.0052	0.80%	-5.2530052		
	6	31	20	0	0	0.00%			
	7	41	30	0	0	0.00%			
	8	51	40	0	0	0.00%			
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
	QCC-3 (0.381mg/mL)			53628.8	0.4213	110.60%			

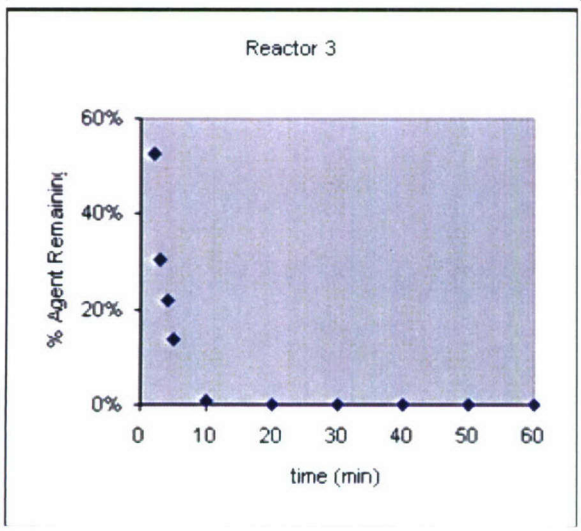
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.496 min-1
	Start	22						half life	1.4 min
	1	24	2	42681.1	0.3353	52.80%	-1.0926332		
	2	25	3	24575.3	0.1931	30.40%	-1.6446475		
	3	26	4	17601	0.1383	21.80%	-1.9784337		
	4	27	5	11057.7	0.0869	13.70%	-2.4432624		
	5	32	10	800.3	0.0063	1.00%	-5.069158		
	6	42	20	0	0	0.00%			
	7	52	30	0	0	0.00%			
	8	62	40	0	0	0.00%			
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
	QCC-4 (0.508mg/mL)			63907.9	0.5021	98.80%			

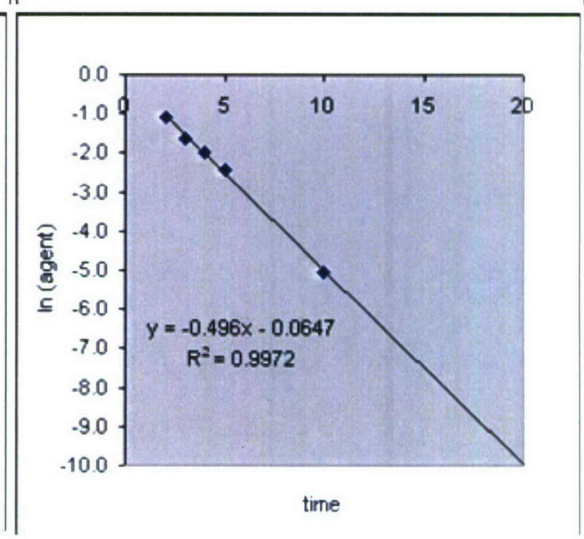

Reactor 1
% Agent Remaining
60%
40%
20%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.4993x - 0.2278
R² = 0.9991
time


Reactor 2
% Agent Remaining
60%
40%
20%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.5171x - 0.0309
R² = 0.9976
time


Reactor 3
% Agent Remaining
60%
40%
20%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.496x - 0.0647
R² = 0.9972
time

Appendix A8: Sulfur Channel GC-AED Data for CDS-HD Samples

Run: 041106

HD vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.27g/mL

Initial agent concentration: 2% v/v

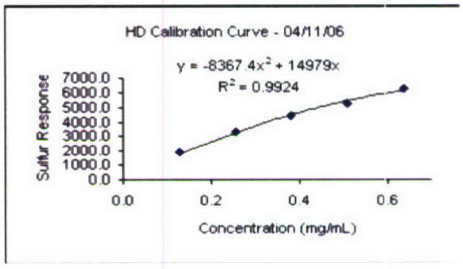
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite/0.2 M Sodium Carbonate and 2.0 mL chloroform

GC: GC-AED, method HD, monitoring Sulfur 181

Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Sulf)
STD HD-1	1000	0	0.635	6254.4
STD HD-2	800	200	0.508	5277.1
STD HD-3	600	400	0.381	4434.7
STD HD-4	400	600	0.254	3310.5
STD HD-5	200	800	0.127	1958.6



Reactor 1

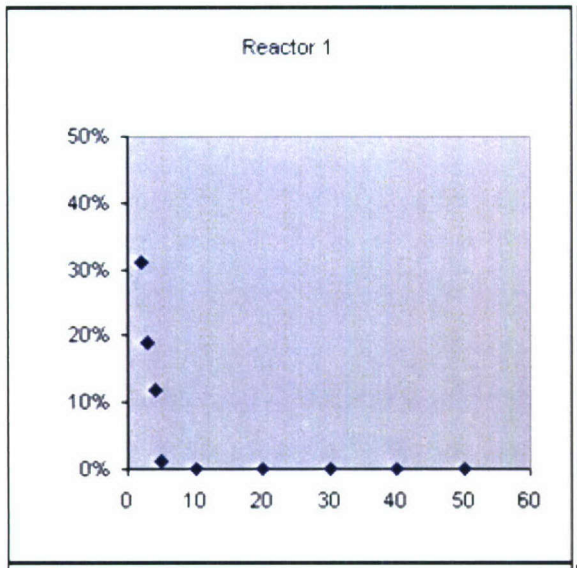
Sample	Time (min)	Corr.Time (min)	Area (Sulf)	[Agent] µg	% agent remaining	ln (agent)		
							rate	-0.4694 min-1
1	2	2	3399.6	0.2667	42.00%	-1.32167914	half life	1.48 min
2	3	3	2634.7	0.1977	31.10%	-1.62083381		
3	4	4	1679.4	0.1202	18.90%	-2.11871584		
4	5	5	1077.4	0.0751	11.80%	-2.58925565		
5	10	10	100.4	0.0067	1.10%	-5.00147689		
6	20	20	0	0	0.00%			
7	30	30	0	0	0.00%			
8	40	40	0	0	0.00%			
9	50	50	0	0	0.00%			
10	60	60	0	0	0.00%			
QCC-2 (0.254mg/mL)			3326.8	0.2598	102.30%			

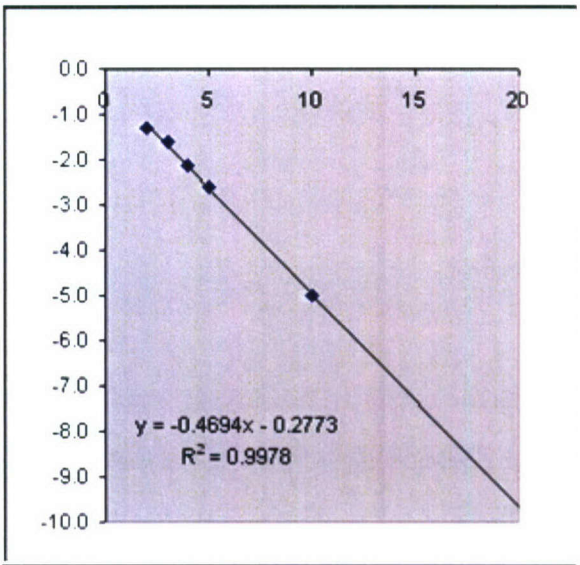
Reactor 2

Sample	Time (min)	Corr.Time (min)	Area (Sulf)	[Agent] µg	% agent remaining	ln (agent)		
							rate	-0.4348 min-1
Start	11						half life	1.59 min
1	13	2	3835.2	0.3096	48.80%	-1.17256151		
2	14	3	2859.9	0.2173	34.20%	-1.52644962		
3	15	4	1703.5	0.122	19.20%	-2.10335264		
4	16	5	1248.7	0.0877	13.80%	-2.4343417		
5	21	10	144.7	0.0097	1.50%	-4.63430139		
6	31	20	0	0	0.00%			
7	41	30	0	0	0.00%			
8	51	40	0	0	0.00%			
9	61	50	0	0	0.00%			
10	71	60	0	0	0.00%			
QCC-3 (0.381mg/mL)			4431.7	0.374	98.20%			

Reactor 3

Sample	Time (min)	Corr.Time (min)	Area (Sulf)	[Agent] µg	% agent remaining	ln (agent)		
							rate	-0.4458 min-1
Start	22						half life	1.55 min
1	24	2	3834.5	0.3095	48.70%	-1.17279229		
2	25	3	2724.9	0.2055	32.40%	-1.58227851		
3	26	4	1778.6	0.1279	20.10%	-2.05671166		
4	27	5	1283	0.0902	14.20%	-2.40574914		
5	32	10	131.6	0.0088	1.40%	-4.72969322		
6	42	20	0	0	0.00%			
7	52	30	0	0	0.00%			
8	62	40	0	0	0.00%			
9	72	50	0	0	0.00%			
10	82	60	0	0	0.00%			
QCC-4 (0.508mg/mL)			5118.1	0.4598	90.50%			


Reactor 1
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60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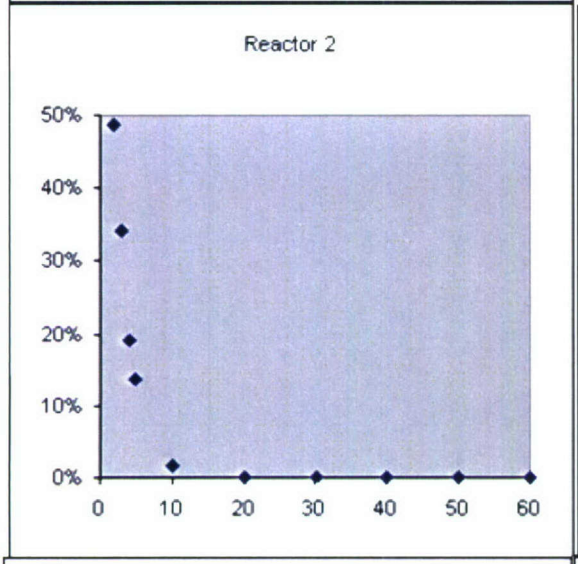
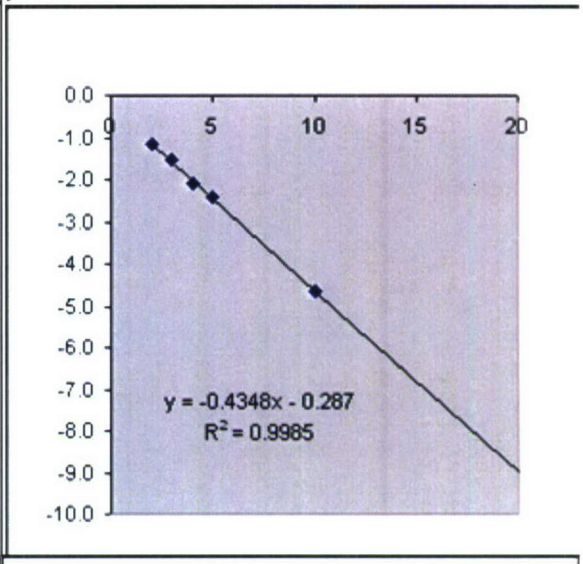
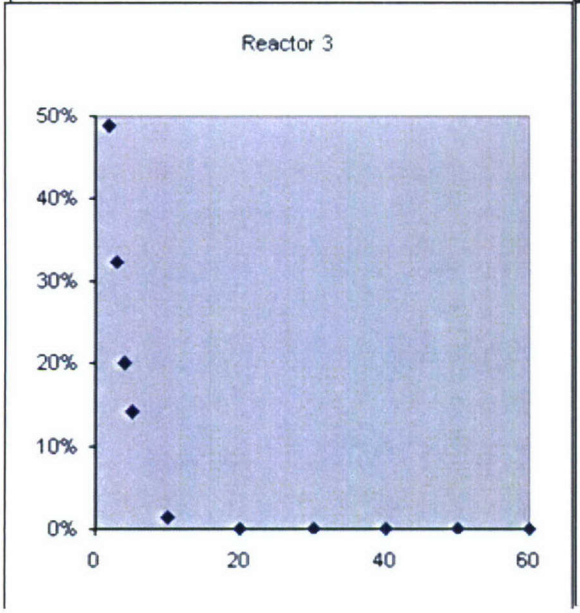
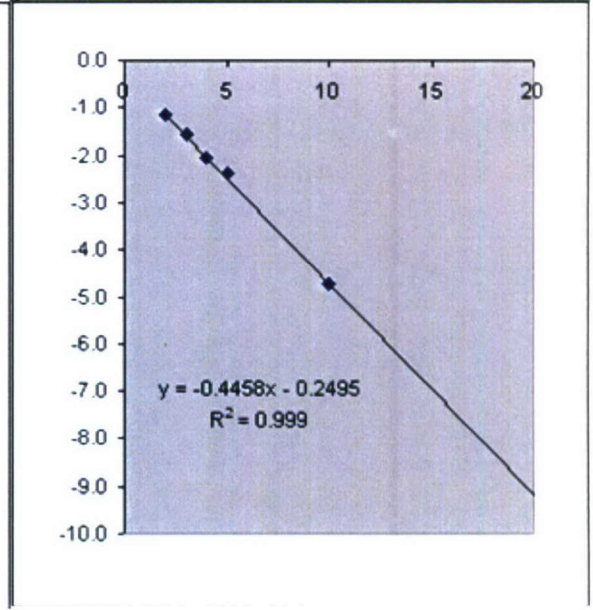
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.4694x - 0.2773
R² = 0.9978


Reactor 2
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.4348x - 0.287
R² = 0.9985


Reactor 3
50%
40%
30%
20%
10%
0%
0
20
40
60


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.4458x - 0.2495
R² = 0.999

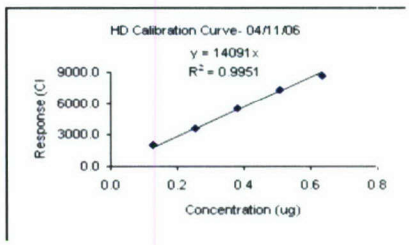
Appendix A9: Chlorine Channel GC-AED Data for CDS-HD Samples

HD vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.27g/mL
Initial agent concentration: 2% v/v
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite/0.2 M Sodium Carbonate and 2.0 mL chloroform
GC: GC-AED, method HD, monitoring Chlorine 479
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

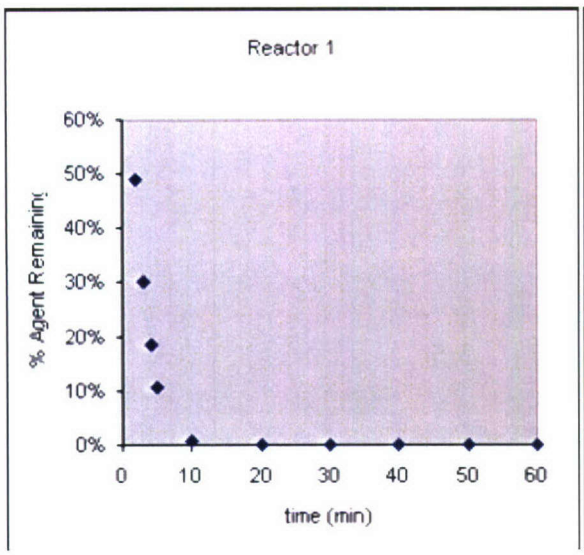
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Chlorine)
STD HD-1	1000	0	0.635	8701.2
STD HD-2	800	200	0.508	7297.8
STD HD-3	600	400	0.381	5524.9
STD HD-4	400	600	0.254	3583.2
STD HD-5	200	800	0.127	1986.1

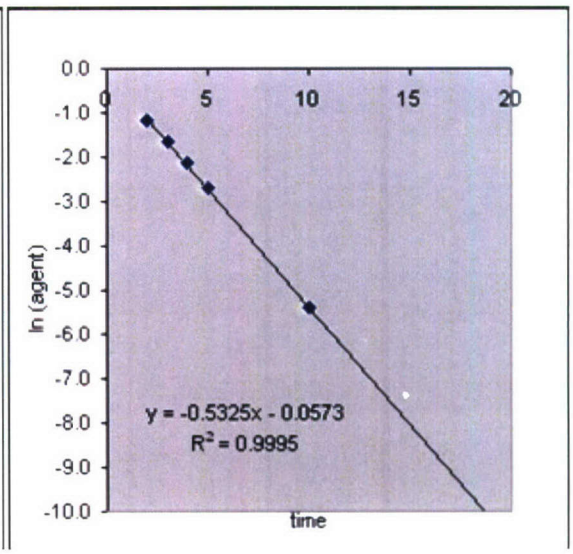


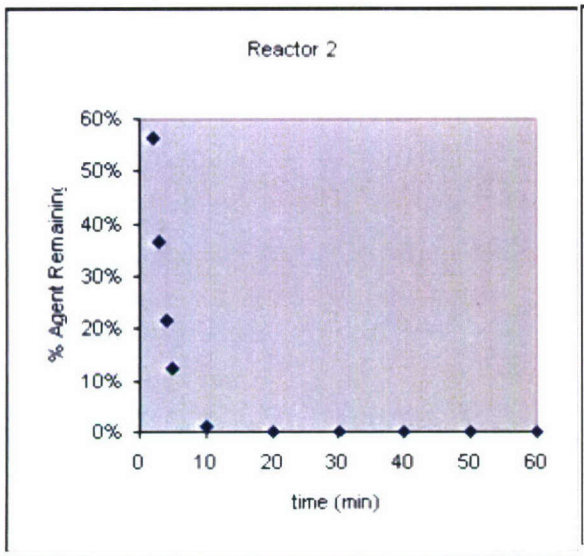
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Chlorine)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.5325 min-1
	1	2	2	4371.8	0.3103	48.90%	-1.17036147	half life	1.3 min
	2	3	3	2690.8	0.191	30.10%	-1.65569775		
	3	4	4	1657	0.1176	18.50%	-2.14052756		
	4	5	5	949.1	0.0674	10.60%	-2.69777741		
	5	10	10	63.5	0.0045	0.70%	-5.40225167		
	6	20	20	0	0	0.00%			
	7	30	30	0	0	0.00%			
	8	40	40	0	0	0.00%			
	9	50	50	0	0	0.00%			
	10	60	60	0	0	0.00%			
	QCC-2 (0.254mg/mL)			3921.6	0.2783	109.60%			

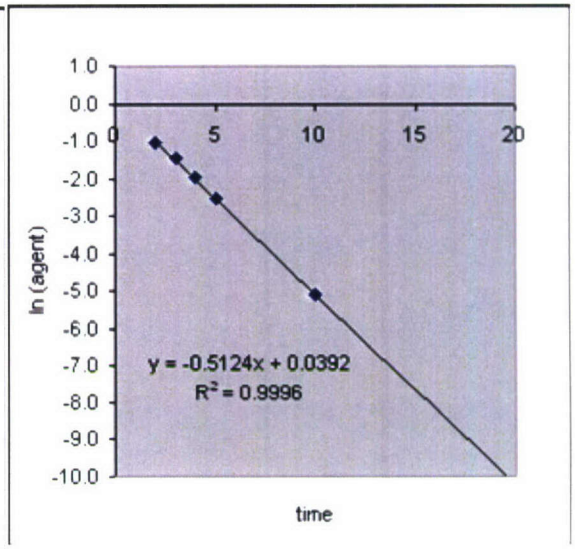
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Chlorine)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.5124 min-1
	Start	11						half life	1.35 min
	1	13	2	5057.6	0.3589	56.50%	-1.02464423		
	2	14	3	3263.1	0.2316	36.50%	-1.46285863		
	3	15	4	1938.4	0.1376	21.70%	-1.98367341		
	4	16	5	1110.2	0.0788	12.40%	-2.54099612		
	5	21	10	86.9	0.0062	1.00%	-5.08853354		
	6	31	20	0	0	0.00%			
	7	41	30	0	0	0.00%			
	8	51	40	0	0	0.00%			
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
	QCC-3 (0.381mg/mL)			5980.4	0.4244	111.40%			

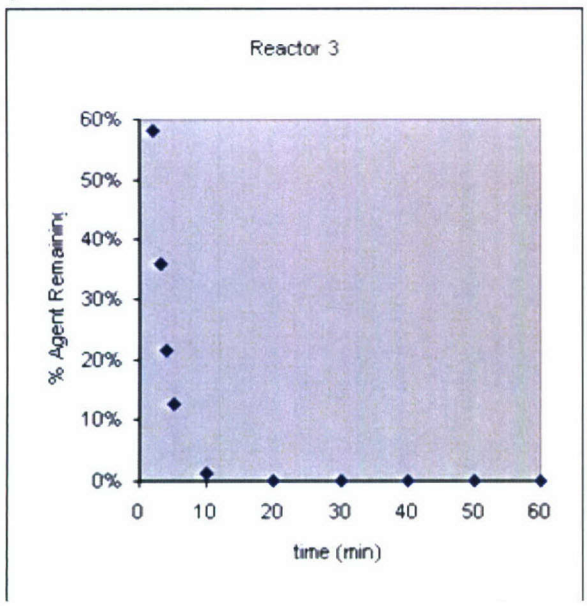
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Chlorine)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.4945 min-1
	Start	22						half life	1.4 min
	1	24	2	5206.5	0.3695	58.20%	-0.99562845		
	2	25	3	3214.4	0.2281	35.90%	-1.47789558		
	3	26	4	1917.7	0.1361	21.40%	-1.99440974		
	4	27	5	1124.7	0.0798	12.60%	-2.52801996		
	5	32	10	100.1	0.0071	1.10%	-4.94712189		
	6	42	20	0	0	0.00%			
	7	52	30	0	0	0.00%			
	8	62	40	0	0	0.00%			
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
	QCC-4 (0.508mg/mL)			7271.4	0.516	101.60%			

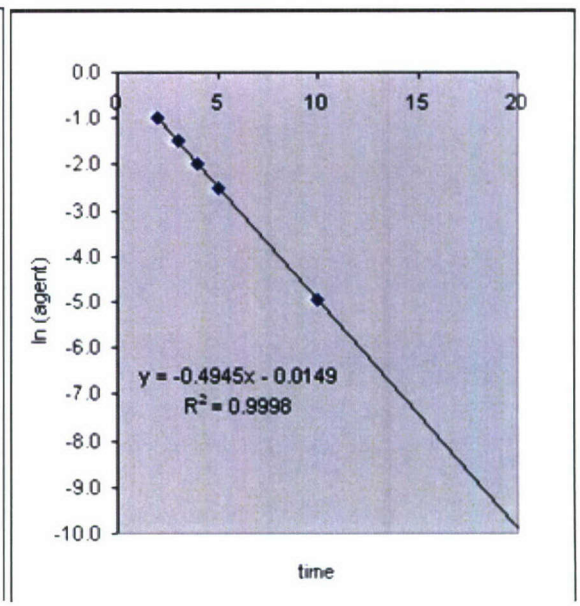

Reactor 1
% Agent Remaining
60%
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.5325x - 0.0573
R² = 0.9995
time


Reactor 2
% Agent Remaining
60%
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
1.0
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.5124x + 0.0392
R² = 0.9996
time


Reactor 3
% Agent Remaining
60%
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -0.4945x - 0.0149
R² = 0.9998
time

Appendix A10: Carbon Channel GC-AED Data for DF200-HD Samples

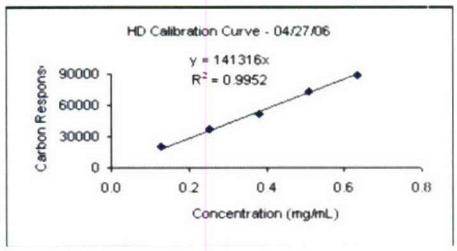
Run: 042706

HD vs. DF200 @ 25 deg C

Reference: d= 1.27g/mL
Initial agent concentration: 2% v/v
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite/0.2 M Sodium Carbonate and 2.0 mL chloroform
GC: GC-AED, method HD, monitoring Carbon 193
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

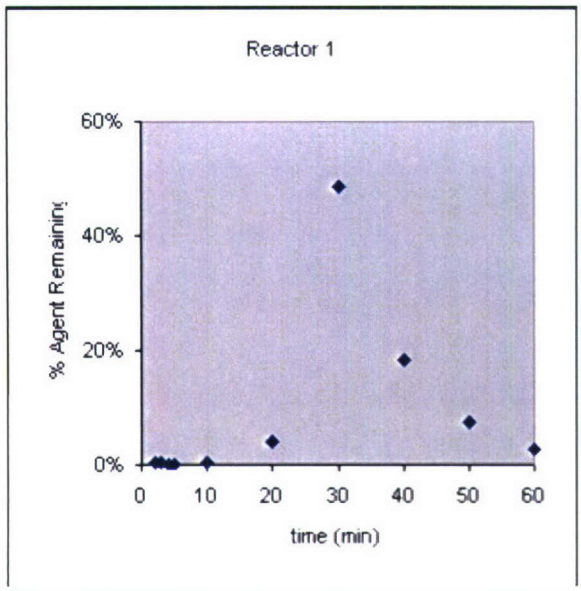
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Carbon)
STD HD-1	1000	0	0.635	88760.3
STD HD-2	800	200	0.508	72736.4
STD HD-3	600	400	0.381	52166.8
STD HD-4	400	600	0.254	37688.7
STD HD-5	200	800	0.127	20467.7

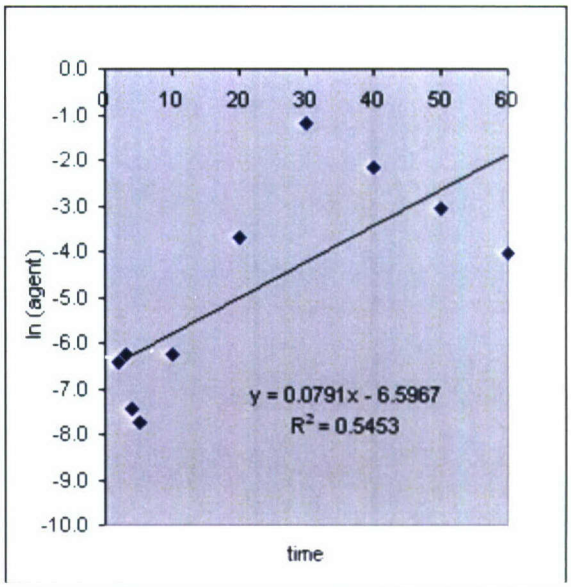


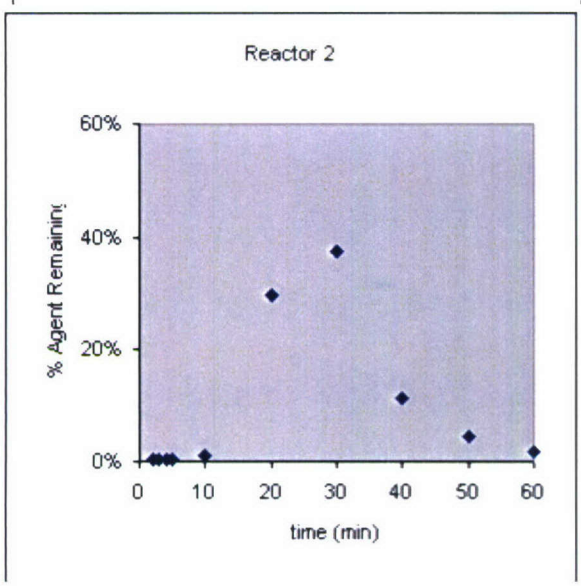
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
								rate	positive	min-1
	1	2	2	228	0.0016	0.30%	-6.4294082	half life	#VALUE!	min
	2	3	3	273.1	0.0019	0.30%	-6.2489158			
	3	4	4	81.2	0.0006	0.10%	-7.4618385			
	4	5	5	62.4	0.0004	0.10%	-7.7251885			
	5	10	10	271	0.0019	0.30%	-6.256635			
	6	20	20	3525.3	0.0249	3.90%	-3.691033			
	7	30	30	43496.2	0.3078	48.50%	-1.1783249			
	8	40	40	16373.9	0.1159	18.20%	-2.1553099			
	9	50	50	6630.7	0.0469	7.40%	-3.0592881			
	10	60	60	2476.2	0.0175	2.80%	-4.0442734			
	QCC-2 (0.254mg/mL)			39984	0.2829	111.40%				

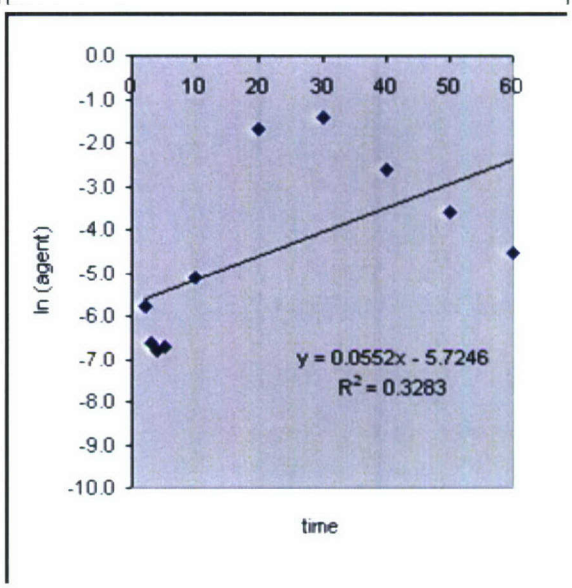
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
								rate	positive	min-1
	Start	11						half life	#VALUE!	min
	1	13	2	439.5	0.0031	0.50%	-5.7731161			
	2	14	3	190.7	0.0013	0.20%	-6.6080523			
	3	15	4	156	0.0011	0.20%	-6.8088978			
	4	16	5	169.9	0.0012	0.20%	-6.7235438			
	5	21	10	868.6	0.0061	1.00%	-5.0918711			
	6	31	20	26483.1	0.1874	29.50%	-1.6744917			
	7	41	30	33564.5	0.2375	37.40%	-1.4375296			
	8	51	40	10090.4	0.0714	11.20%	-2.639414			
	9	61	50	3859.4	0.0273	4.30%	-3.6004868			
	10	71	60	1515.6	0.0107	1.70%	-4.5351871			
	QCC-3 (0.381mg/mL)			58758.8	0.4158	109.10%				

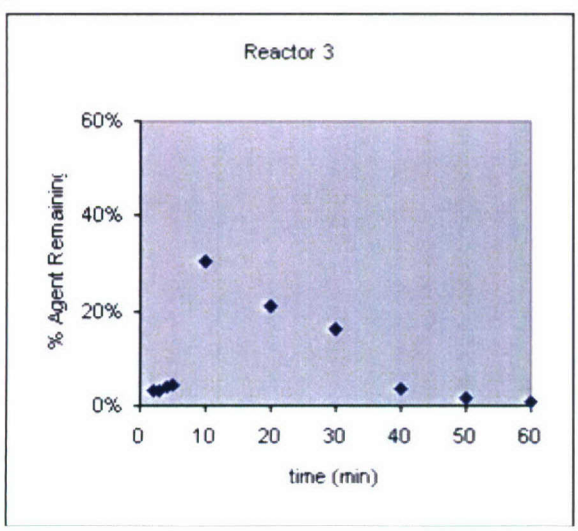
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)			
								rate	-0.0267	min-1
	Start	22						half life	25.96	min
	1	24	2	2946.4	0.0208	3.30%	-3.8704144			
	2	25	3	2954.5	0.0209	3.30%	-3.8676691			
	3	26	4	3589.6	0.0254	4.00%	-3.6729577			
	4	27	5	4136	0.0293	4.60%	-3.5312694			
	5	32	10	27393.3	0.1938	30.50%	-1.6407001			
	6	42	20	18956.4	0.1341	21.10%	-2.0088569			
	7	52	30	14485.6	0.1025	16.10%	-2.2778435			
	8	62	40	3154.8	0.0223	3.50%	-3.8020734			
	9	72	50	1326.6	0.0094	1.50%	-4.6683792			
	10	82	60	597.4	0.0042	0.70%	-5.4661669			
	QCC-4 (0.508mg/mL)			77963.6	0.5517	108.60%				

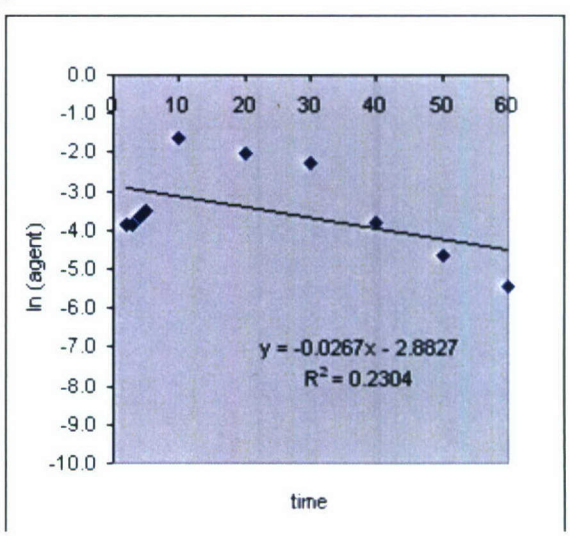

Reactor 1
% Agent Remaining
60%
40%
20%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = 0.0791x - 6.5967
R² = 0.5453
time


Reactor 2
% Agent Remaining
60%
40%
20%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = 0.0552x - 5.7246
R² = 0.3283
time


Reactor 3
% Agent Remaining
60%
40%
20%
0%
0
10
20
30
40
50
60
time (min)


ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.0267x - 2.8827
R² = 0.2304
time

Appendix A11: Sulfur Channel GC-AED Data for DF200-HD Samples

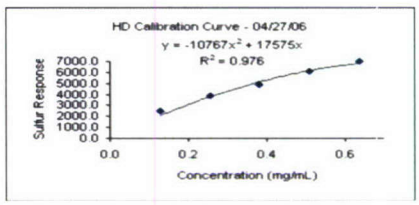
Run: 042706

HD vs. DF200 @ 25 deg C

Reference: d= 1.27g/mL
Initial agent concentration: 2% v/v
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite/0.2 M Sodium Carbonate and 2.0 mL chloroform
GC: GC-AED, method HD, monitoring Sulfur 181
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

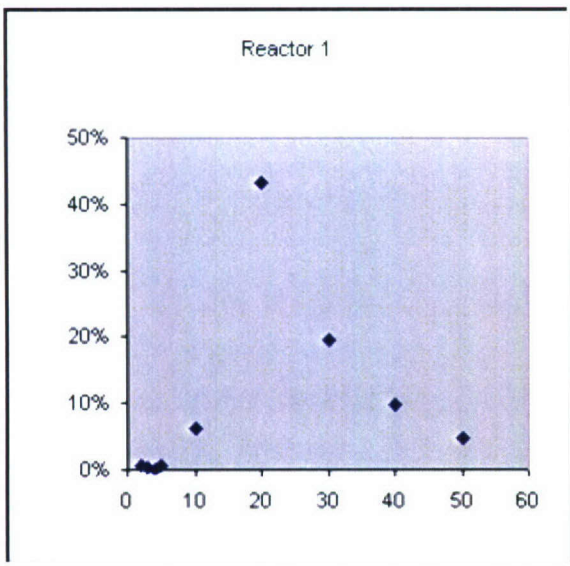
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Sulf)
STD HD-1	1000	0	0.635	6959.1
STD HD-2	800	200	0.508	6047.5
STD HD-3	600	400	0.381	4844.6
STD HD-4	400	600	0.254	3842.2
STD HD-5	200	800	0.127	2484.1



Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Sulf)	[Agent] µg	% agent remaining	ln (agent)			
								rate	positive	min-1
	1	2	2	46	0.0026	0.40%	-5.94398398	half life	#VALUE!	min
	2	3	3	64.5	0.0037	0.60%	-5.60531152			
	3	4	4	18.3	0.001	0.20%	-6.86669314			
	4	5	5	15.7	0.0009	0.10%	-7.02002428			
	5	10	10	64.1	0.0037	0.60%	-5.61154642			
	6	20	20	685.8	0.04	6.30%	-3.21883514			
	7	30	30	4016.4	0.2748	43.30%	-1.29175529			
	8	40	40	2016.4	0.1242	19.60%	-2.08603889			
	9	50	50	1054.3	0.0624	9.80%	-2.77464021			
	10	60	60	532.8	0.0309	4.90%	-3.47697433			
	QCC-2 (0.254mg/mL)			3926.1	0.2671	105.20%				

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Sulf)	[Agent] µg	% agent remaining	ln (agent)			
								rate	positive	min-1
	Start	11						half life	#VALUE!	min
	1	13	2	112.6	0.0064	1.00%	-5.04644266			
	2	14	3	39.9	0.0023	0.40%	-6.08646264			
	3	15	4	33.2	0.0019	0.30%	-6.27052354			
	4	16	5	41.2	0.0023	0.40%	-6.0543552			
	5	21	10	209.2	0.012	1.90%	-4.42356858			
	6	31	20	2862.4	0.1835	28.90%	-1.69556597			
	7	41	30	3273.3	0.2144	33.80%	-1.53985766			
	8	51	40	1428.7	0.0858	13.50%	-2.45571569			
	9	61	50	707.4	0.0413	6.50%	-3.18701227			
	10	71	60	370.7	0.0214	3.40%	-3.84565979			
	QCC-3 (0.381mg/mL)			5023	0.3694	97.00%				

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Sulf)	[Agent] µg	% agent remaining	ln (agent)			
								rate	-0.0226	min-1
	Start	22						half life	30.66	min
	1	24	2	573.6	0.0333	5.20%	-3.401678			
	2	25	3	570.1	0.0331	5.20%	-3.40792832			
	3	26	4	685.1	0.04	6.30%	-3.21988267			
	4	27	5	749.3	0.0438	6.90%	-3.12788696			
	5	32	10	2949.4	0.1899	29.90%	-1.66118458			
	6	42	20	2186.6	0.1357	21.40%	-1.99733823			
	7	52	30	1819.8	0.1111	17.50%	-2.19725532			
	8	62	40	651.7	0.038	6.00%	-3.27111567			
	9	72	50	359.6	0.0207	3.30%	-3.87646295			
	10	82	60	121.5	0.0069	1.10%	-4.97005603			
	QCC-4 (0.508mg/mL)			6094.4	0.4998	98.40%				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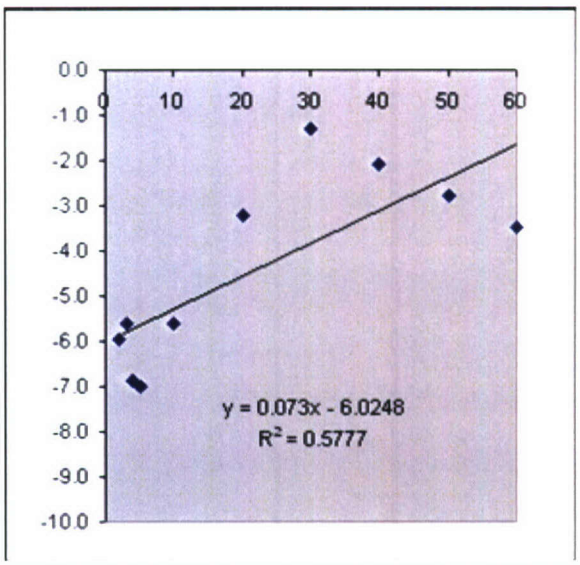
40%
30%
20%
10%
0%
0
10
20
30
40
50
60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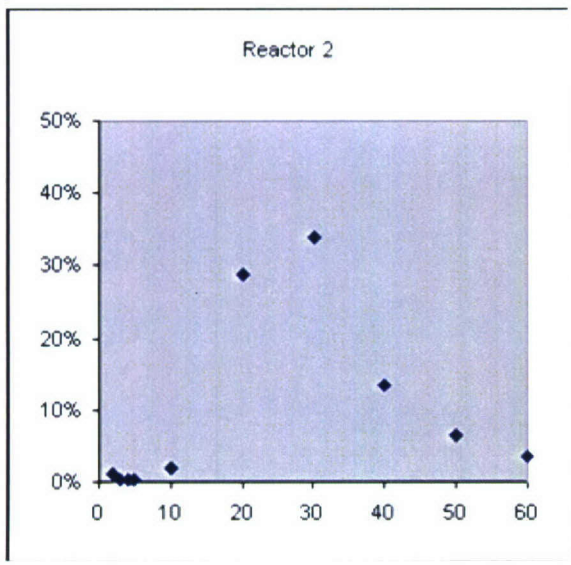
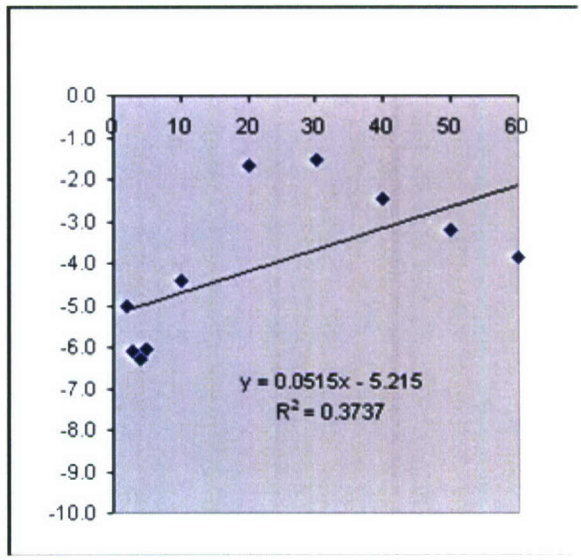
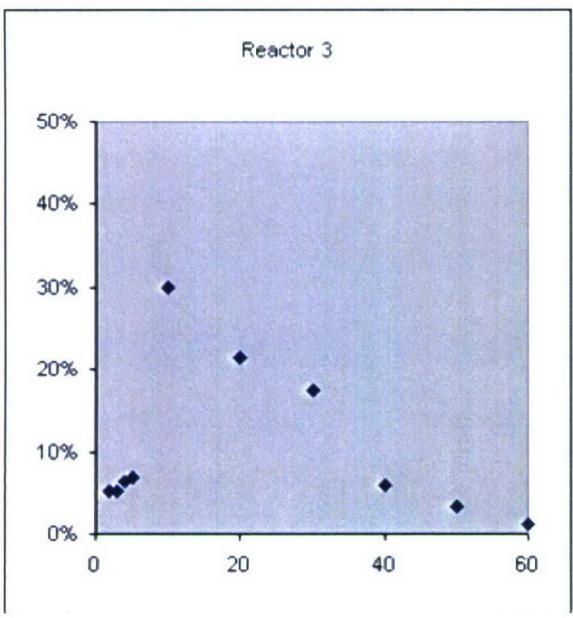
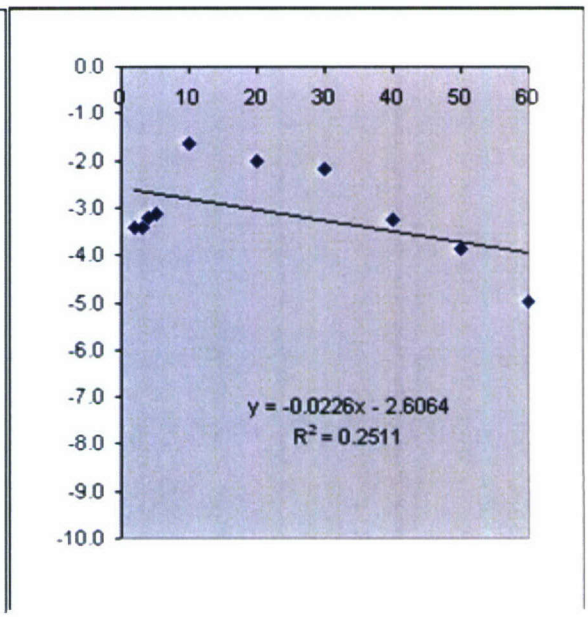
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = 0.073x - 6.0248
R² = 0.5777


Reactor 2
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = 0.0515x - 5.215
R² = 0.3737


Reactor 3
50%
40%
30%
20%
10%
0%
0
20
40
60


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.0226x - 2.6064
R² = 0.2511

Appendix A12: Chlorine Channel GC-AED Data for DF200-HD Samples

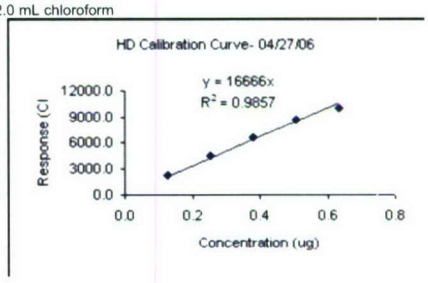
Run: 042706

HD vs. DF200 @ 25 deg C

Reference: d= 1.27g/mL
Initial agent concentration: 2% v/v
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite/0.2 M Sodium Carbonate and 2.0 mL chloroform
GC: GC-AED, method HD, monitoring Chlorine 479
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

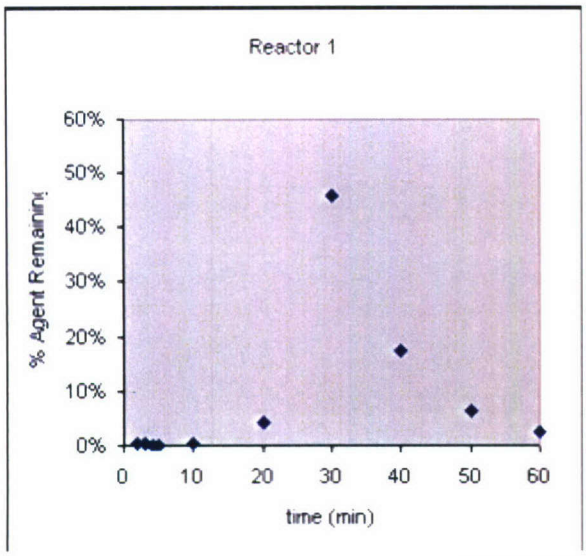
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Chlorine)
STD HD-1	1000	0	0.635	10055.9
STD HD-2	800	200	0.508	8690.9
STD HD-3	600	400	0.381	6657.6
STD HD-4	400	600	0.254	4545.7
STD HD-5	200	800	0.127	2303.2

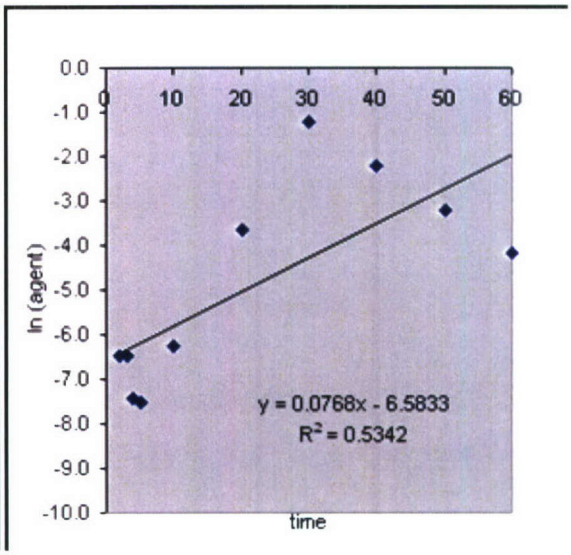


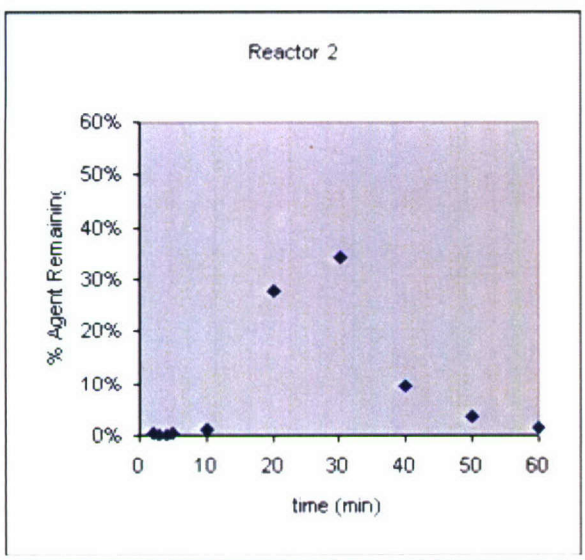
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Chlorine)	[Agent] µg	% agent remaining	ln (agent)			
								rate	positive	min-1
	1	2	2	25.6	0.0015	0.20%	-6.47853364	half life	#VALUE!	min
	2	3	3	25.4	0.0015	0.20%	-6.48637682			
	3	4	4	10	0.0006	0.10%	-7.4185409			
	4	5	5	9.1	0.0005	0.10%	-7.51285158			
	5	10	10	31.8	0.0019	0.30%	-6.26165971			
	6	20	20	436.4	0.0262	4.10%	-3.64256674			
	7	30	30	4864.7	0.2919	46.00%	-1.23136567			
	8	40	40	1850.9	0.1111	17.50%	-2.19769871			
	9	50	50	666.6	0.04	6.30%	-3.21893583			
	10	60	60	256.9	0.0154	2.40%	-4.17243909			
	QCC-2 (0.254mg/mL)			4284.5	0.2571	101.20%				

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Chlorine)	[Agent] µg	% agent remaining	ln (agent)			
								rate	positive	min-1
	Start	11						half life	#VALUE!	min
	1	13	2	54	0.0032	0.50%	-5.73214195			
	2	14	3	19	0.0011	0.20%	-6.77668702			
	3	15	4	16.2	0.001	0.20%	-6.93611475			
	4	16	5	19.4	0.0012	0.20%	-6.75585293			
	5	21	10	98.1	0.0059	0.90%	-5.13513863			
	6	31	20	2943.6	0.1766	27.80%	-1.73373739			
	7	41	30	3641.3	0.2185	34.40%	-1.52102996			
	8	51	40	1023.9	0.0614	9.70%	-2.78975185			
	9	61	50	386.5	0.0232	3.70%	-3.76399413			
	10	71	60	161.4	0.0097	1.50%	-4.63724024			
	QCC-3 (0.381mg/mL)			7018.8	0.4211	110.50%				

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Chlorine)	[Agent] µg	% agent remaining	ln (agent)			
								rate	-0.0251	min-1
	Start	22						half life	27.61	min
	1	24	2	306.8	0.0184	2.90%	-3.99492993			
	2	25	3	285.6	0.0171	2.70%	-4.06653376			
	3	26	4	436.8	0.0262	4.10%	-3.64165057			
	4	27	5	461.3	0.0277	4.40%	-3.5870774			
	5	32	10	3315.8	0.199	31.30%	-1.61467179			
	6	42	20	2149.4	0.129	20.30%	-2.04818198			
	7	52	30	1525.6	0.0915	14.40%	-2.39098294			
	8	62	40	342.3	0.0205	3.20%	-3.88543845			
	9	72	50	142.7	0.0086	1.30%	-4.76038147			
	10	82	60	80.3	0.0048	0.80%	-5.33535637			
	QCC-4 (0.508mg/mL)			8560.6	0.5137	101.10%				


Reactor 1
% Agent Remainin
60%
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
y = 0.0768x - 6.5833
R² = 0.5342
time


Reactor 2
% Agent Remainin
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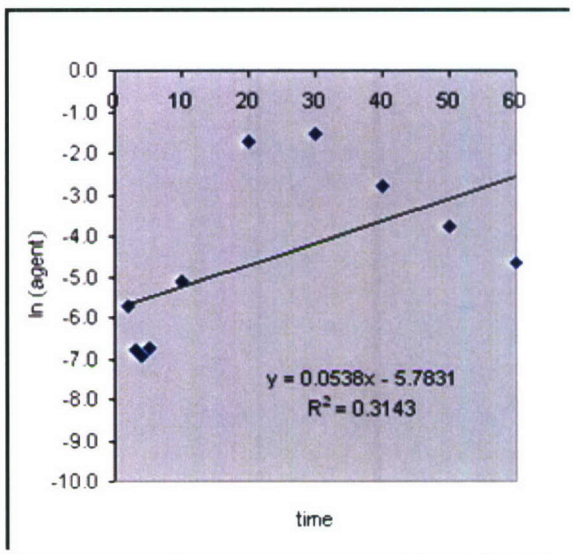
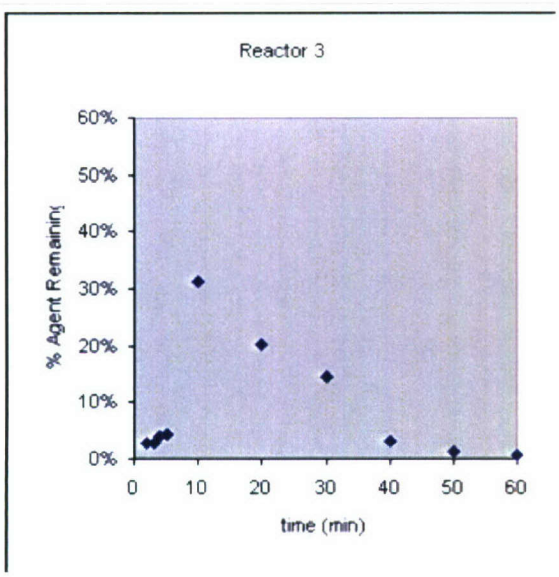
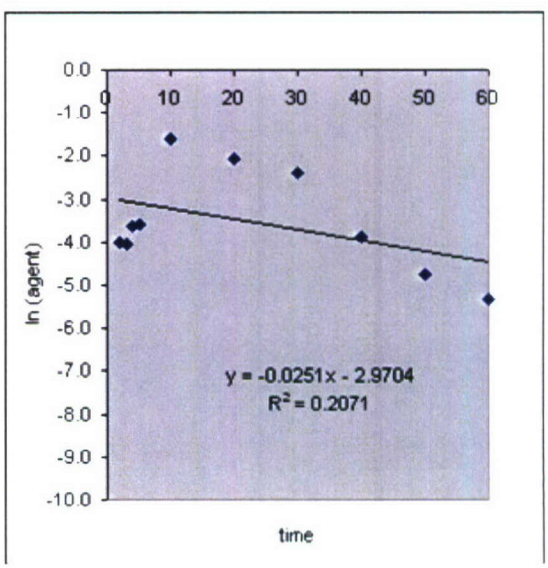
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
y = 0.0538x - 5.7831
R² = 0.3143
time


Reactor 3
% Agent Remainin
60%
50%
40%
30%
20%
10%
0%
0
10
20
30
40
50
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
ln (agent)
y = -0.0251x - 2.9704
R² = 0.2071
time

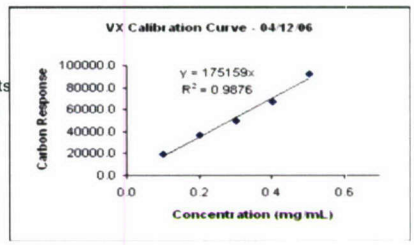
Appendix A13: Carbon Channel GC-AED Data for CDS-VX Samples

Run 041206

VX vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.008g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method VX, monitoring Carbon 193
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

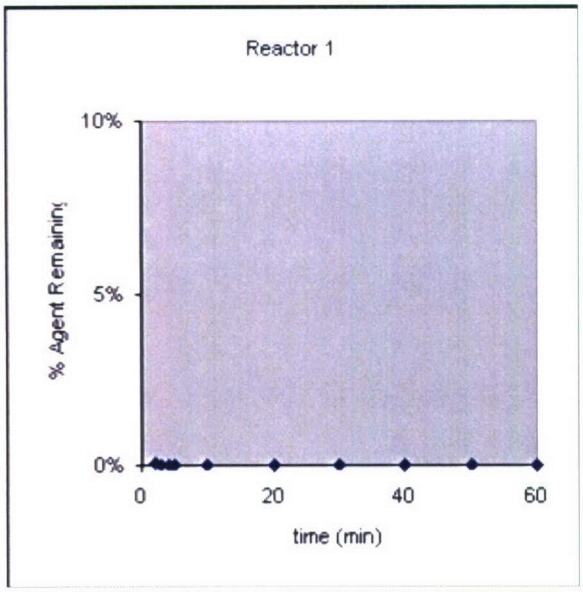
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Carbon)
STD VX-1	1000	0	0.504	91819.5
STD VX-2	800	200	0.4032	67244.7
STD VX-3	600	400	0.3024	49921.5
STD VX-4	400	600	0.2016	36974.9
STD VX-5	200	800	0.1008	19288.6

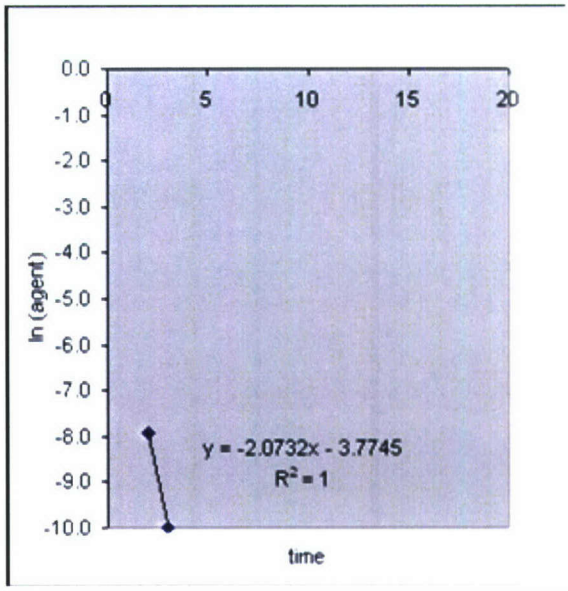


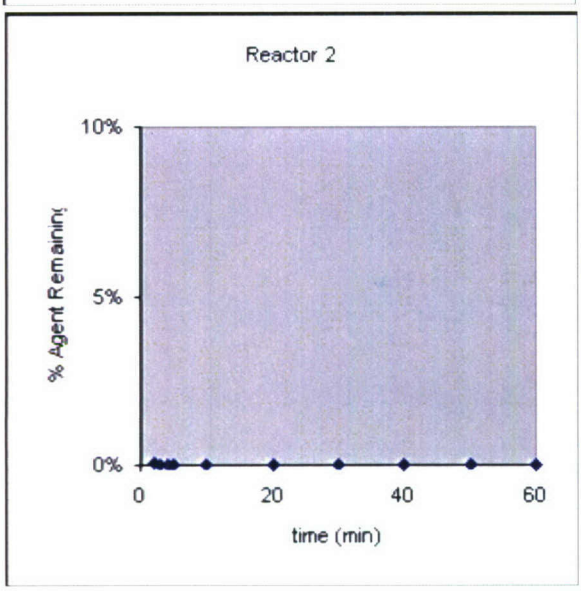
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-2.0732 min-1
	1	2	2	63.6	0.0004	0.10%	-7.92083594	half life	0.33 min
	2	3	3	8	0	0.00%	-9.99400787		
	3	4	4	0	0	0.00%			
	4	5	5	0	0	0.00%			
	5	10	10	0	0	0.00%			
	6	20	20	0	0	0.00%			
	7	30	30	0	0	0.00%			
	8	40	40	0	0	0.00%			
	9	50	50	0	0	0.00%			
	10	60	60	0	0	0.00%			
QCC-2 (0.2016mg/mL)				34799.6	0.1987	98.50%			

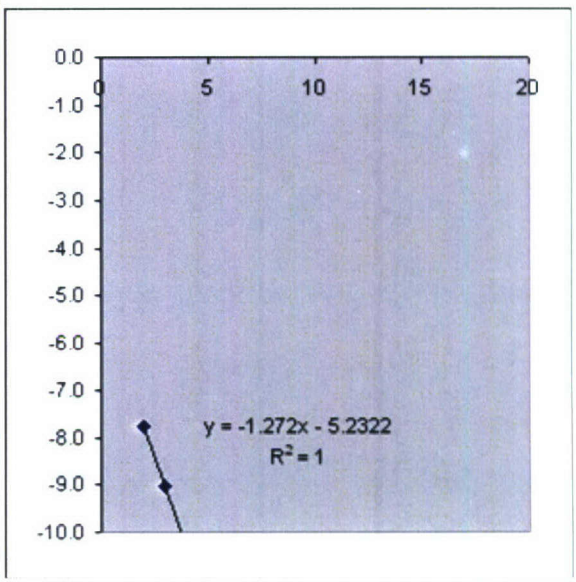
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.272 min-1
	Start	11						half life	0.54 min
	1	13	2	73.5	0.0004	0.10%	-7.77616401		
	2	14	3	20.6	0.0001	0.00%	-9.04815834		
	3	15	4	0	0	0.00%			
	4	16	5	0	0	0.00%			
	5	21	10	0	0	0.00%			
	6	31	20	0	0	0.00%			
	7	41	30	0	0	0.00%			
	8	51	40	0	0	0.00%			
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
QCC-3 (0.3024mg/ml)				51945.6	0.2966	98.10%			

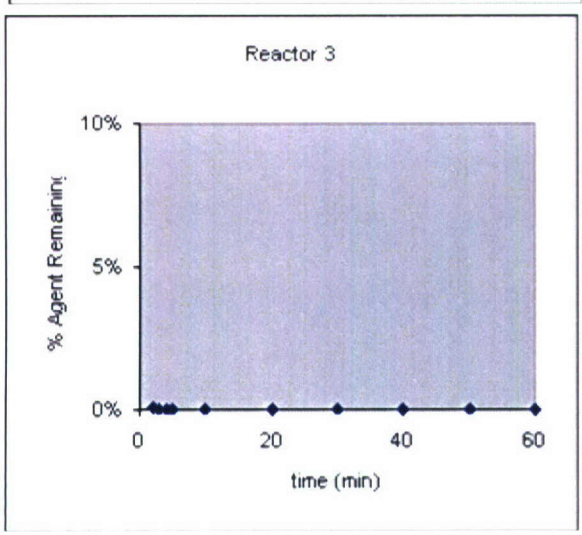
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.5356 min-1
	Start	22						half life	0.45 min
	1	24	2	69.2	0.0004	0.10%	-7.83644855		
	2	25	3	14.9	0.0001	0.00%	-9.3720882		
	3	26	4	0	0	0.00%			
	4	27	5	0	0	0.00%			
	5	32	10	0	0	0.00%			
	6	42	20	0	0	0.00%			
	7	52	30	0	0	0.00%			
	8	62	40	0	0	0.00%			
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
QCC-4 (0.4032mg/mL)				74337.7	0.4244	105.30%			

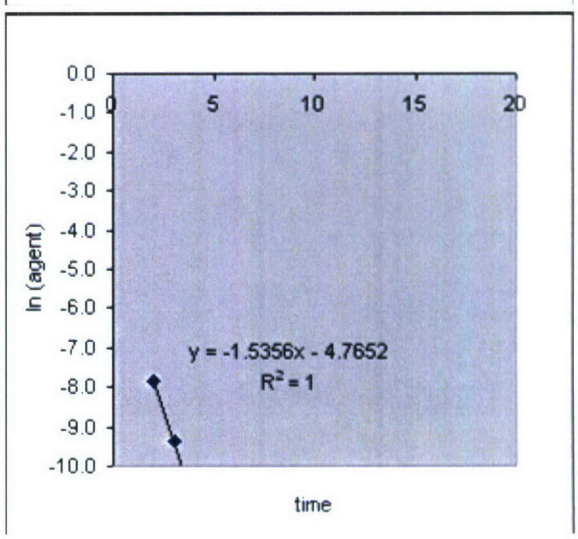

Reactor 1
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -2.0732x - 3.7745
R² = 1
time


Reactor 2
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
y = -1.272x - 5.2322
R² = 1

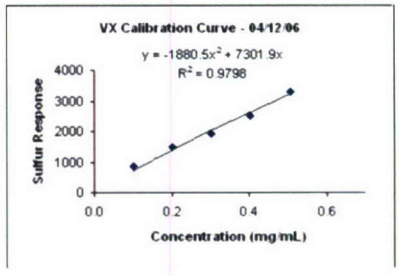

Reactor 3
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -1.5356x - 4.7652
R² = 1
time

Appendix A14: Sulfur Channel GC-AED Data for CDS-VX Samples

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method VX, monitoring Sulfur 181
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

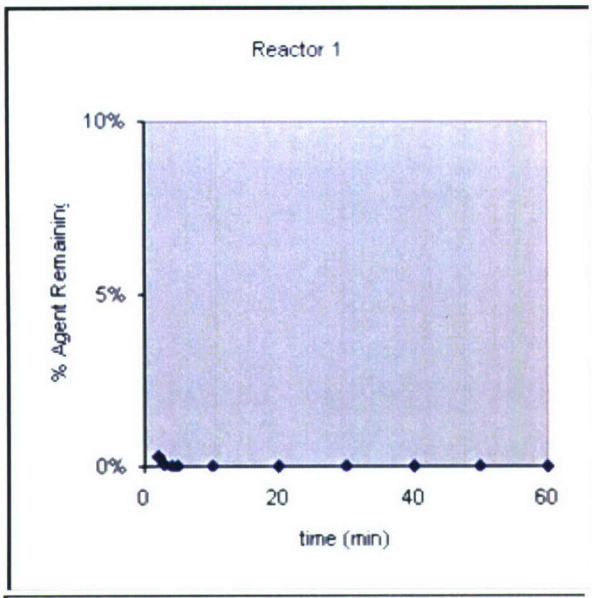
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Sulfur)
STD VX-1	1000	0	0.504	3303.6
STD VX-2	800	200	0.4032	2508.8
STD VX-3	600	400	0.3024	1926
STD VX-4	400	600	0.2016	1492.3
STD VX-5	200	800	0.1008	866.7

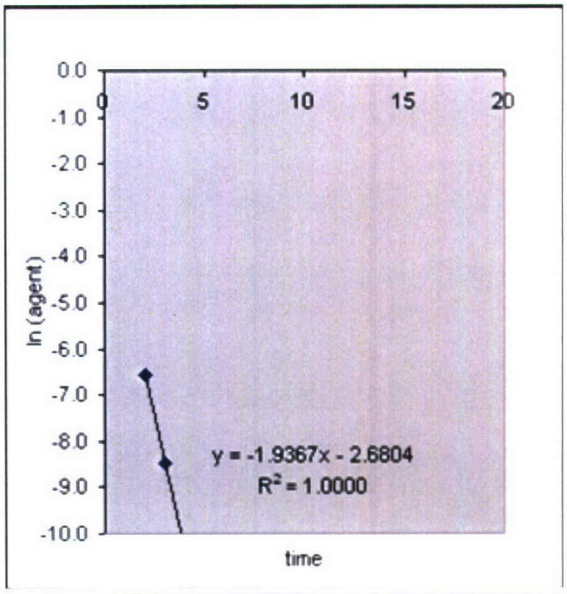


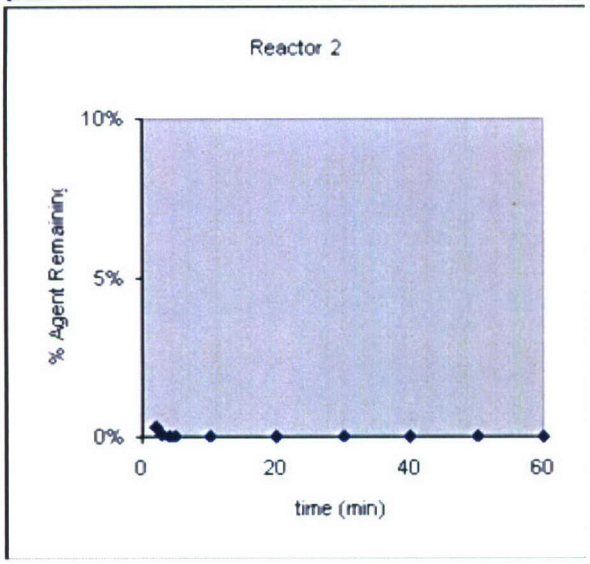
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Sulfur)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.9367 min-1
	1	2	2	10.4	0.0014	0.30%	-6.55371705	half life	0.36 min
	2	3	3	1.5	0.0002	0.00%	-8.49037185		
	3	4	4	0	0	0.00%			
	4	5	5	0	0	0.00%			
	5	10	10	0	0	0.00%			
	6	20	20	0	0	0.00%			
	7	30	30	0	0	0.00%			
	8	40	40	0	0	0.00%			
	9	50	50	0	0	0.00%			
	10	60	60	0	0	0.00%			
	QCC-2 (0.2016mg/mL)			1451.3	0.2101	104.10%			

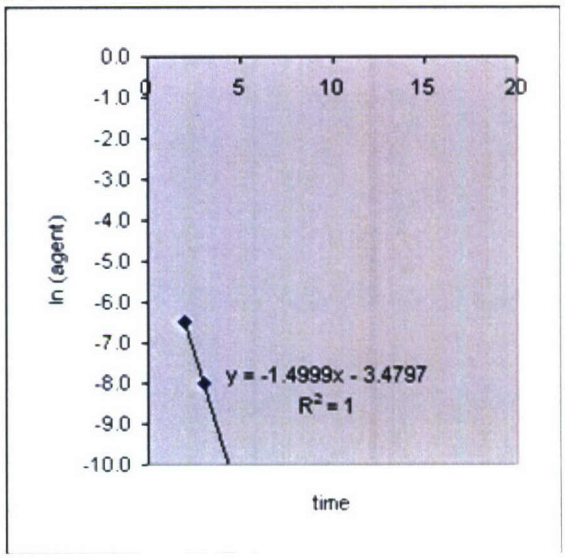
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Sulfur)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.4999 min-1
	Start	11						half life	0.46 min
	1	13	2	11.2	0.0015	0.30%	-6.47958083		
	2	14	3	2.5	0.0003	0.10%	-7.97951095		
	3	15	4	0	0	0.00%			
	4	16	5	0	0	0.00%			
	5	21	10	0	0	0.00%			
	6	31	20	0	0	0.00%			
	7	41	30	0	0	0.00%			
	8	51	40	0	0	0.00%			
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
	QCC-3 (0.3024mg/mL)			2022.7	0.3002	99.28%			

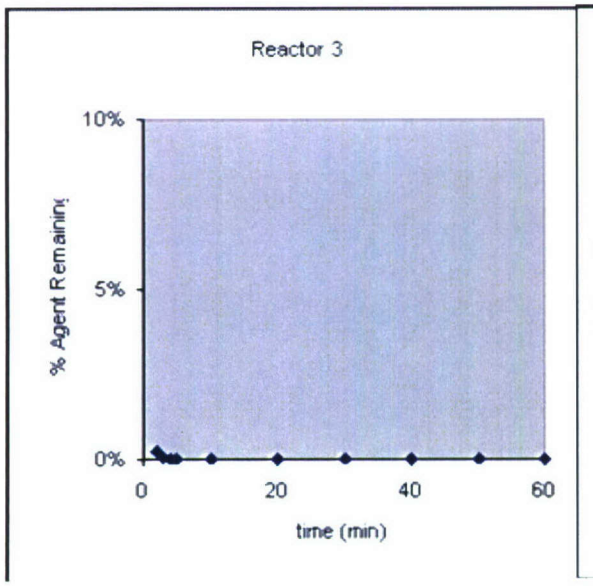
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Sulfur)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.6669 min-1
	Start	22						half life	0.42 min
	1	24	2	9	0.0012	0.20%	-6.69834771		
	2	25	3	1.7	0.0002	0.00%	-8.36520165		
	3	26	4	0	0	0.00%			
	4	27	5	0	0	0.00%			
	5	32	10	0	0	0.00%			
	6	42	20	0	0	0.00%			
	7	52	30	0	0	0.00%			
	8	62	40	0	0	0.00%			
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
	QCC-4 (0.4032mg/mL)			2716.5	0.4168	103.40%			

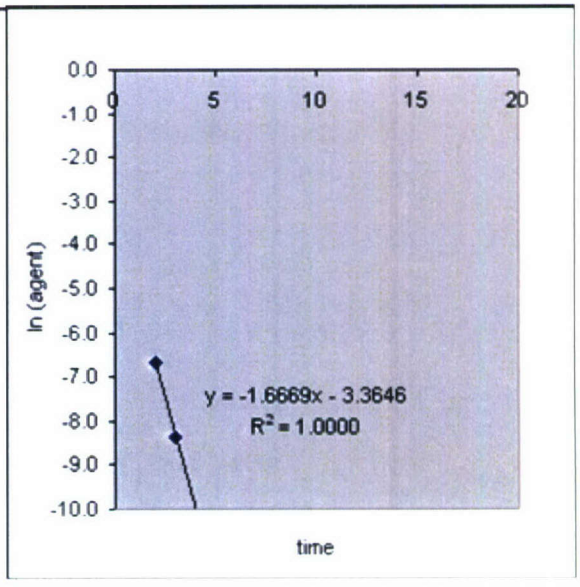

Reactor 1
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -1.9367x - 2.6804
R² = 1.0000
time


Reactor 2
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -1.4999x - 3.4797
R² = 1
time


Reactor 3
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -1.6669x - 3.3646
R² = 1.0000
time

Appendix A15: Phosphorus Channel GC-AED Data for CDS-VX Samples

Run 041206

VX vs. Clean Earth CDS @ 25 deg C

Reference: d= 1.0222g/mL

Initial agent concentration: 2% v/v

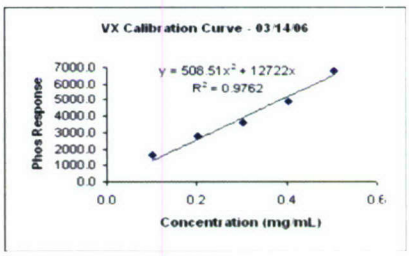
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform

GC: GC-AED, method VX, monitoring Phosphorus 178

Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

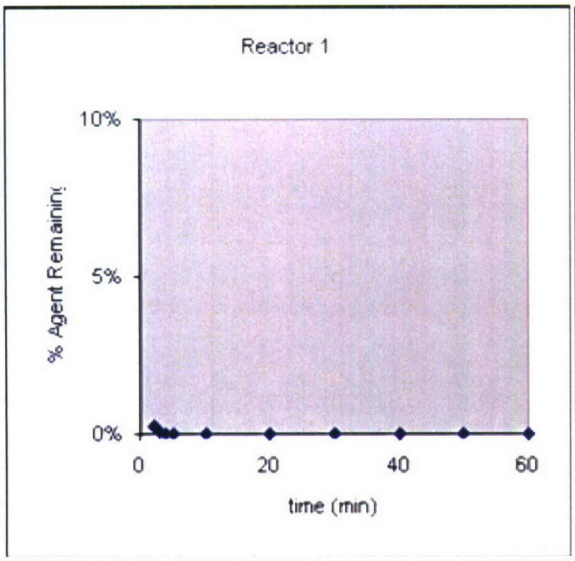
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Phos)
STD VX-1	1000	0	0.504	6769.9
STD VX-2	800	200	0.4032	4924.2
STD VX-3	600	400	0.3024	3632.3
STD VX-4	400	600	0.2016	2807.8
STD VX-5	200	800	0.1008	1633.8

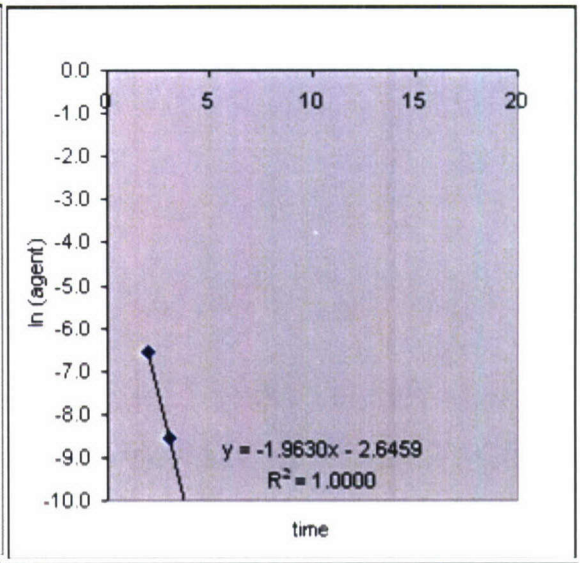


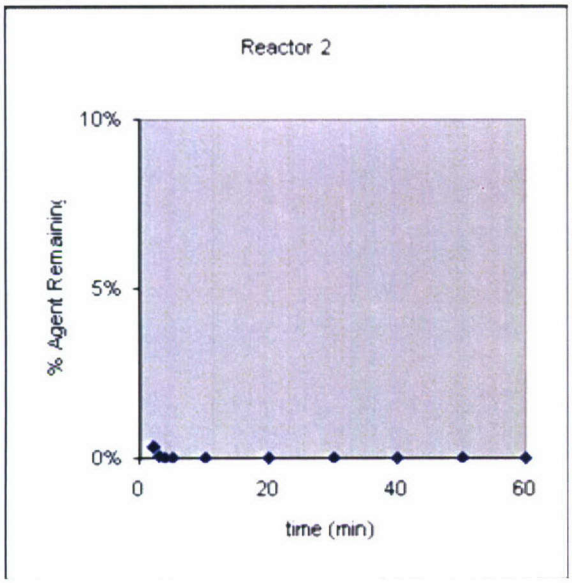
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.963 min-1
	1	2	2	17.8	0.0014	0.30%	-6.57183367	half life	0.35 min
	2	3	3	2.5	0.0002	0.00%	-8.53478947		
	3	4	4	0	0	0.00%			
	4	5	5	0	0	0.00%			
	5	10	10	0	0	0.00%			
	6	20	20	0	0	0.00%			
	7	30	30	0	0	0.00%			
	8	40	40	0	0	0.00%			
	9	50	50	0	0	0.00%			
	10	60	60	0	0	0.00%			
	QCC-2 (0.2016mg/mL)			2682.4	0.2127	105.50%			

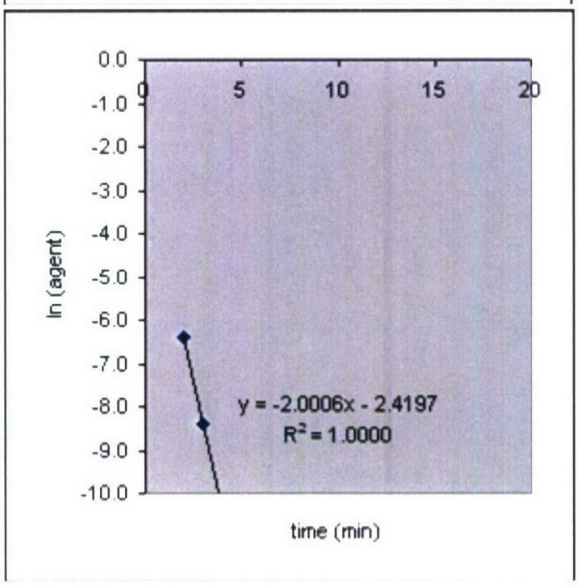
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-2.0006 min-1
	Start	11						half life	0.35 min
	1	13	2	20.7	0.0016	0.30%	-6.42088932		
	2	14	3	2.8	0.0002	0.00%	-8.42145984		
	3	15	4	0	0	0.00%			
	4	16	5	0	0	0.00%			
	5	21	10	0	0	0.00%			
	6	31	20	0	0	0.00%			
	7	41	30	0	0	0.00%			
	8	51	40	0	0	0.00%			
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
	QCC-3 (0.3024mg/mL)			3776.6	0.3005	99.40%			

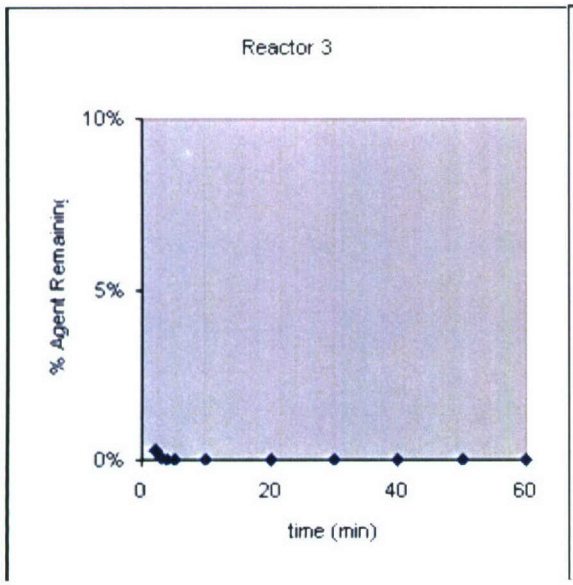
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-1.9111 min-1
	Start	22						half life	0.36 min
	1	24	2	16.9	0.0013	0.30%	-6.62372133		
	2	25	3	2.5	0.0002	0.00%	-8.53478947		
	3	26	4	0	0	0.00%			
	4	27	5	0	0	0.00%			
	5	32	10	0	0	0.00%			
	6	42	20	0	0	0.00%			
	7	52	30	0	0	0.00%			
	8	62	40	0	0	0.00%			
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
	QCC-4 (0.4032mg/mL)			5270.2	0.4214	104.50%			

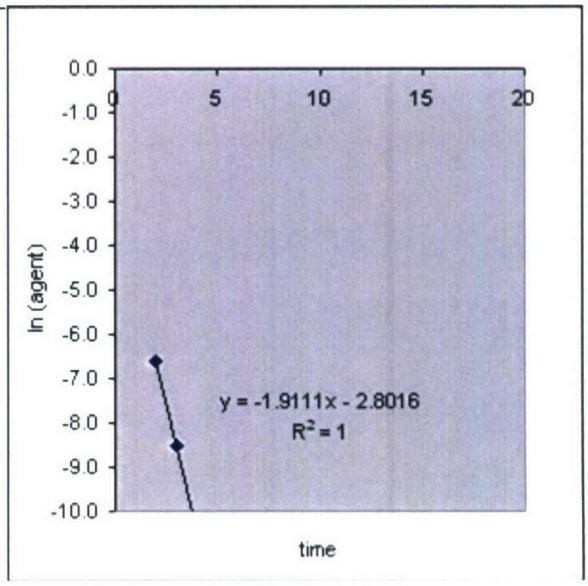

Reactor 1
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -1.9630x - 2.6459
R² = 1.0000
time


Reactor 2
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -2.0006x - 2.4197
R² = 1.0000
time (min)

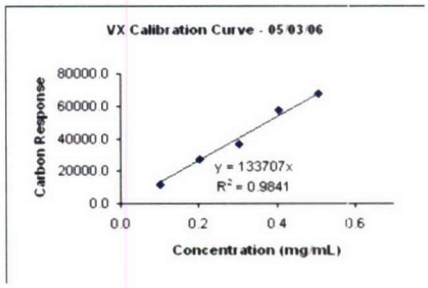

Reactor 3
10%
5%
0%
% Agent Remainin
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
5
10
15
20
ln (agent)
y = -1.9111x - 2.8016
R² = 1
time

Appendix A16: Carbon Channel GC-AED Data for DF200-VX Samples

Run 050306

VX vs. DF200 @ 25 deg C



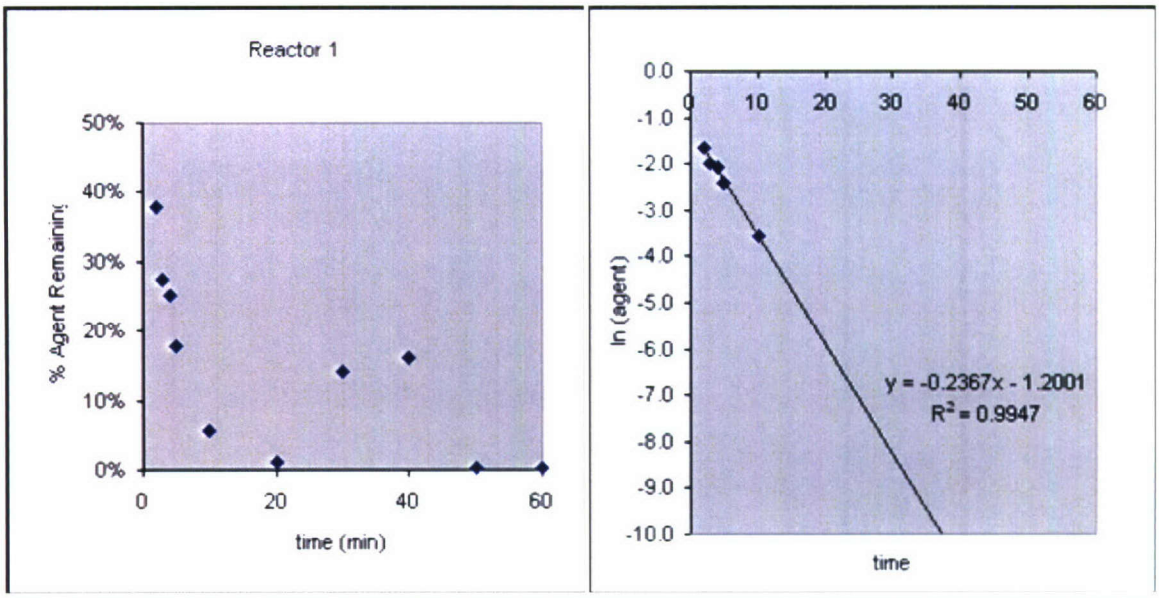
Reference: d= 1.008g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method VX, monitoring Carbon 193
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

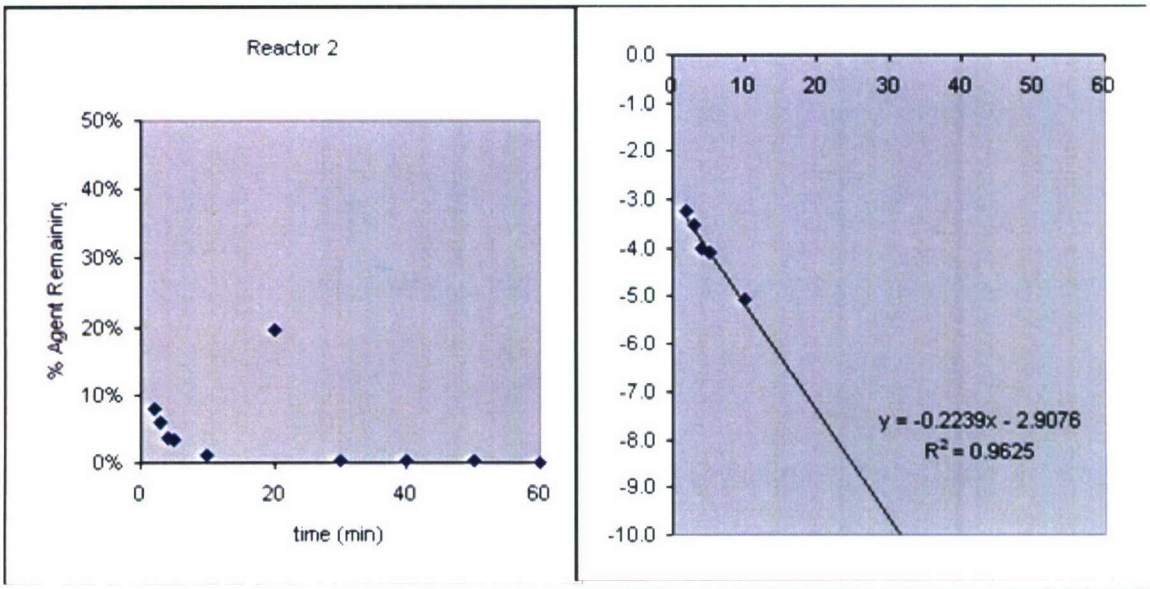
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Carbon)
STD VX-1	1000	0	0.504	67241.6
STD VX-2	800	200	0.4032	57358.5
STD VX-3	600	400	0.3024	36477.4
STD VX-4	400	600	0.2016	27429.1
STD VX-5	200	800	0.1008	11341.5

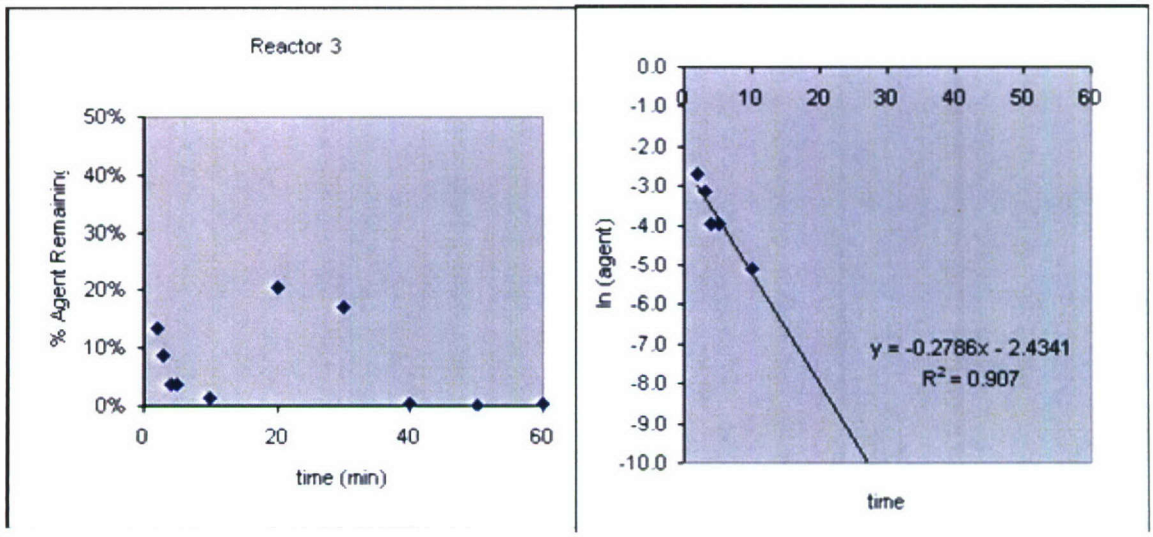
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.2367 min-1
	1	2	2	25552.4	0.1911	37.90%	-1.65491959	half life	2.93 min
	2	3	3	18542.4	0.1387	27.50%	-1.97559084		
	3	4	4	16877.5	0.1262	25.00%	-2.06966946		
	4	5	5	11952.3	0.0894	17.70%	-2.41472711		
	5	10	10	3776.6	0.0282	5.60%	-3.5668267		
	6	20	20	738.5	0.0055	1.10%			
	7	30	30	9460.1	0.0708	14.00%			
	8	40	40	10939.5	0.0818	16.20%			
	9	50	50	112.6	0.0008	0.20%			
	10	60	60	198.1	0.0015	0.30%			
	QCC-2 (0.2016mg/mL)			33015.8	0.2469	122.50%			

Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.2239 min-1
	Start	11						half life	3.1 min
	1	13	2	5336.3	0.0399	7.90%	-3.22111831		
	2	14	3	4014.6	0.03	6.00%	-3.50571312		
	3	15	4	2425	0.0181	3.60%	-4.00981931		
	4	16	5	2220.9	0.0166	3.30%	-4.09773832		
	5	21	10	833.1	0.0062	1.20%	-5.07825243		
	6	31	20	13218.4	0.0989	19.60%			
	7	41	30	137.7	0.001	0.20%			
	8	51	40	195.9	0.0015	0.30%			
	9	61	50	107.9	0.0008	0.20%			
	10	71	60	0	0	0.00%			
	QCC-3 (0.3024mg/ml)			44811.6	0.3351	110.80%			

Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Carbon)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.2786 min-1
	Start	22						half life	2.49 min
	1	24	2	8934.7	0.0668	13.30%	-2.70570827		
	2	25	3	5798.4	0.0434	8.60%	-3.13806882		
	3	26	4	2557.8	0.0191	3.80%	-3.95650332		
	4	27	5	2503.3	0.0187	3.70%	-3.97804098		
	5	32	10	833.9	0.0062	1.20%	-5.07729263		
	6	42	20	13902.3	0.104	20.60%			
	7	52	30	11540.6	0.0863	17.10%			
	8	62	40	173.4	0.0013	0.30%			
	9	72	50	86.9	0.0006	0.10%			
	10	82	60	192.4	0.0014	0.30%			
	QCC-4 (0.4032mg/mL)			54646	0.4087	101.40%			


Reactor 1
% Agent Remaining
50%
40%
30%
20%
10%
0%
0
20
40
60
time (min)
ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.2367x - 1.2001
R² = 0.9947
time


Reactor 2
% Agent Remaining
50%
40%
30%
20%
10%
0%
0
20
40
60
time (min)
ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.2239x - 2.9076
R² = 0.9625


Reactor 3
% Agent Remaining
50%
40%
30%
20%
10%
0%
0
20
40
60
time (min)
ln (agent)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.2786x - 2.4341
R² = 0.907
time

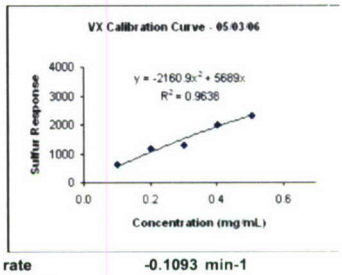
Appendix A17: Sulfur Channel GC-AED Data for DF200-VX Samples

Run 050306

VX vs. DF200 @ 25 deg C

Reference: d= 1.0222g/mL
Initial agent concentration: 2% v/v
ppm
Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform
GC: GC-AED, method VX, monitoring Sulfur 181
Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

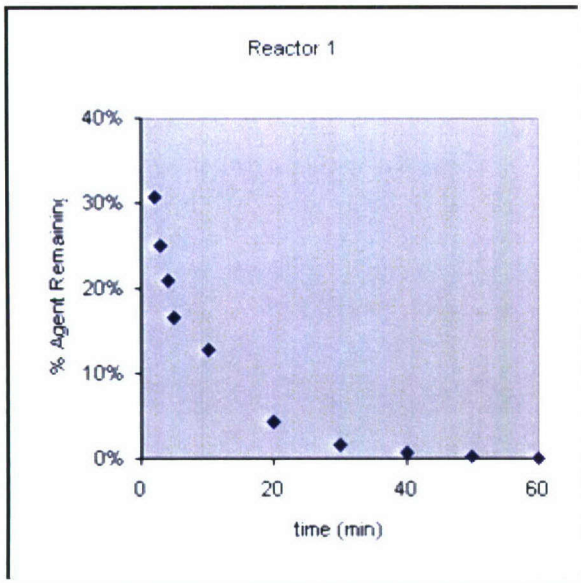
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Sulfur)
STD VX-1	1000	0	0.504	2324
STD VX-2	800	200	0.4032	2018.6
STD VX-3	600	400	0.3024	1312.5
STD VX-4	400	600	0.2016	1176.8
STD VX-5	200	800	0.1008	614.2

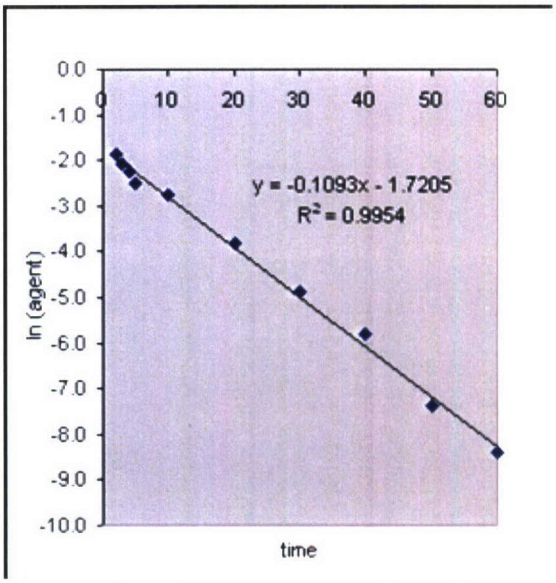


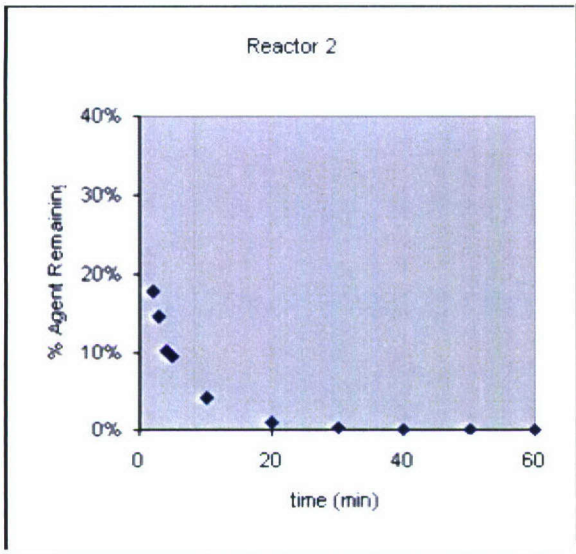
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Sulfur)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.1093 min-1
	1	2	2	826.6	0.1543	30.60%	-1.86855322	half life	6.34 min
	2	3	3	679.8	0.1255	24.90%	-2.07565822		
	3	4	4	575.3	0.1053	20.90%	-2.25056351		
	4	5	5	458.9	0.0833	16.50%	-2.4853055		
	5	10	10	358.7	0.0646	12.80%	-2.73894476		
	6	20	20	122.9	0.0218	4.30%	-3.82661018		
	7	30	30	43.5	0.0077	1.50%	-4.87061172		
	8	40	40	17.2	0.003	0.60%	-5.80023		
	9	50	50	3.5	0.0006	0.10%	-7.39329303		
	10	60	60	1.3	0.0002	0.00%	-8.38383869		
QCC-2 (0.2016mg/mL)				1366.1	0.2673	132.40%			

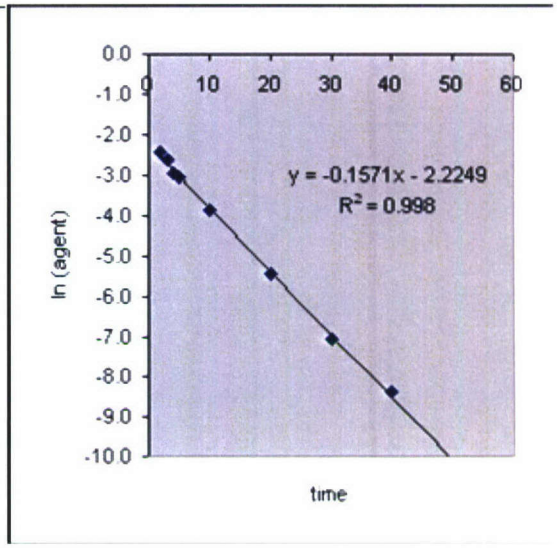
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Sulfur)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.1571 min-1
	Start	11						half life	4.41 min
	1	13	2	491.4	0.0894	17.70%	-2.41447832		
	2	14	3	408.5	0.0739	14.70%	-2.60533475		
	3	15	4	289.6	0.0519	10.30%	-2.95786714		
	4	16	5	268.9	0.0481	9.60%	-3.03349276		
	5	21	10	121.2	0.0215	4.30%	-3.84065548		
	6	31	20	25.2	0.0044	0.90%	-5.41775898		
	7	41	30	4.9	0.0009	0.20%	-7.05672724		
	8	51	40	1.3	0.0002	0.00%	-8.38383869		
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
QCC-3 (0.3024mg/mL)				1539.7	0.3063	101.28%			

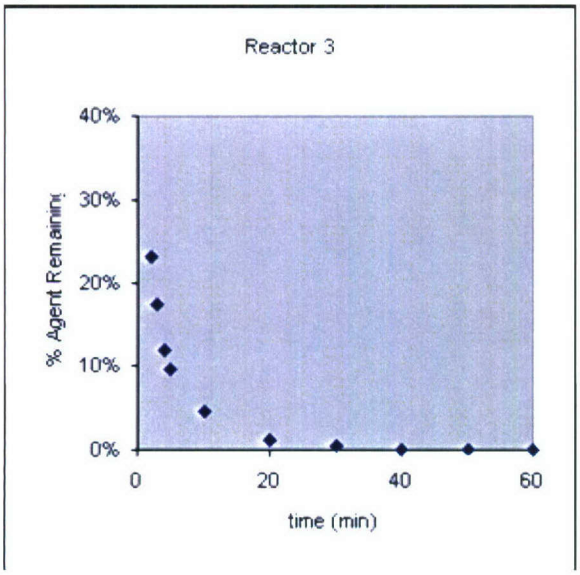
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Sulfur)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.146 min-1
	Start	22						half life	4.75 min
	1	24	2	634.8	0.1168	23.20%	-2.14761542		
	2	25	3	483.3	0.0879	17.40%	-2.43169927		
	3	26	4	335.6	0.0604	12.00%	-2.80716976		
	4	27	5	271.3	0.0486	9.60%	-3.02443764		
	5	32	10	130.9	0.0232	4.60%	-3.76299942		
	6	42	20	33.9	0.006	1.20%	-5.12060362		
	7	52	30	10.2	0.0018	0.40%	-6.32322032		
	8	62	40	1.9	0.0003	0.10%	-8.004309		
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
QCC-4 (0.4032mg/mL)				1938.5	0.4022	99.70%			

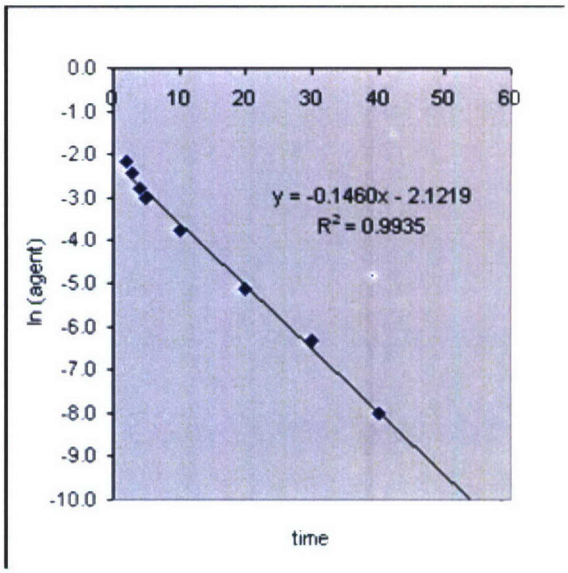

Reactor 1
% Agent Remaining
40%
30%
20%
10%
0%
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.1093x - 1.7205
R² = 0.9954
ln (agent)
time


Reactor 2
% Agent Remaining
40%
30%
20%
10%
0%
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.1571x - 2.2249
R² = 0.998
ln (agent)
time


Reactor 3
% Agent Remaining
40%
30%
20%
10%
0%
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
y = -0.1460x - 2.1219
R² = 0.9935
ln (agent)
time

Appendix A18: Phosphorus Channel GC-AED Data for DF200-VX Sample

Run 050306

VX vs. DF200 @ 25 deg C

Reference: d= 1.0222g/mL

Initial agent concentration: 2% v/v

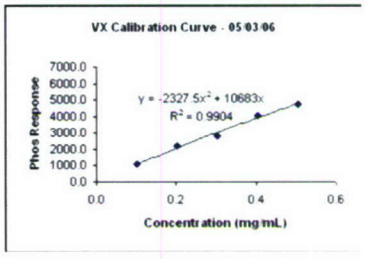
ppm

Extraction: 50 µL sample mixture into 1.0 mL 0.2 M Sodium Sulfite and 2.0 mL chloroform

GC: GC-AED, method VX, monitoring Phosphorus 178

Standards: Stock solution: 5.0 µL agent into 10.0 mL chloroform

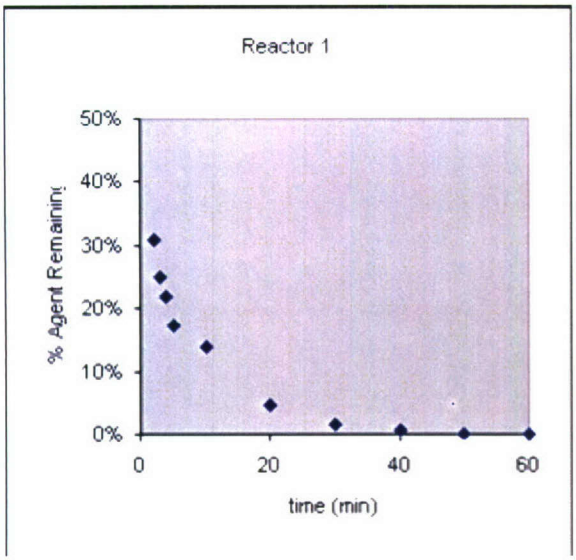
Solution	µL stock	µL CHCl3	µg agent per injection	Area Counts (Phos)
STD VX-1	1000	0	0.504	4751
STD VX-2	800	200	0.4032	4087.8
STD VX-3	600	400	0.3024	2803.5
STD VX-4	400	600	0.2016	2164.9
STD VX-5	200	800	0.1008	1056.4

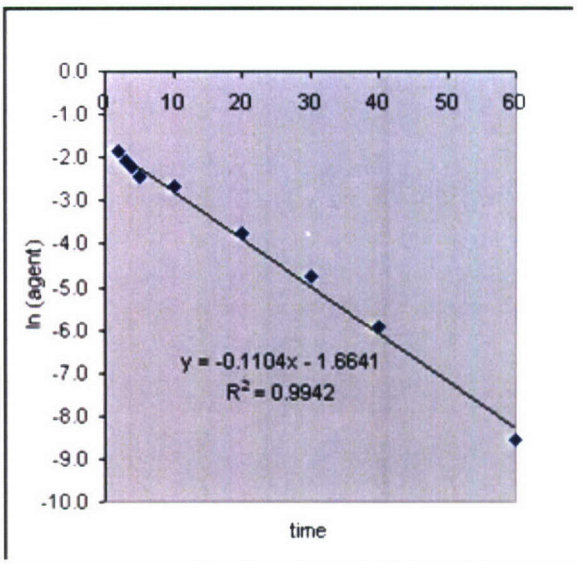


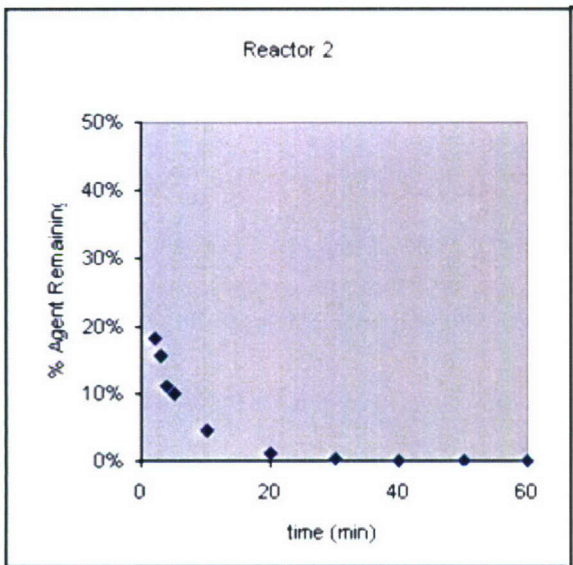
Reactor 1	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.1104 min-1
	1	2	2	1612.9	0.1563	31.00%	-1.85597341	half life	6.28 min
	2	3	3	1312.8	0.1264	25.10%	-2.06857408		
	3	4	4	1150.3	0.1103	21.90%	-2.20430027		
	4	5	5	906.4	0.0865	17.20%	-2.44790842		
	5	10	10	742.8	0.0706	14.00%	-2.65047716		
	6	20	20	254	0.0239	4.70%	-3.7338539		
	7	30	30	90.4	0.0085	1.70%	-4.77031596		
	8	40	40	28.8	0.0027	0.50%	-5.91544572		
	9	50	50	0	0	0.00%			
	10	60	60	2.1	0.0002	0.00%	-8.5344288		
QCC-2 (0.2016mg/mL)				2488.9	0.2462	122.10%			

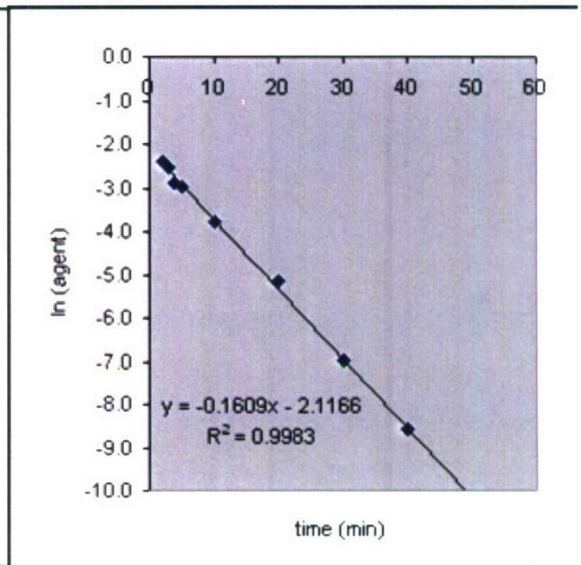
Reactor 2	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.1609 min-1
	Start	11						half life	4.31 min
	1	13	2	962.9	0.092	18.20%	-2.38621692		
	2	14	3	829.8	0.079	15.70%	-2.5378548		
	3	15	4	596.1	0.0565	11.20%	-2.87361578		
	4	16	5	535	0.0506	10.00%	-2.98304839		
	5	21	10	246.5	0.0232	4.60%	-3.76398151		
	6	31	20	61.9	0.0058	1.20%	-5.149624		
	7	41	30	9.8	0.0009	0.20%	-6.99382666		
	8	51	40	2	0.0002	0.00%	-8.583221		
	9	61	50	0	0	0.00%			
	10	71	60	0	0	0.00%			
QCC-3 (0.3024mg/mL)				3010.6	0.3016	99.70%			

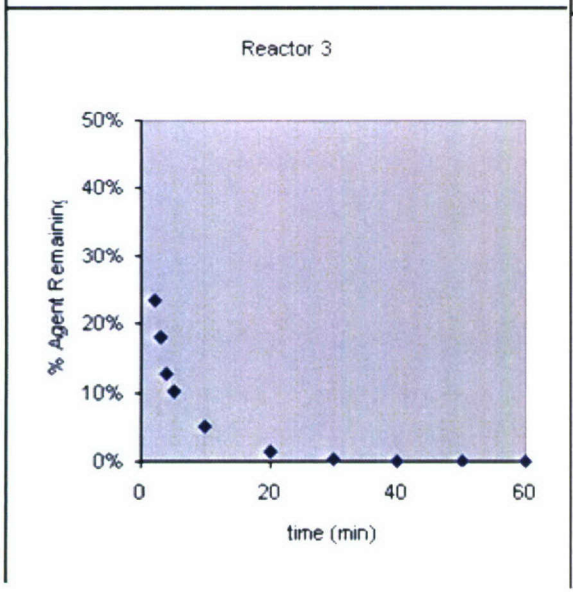
Reactor 3	Sample	Time (min)	Corr.Time (min)	Area (Phos)	[Agent] µg	% agent remaining	ln (agent)		
								rate	-0.1403 min-1
	Start	22						half life	4.94 min
	1	24	2	1232.6	0.1184	23.50%	-2.13338567		
	2	25	3	953.9	0.0911	18.10%	-2.39580271		
	3	26	4	677.6	0.0643	12.80%	-2.74373725		
	4	27	5	545.8	0.0517	10.30%	-2.96283471		
	5	32	10	274.2	0.0258	5.10%	-3.65691166		
	6	42	20	80.4	0.0075	1.50%	-4.88775107		
	7	52	30	16.8	0.0016	0.30%	-6.45468729		
	8	62	40	5.5	0.0005	0.10%	-7.57154869		
	9	72	50	0	0	0.00%			
	10	82	60	0	0	0.00%			
QCC-4 (0.4032mg/mL)				3893.4	0.3992	99.00%			

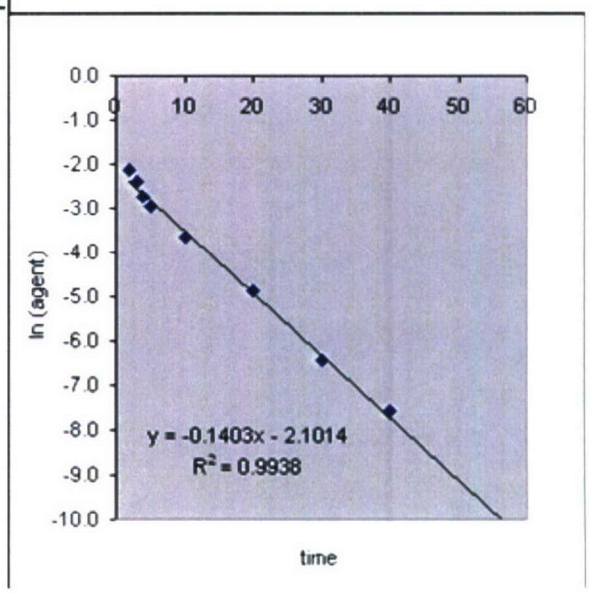

Reactor 1
% Agent Remainin
50%
40%
30%
20%
10%
0%
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
ln (agent)
y = -0.1104x - 1.6641
R² = 0.9942
time


Reactor 2
% Agent Remainin
50%
40%
30%
20%
10%
0%
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
ln (agent)
y = -0.1609x - 2.1166
R² = 0.9983
time (min)


Reactor 3
% Agent Remainin
50%
40%
30%
20%
10%
0%
0
20
40
60
time (min)


0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
-7.0
-8.0
-9.0
-10.0
0
10
20
30
40
50
60
ln (agent)
y = -0.1403x - 2.1014
R² = 0.9938
time

APPENDIX B: NMR SPECTRA OF CDS AND DF200 REACTIONS WITH CHEMICAL AGENTS

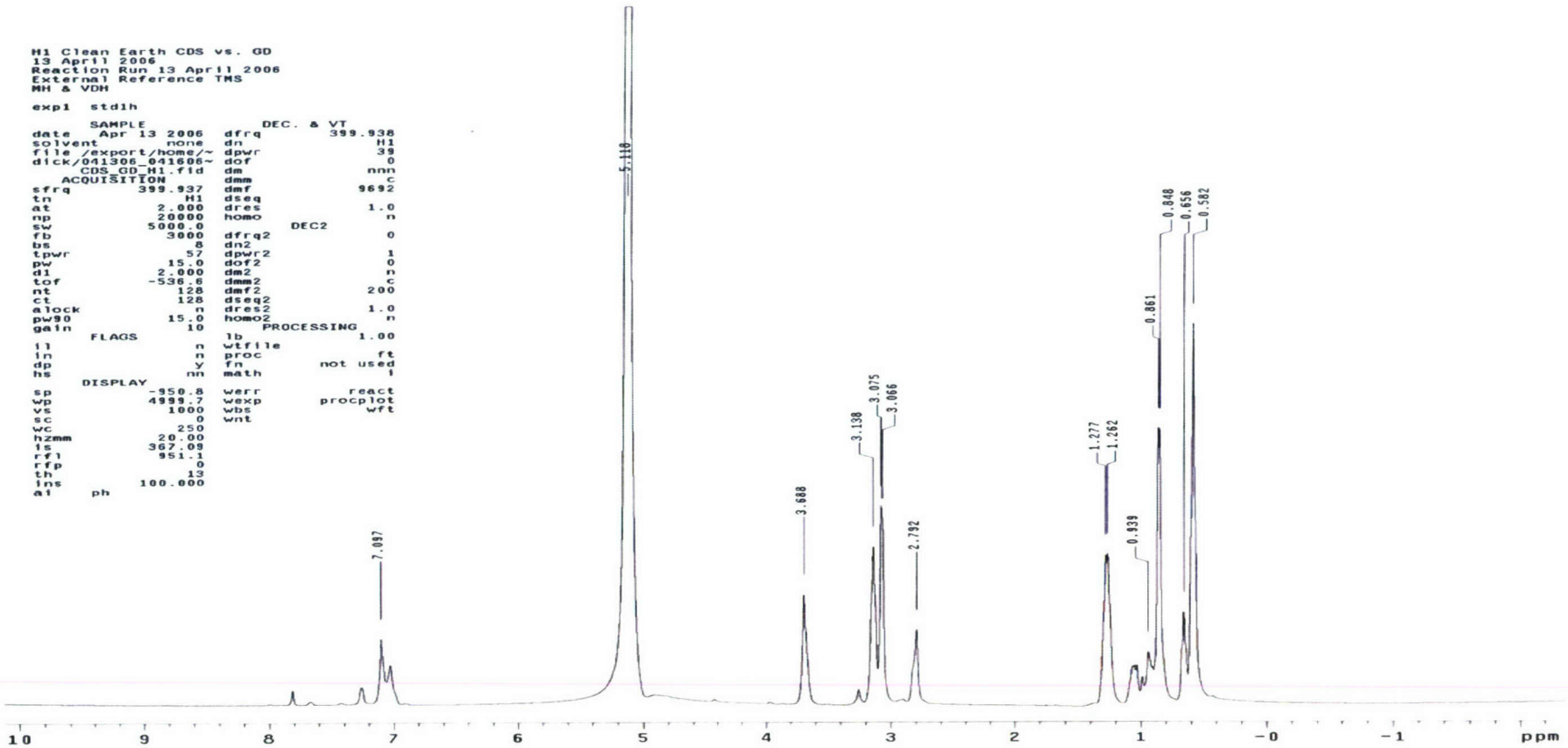


Figure B1: ^{1}H-NMR Spectrum of CDS-GD Reaction Mixture

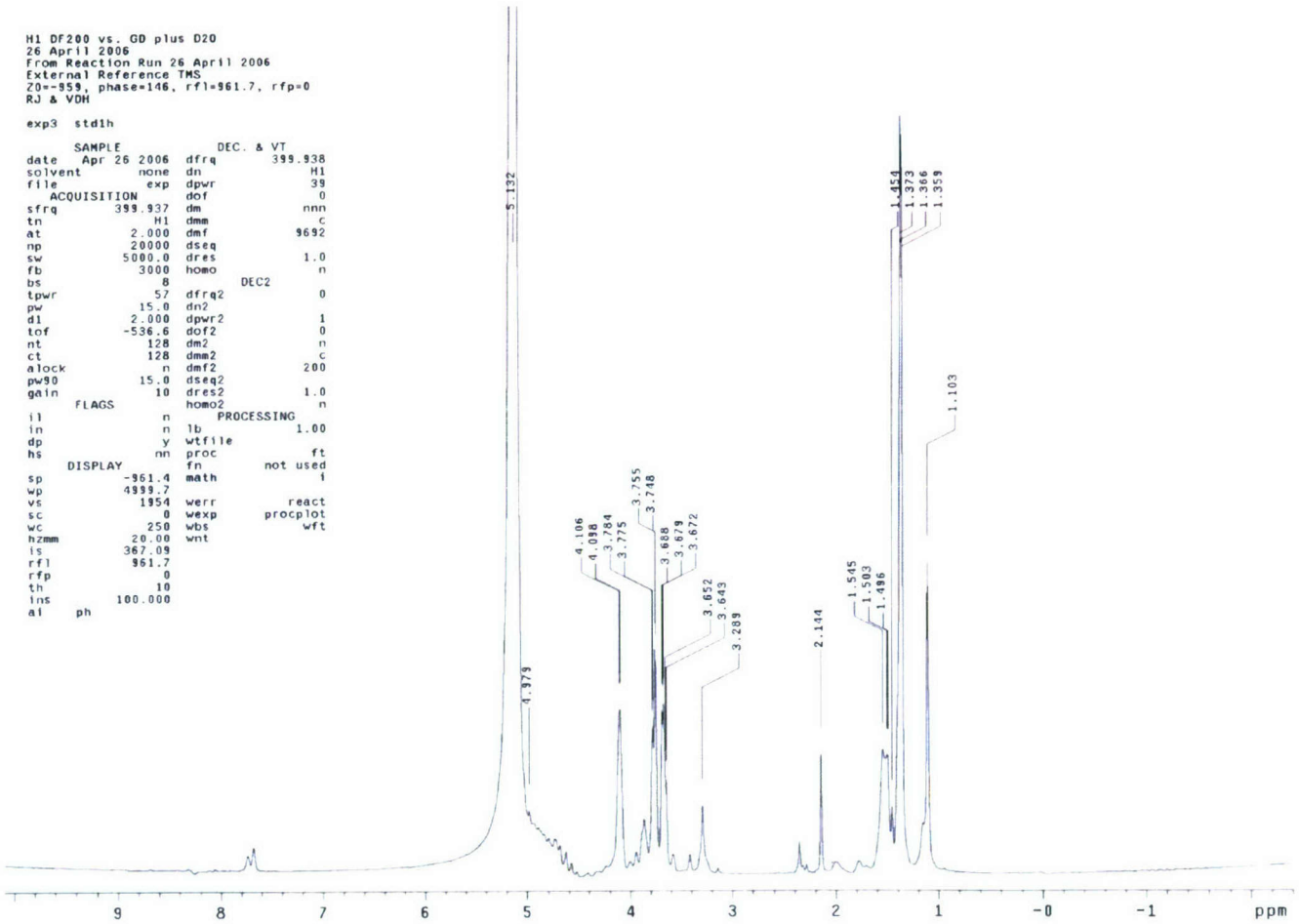


Figure B2: ^{1}H-NMR Spectrum of DF200 -GD Reaction Mixture

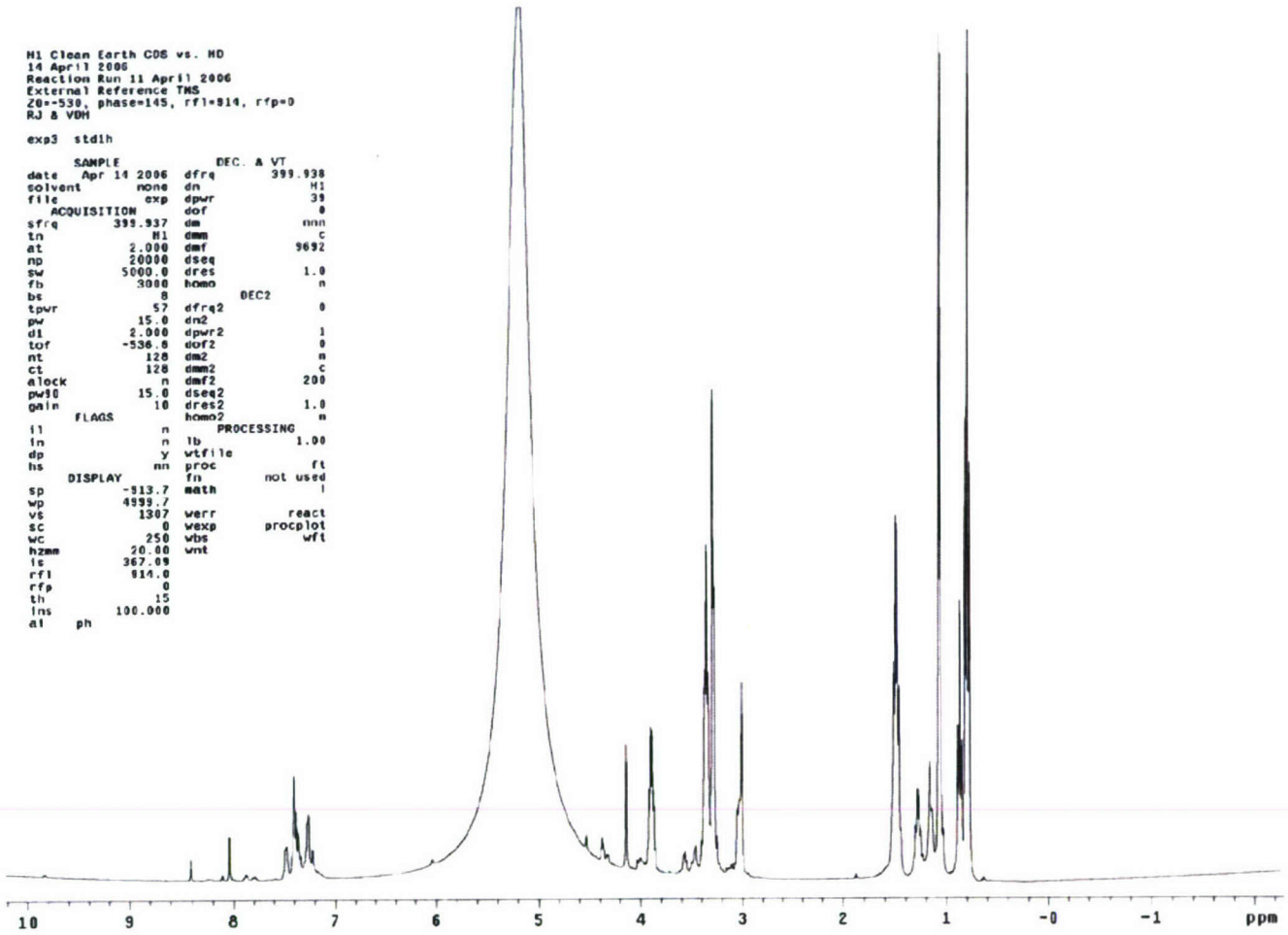


Figure B3: ^{1}H-NMR Spectrum of CDS -HD Reaction Mixture

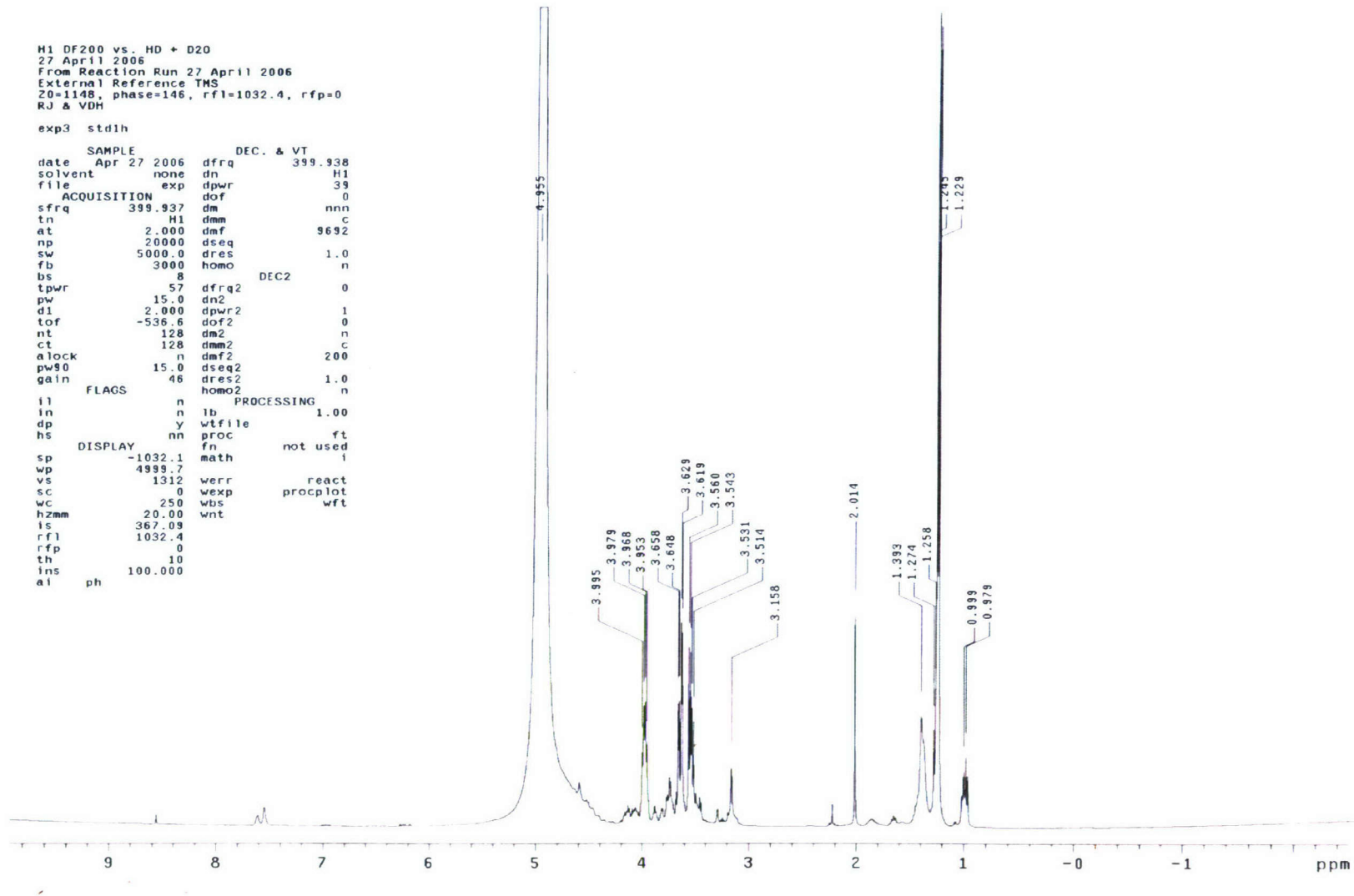


Figure B4: ^{1}H-NMR Spectrum of DF200-HD Reaction Mixture

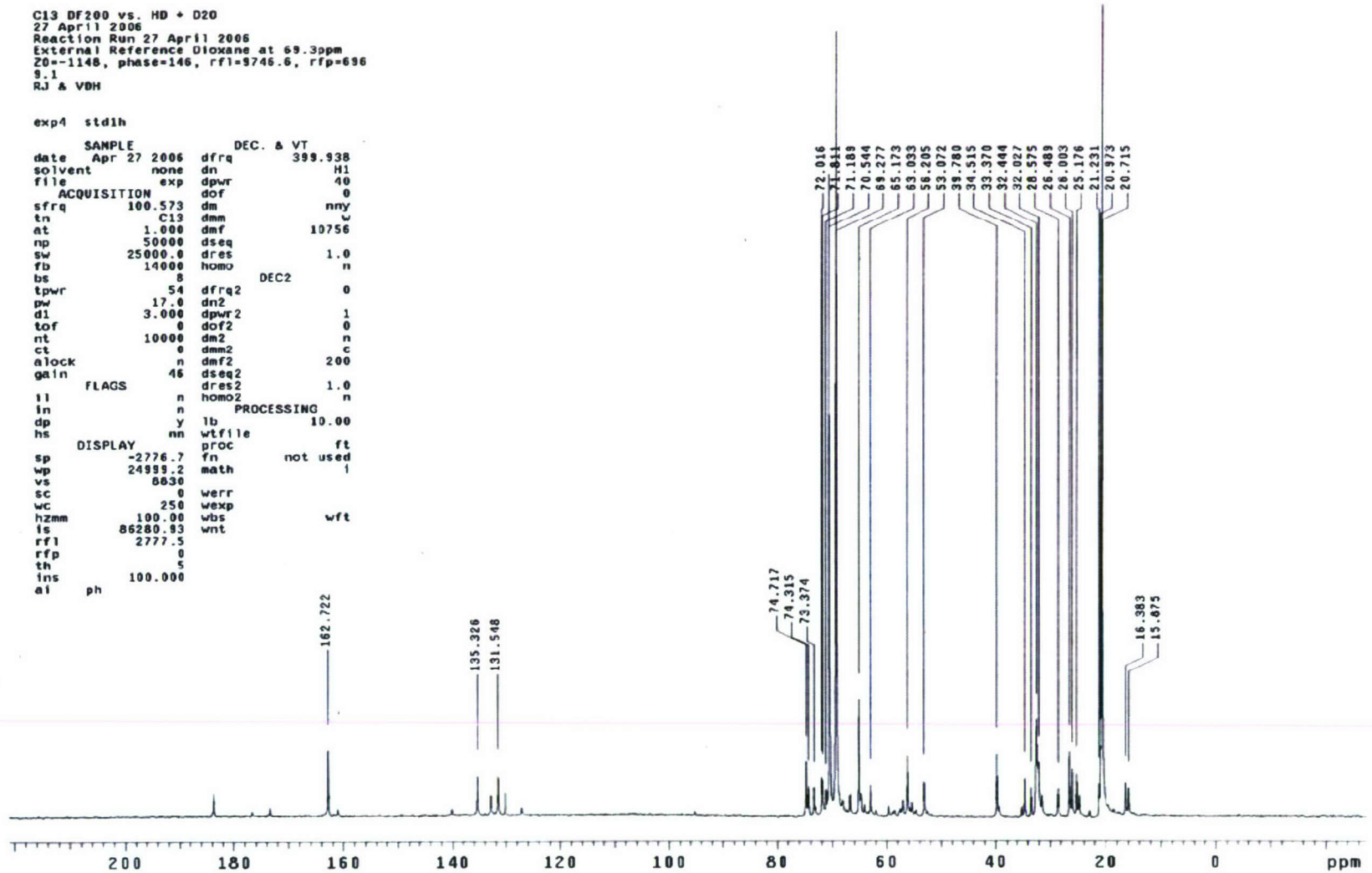


Figure B5: ^{13}C-NMR Spectrum of DF200-HD Reaction Mixture

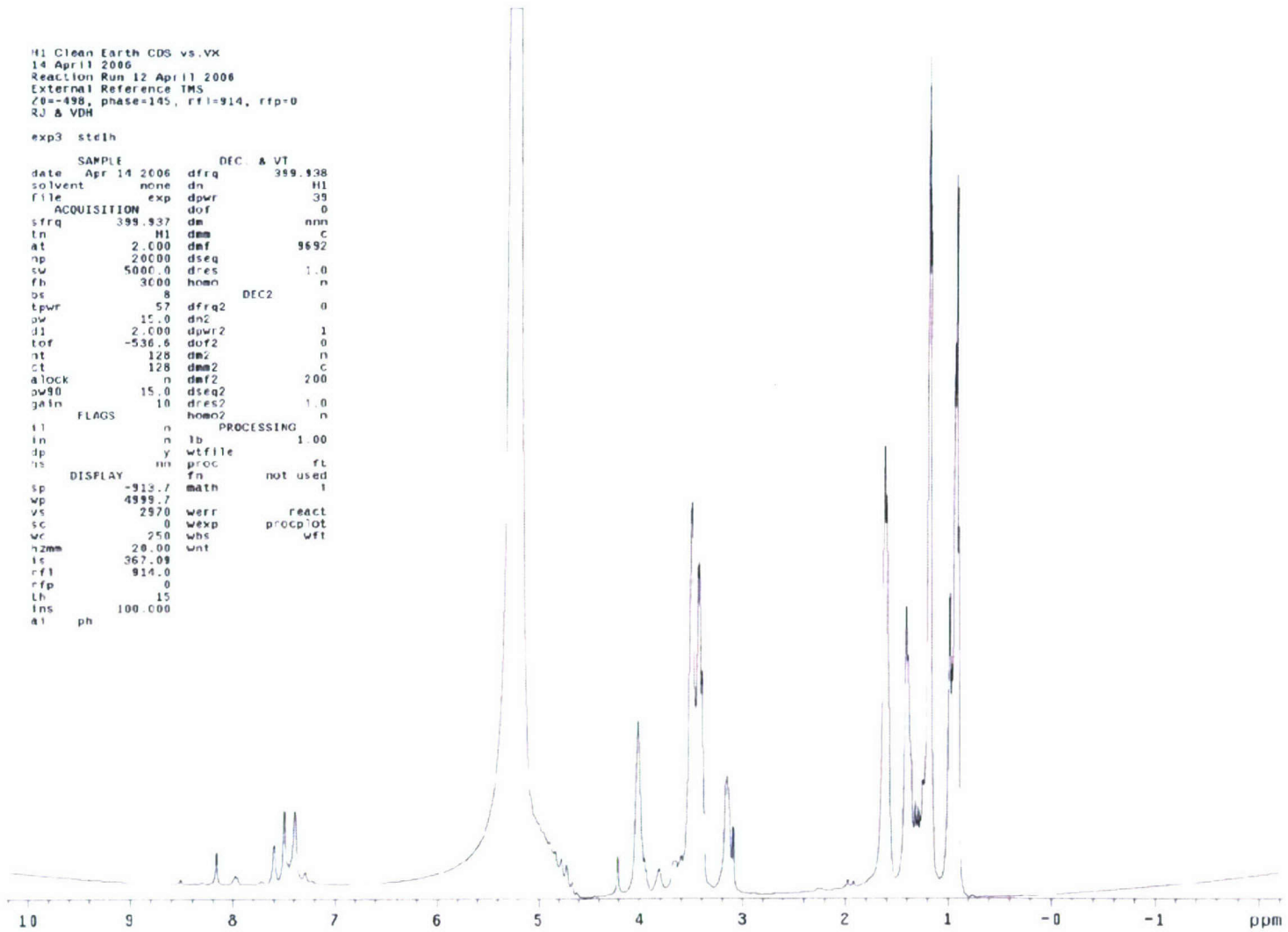


Figure B6: ^{1}H-NMR Spectrum of CDS-VX Reaction Mixture

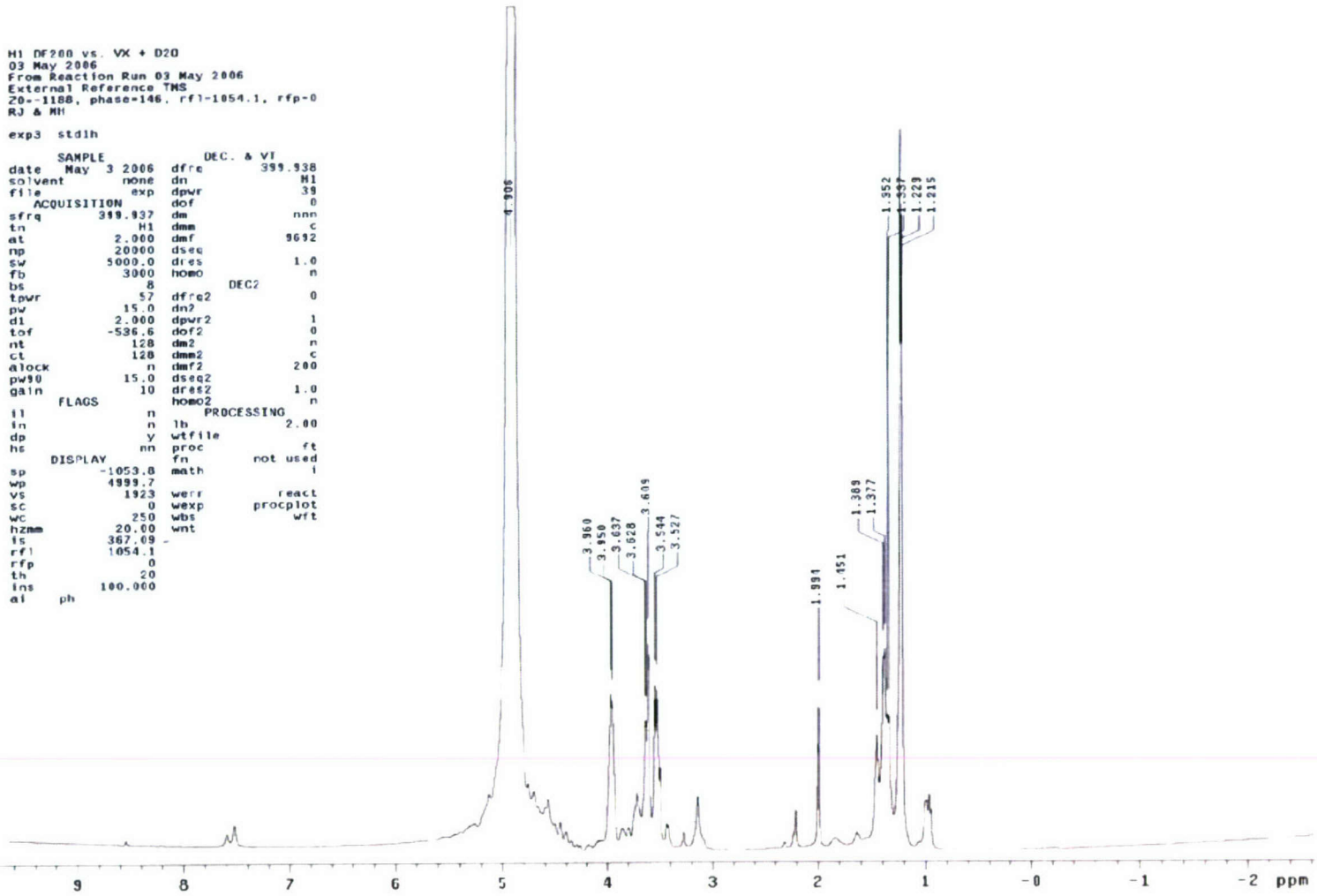


Figure B7: ^{1}H-NMR Spectrum of DF200-VX Reaction Mixture