Energetic Potpourri III*

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*Part I: Gordon Research Conference on Energetic Materials, June 1996
*Part II: Fourth Primary Explosives Workshop, Chandler, AZ, February 2008

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Cyclic Dinitroureas as Self-Remediating Munitions Charges

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Environmental Issues with Unexploded Ordnance

- Hazards are presented by the submunition fills in weapons such as BLU-97/B bomblets if they end up as UXO due to dudding

- Similar problems are posed even by incomplete (low-order) detonation of larger munitions, such as Mark 80 series bombs and 155–165-mm projectiles

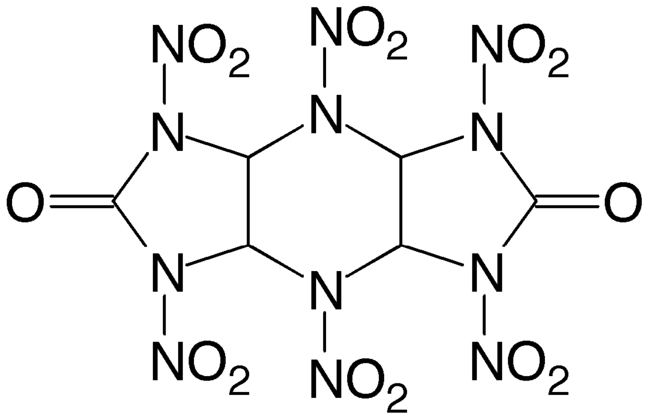
- RDX explosive charge (e.g., in BLU-97/B's PBXN-107 fill) exhibits deleterious environmental effects on water supplies, and its natural aerobic biodegradation is very slow

- Concepts were needed to alleviate such hazards of UXO

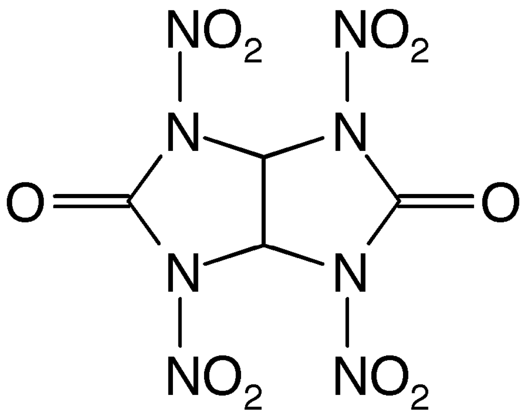
Proposal

- **Cyclic *N,N'*-dinitroureas** have attractive performance but higher hydrolytic reactivity than simple nitramines (RDX, HMX, CL-20)
- Such hydrolytic susceptibility could make them “self-remediating” when exposed to the environment
- Specific application: systems prone to UXO (e.g., BLU-97/B bomblets)

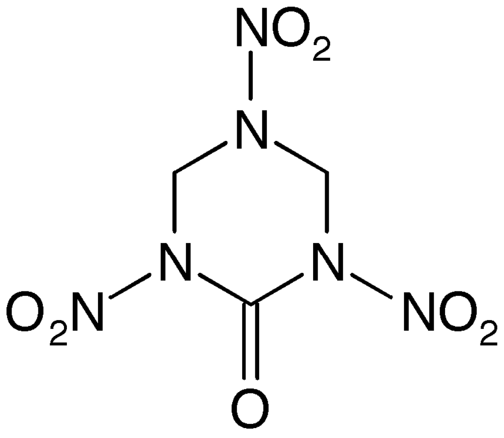
Cyclic Dinitroureas Considered



HHTDD



TNGU
(Sorguyl)



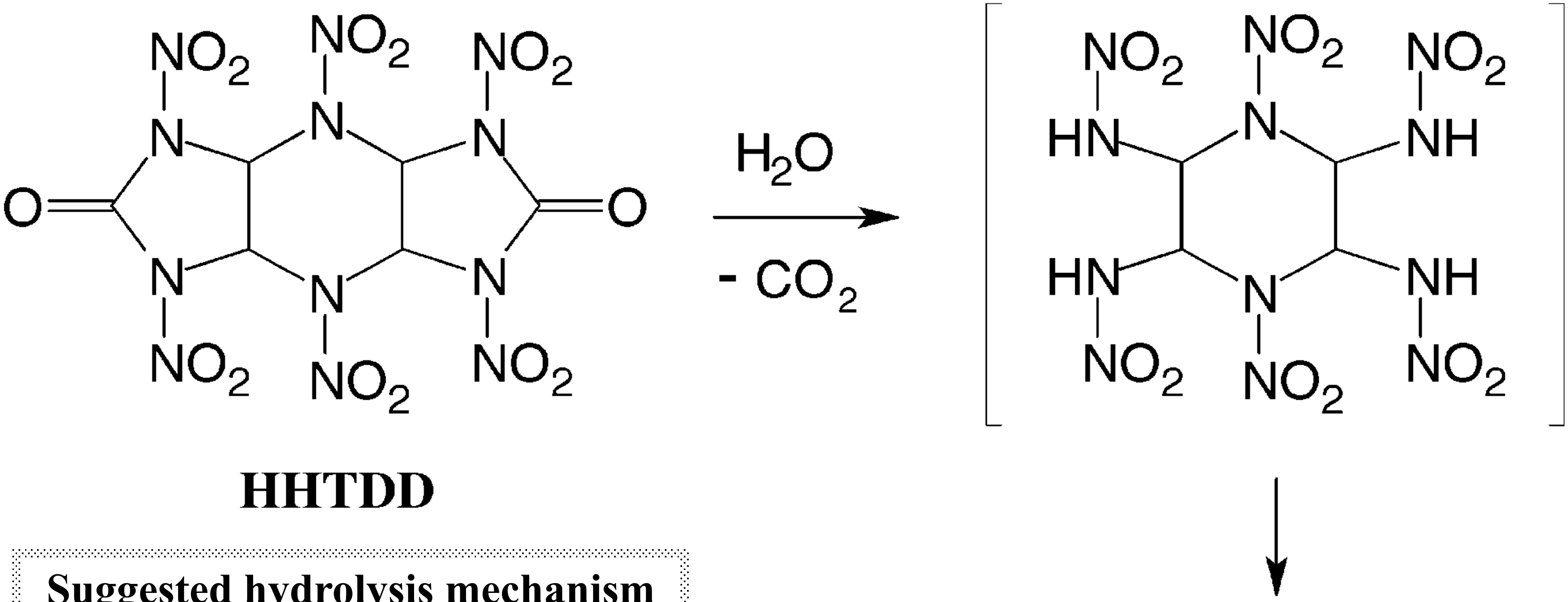
K-6
(keto-RDX)

- **Downselected to HHTDD and TNGU because of past development of K-6 at LLNL.**
- **The two selected dinitroureas have received little U.S. development but more overseas: TNGU in France and China, HHTDD in China and Russia.**

Performance/Properties of Cyclic Dinitroureas

	Density ρ (g/cm^3)	**Detonation pressure P_{CJ} (GPa)**	**Detonation velocity D (km/sec)**	**m.p./ dec. (° C)**
RDX	1.81	33.8	8.74	204
HMX	1.90	39.0	9.11	~277
CL-20	2.04	~46	~9.7	~253
TNGU	1.98–2.01	38.7	~9.20	~200
HHTDD	2.07	46.2	~9.75	215

Cyclic Dinitrourea Hydrolysis (Laboratory Conditions)



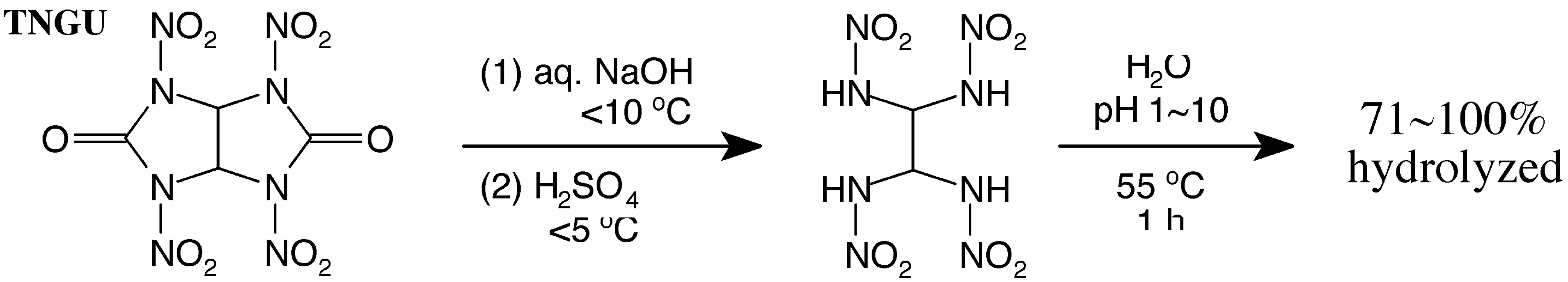
HHTDD

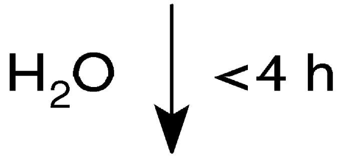
$2\ (CHO)_2 + 6\ N_2O + 2\ H_2O$

Suggested hydrolysis mechanism
[**Ref**: Hu, R.; Lu, X.; Fang, Y.
J. Energ. Mater. **1993**, *11*, 219]

biodegradable, environmentally benign products
[Hazardous Substances Data Bank (http://toxnet.nlm.nih.gov/cgi-bin/sis/htmlgen?HSDB)]

Cyclic Dinitrourea Hydrolysis (Laboratory Conditions)





"completely hydrolyzed"

Ref: Peng, Z.; Wan, D. *Binggong Xuebao* **1980**, *3*(3), 23

Ref: Sikder, A.K. et al. *Propellants Explosives Pyrotechnics* **2001**, *26*, 63

Determine Environmental Stabilities

- Hydrolysis studies in the literature have been under harsh alkaline conditions

- Now measure hydrolysis under “mild” environmental conditions

- Measure degradation rates in dry air, in humid air, in dry soil, and in moist soil

- Determine alternative conditions, if necessary, that make these ingredients “self-remediating”

Environmental Hydrolysis of Cyclic Dinitroureas

Methods:

- **“Dry air” energetic samples contained 14 mg ingredient in open vials (covered by lab wipes) in ambient laboratory air (23 ± 2 °C, 27.6 ± 2.8% RH). Acetonitrile-d_3 (700 µL) was added just prior to analysis.**
- **“Humid air” energetic samples stood in vials in a closed jar containing saturated aqueous potassium chloride solution (85% RH). Acetonitrile-d_3 (700 µL) was added just prior to analysis.**
- **“Dry soil” samples were prepared by mixing 400 mg soil + 40 mg energetic ingredient with a spatula. Soil is Horizon A topsoil (Ward Science), air-dried, sieved with a 0.495-mm screen; pH of 1:1 aqueous suspension ≈ 6. Vials stood open in ambient laboratory air until extraction and analysis.**
- **“Moist soil” samples were prepared by mixing 1 g soil, 0.1 g energetic ingredient, and 0.25 mL D_2O.**

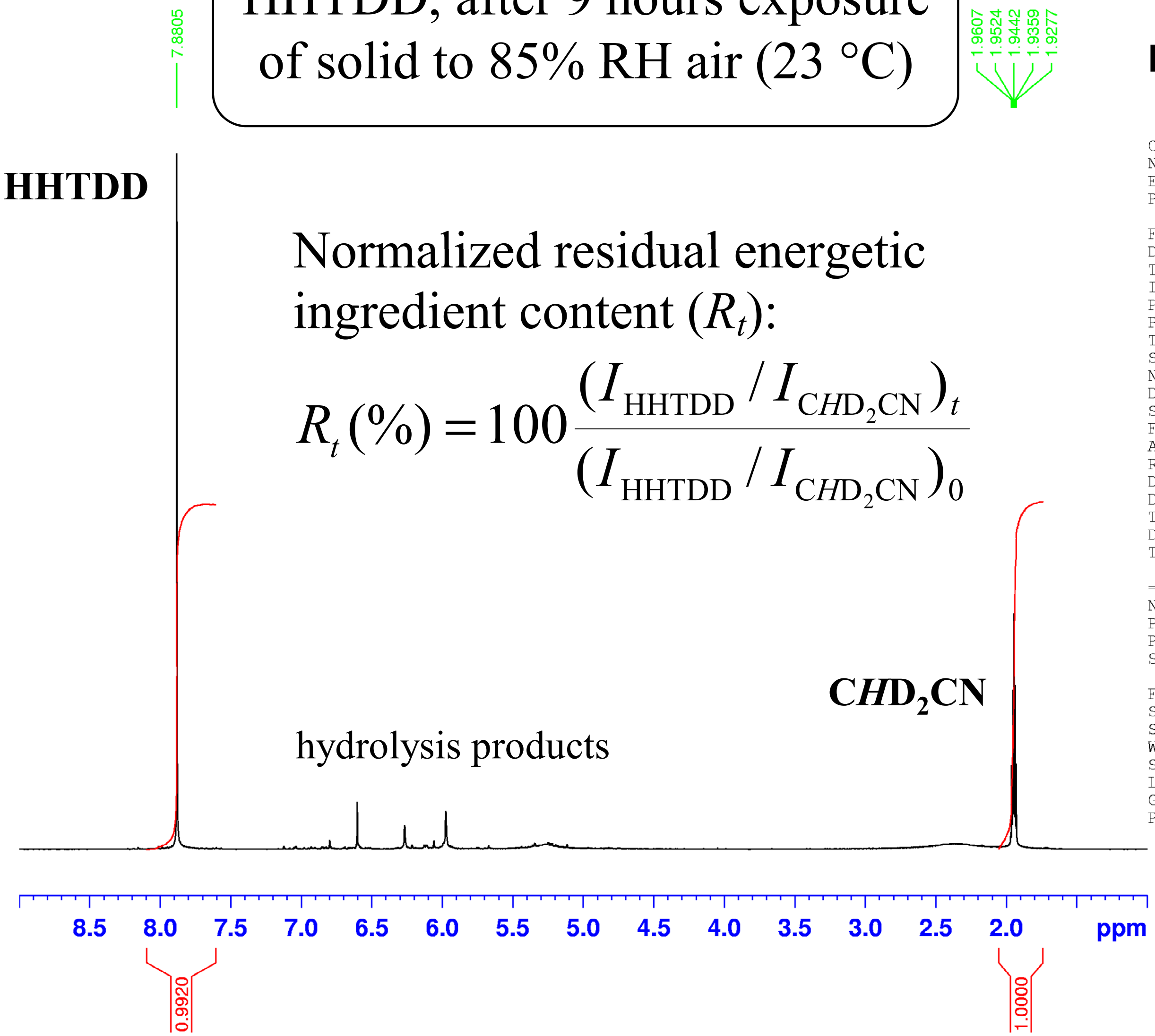
Environmental Hydrolysis of Cyclic Dinitroureas

Methods (cont.):

- **Energetic ingredients were extracted from soil by adding 2.0 mL acetonitrile-d_3, sonicating for 5 minutes, centrifuging, and using supernatant solution for NMR analysis**
- **Quantification of residual energetic ingredient was performed by ^1H NMR, integrating the ingredient's peak vs. residual solvent protons (CHD_2CN) as internal standard. For each series, the same lot of CD_3CN was used. Triplicate spectra were taken of each sample to obtain an average measurement.**

Sample ^{1}H NMR Spectrum (HHTDD)

HHTDD, after 9 hours exposure of solid to 85% RH air (23 °C)



Normalized residual energetic ingredient content (R_t):

$$R_t(\%) = 100\frac{(I_{\mathrm{HHTDD}} / I_{CHD_2\mathrm{CN}})_t}{(I_{\mathrm{HHTDD}} / I_{CHD_2\mathrm{CN}})_0}$$

BRUKER

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Current Data Parameters
NAME              rq t85t1
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20081023
Time                 17.25
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CD3CN
NS                       8
DS                       0
SWH               6188.119 Hz
FIDRES            0.094423 Hz
AQ               5.2953587 sec
RG                     203
DW                  80.800 usec
DE                    6.00 usec
TE                   298.5 K
D1              8.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.05 usec
PL1                   3.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
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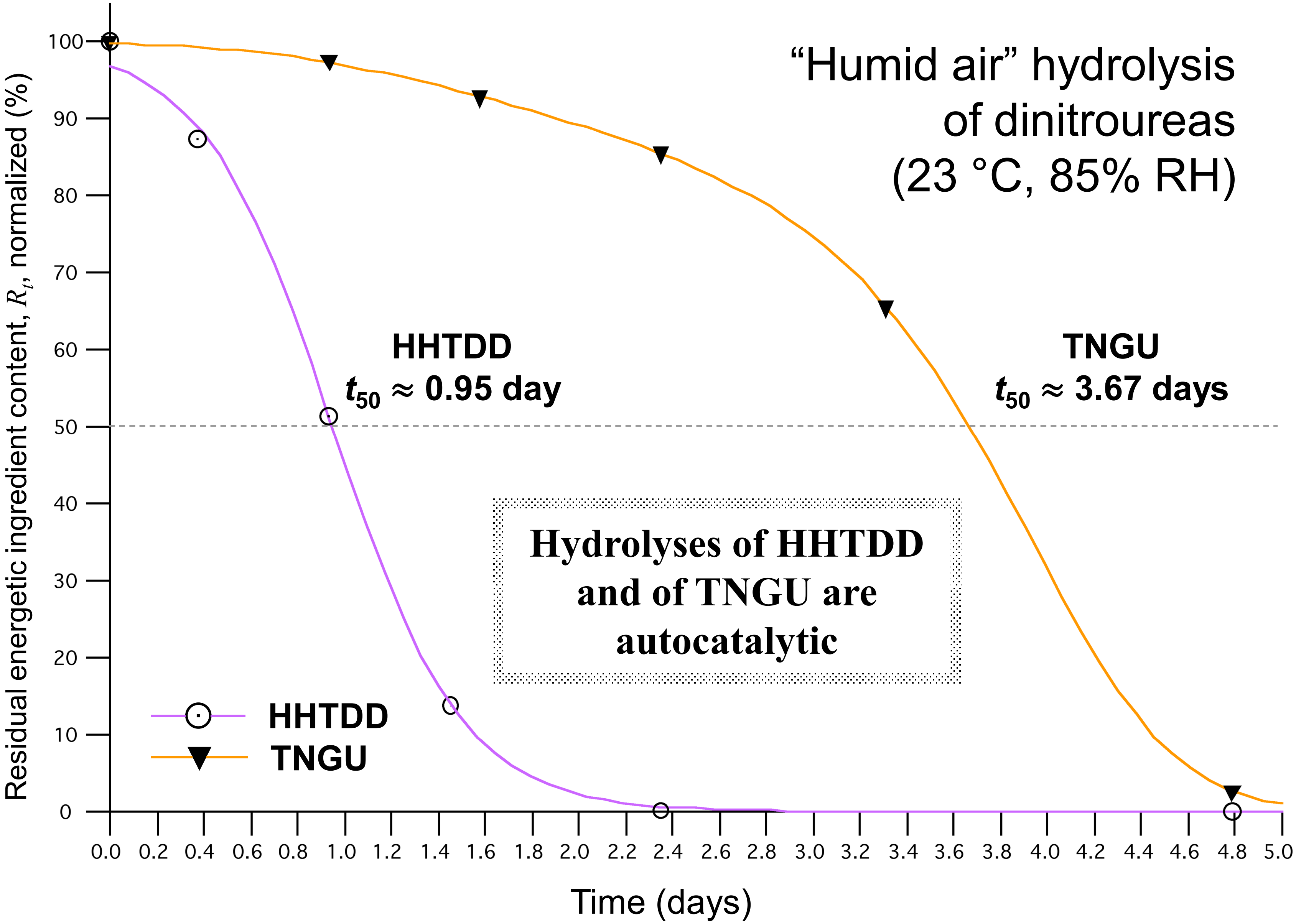
Degradation of HHTDD and TNGU in Humid Air (85% RH)



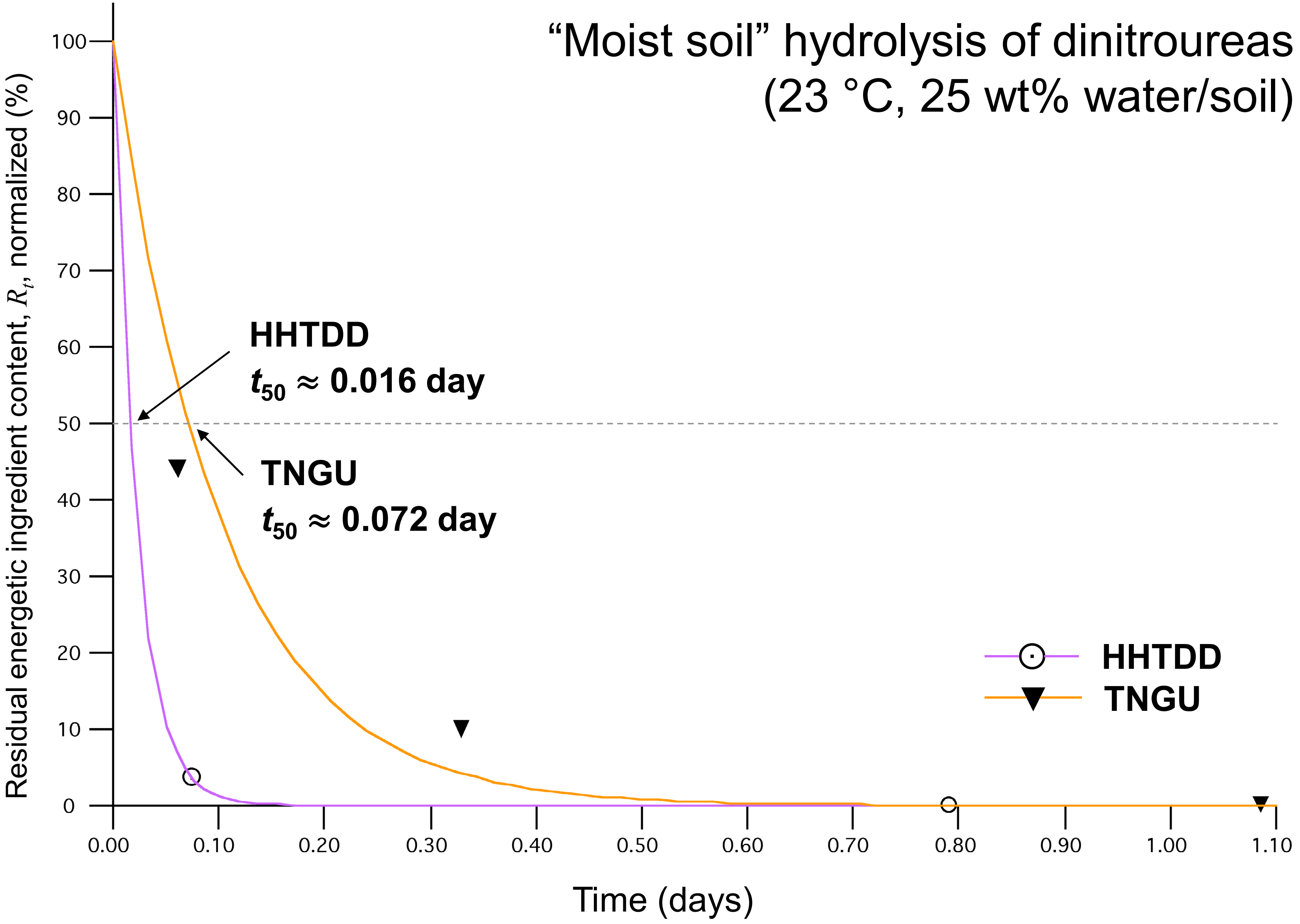
“Humid air” hydrolysis
of dinitroureas
(23 °C, 85% RH)

HHTDD
$t_{50} \approx 0.95$ day

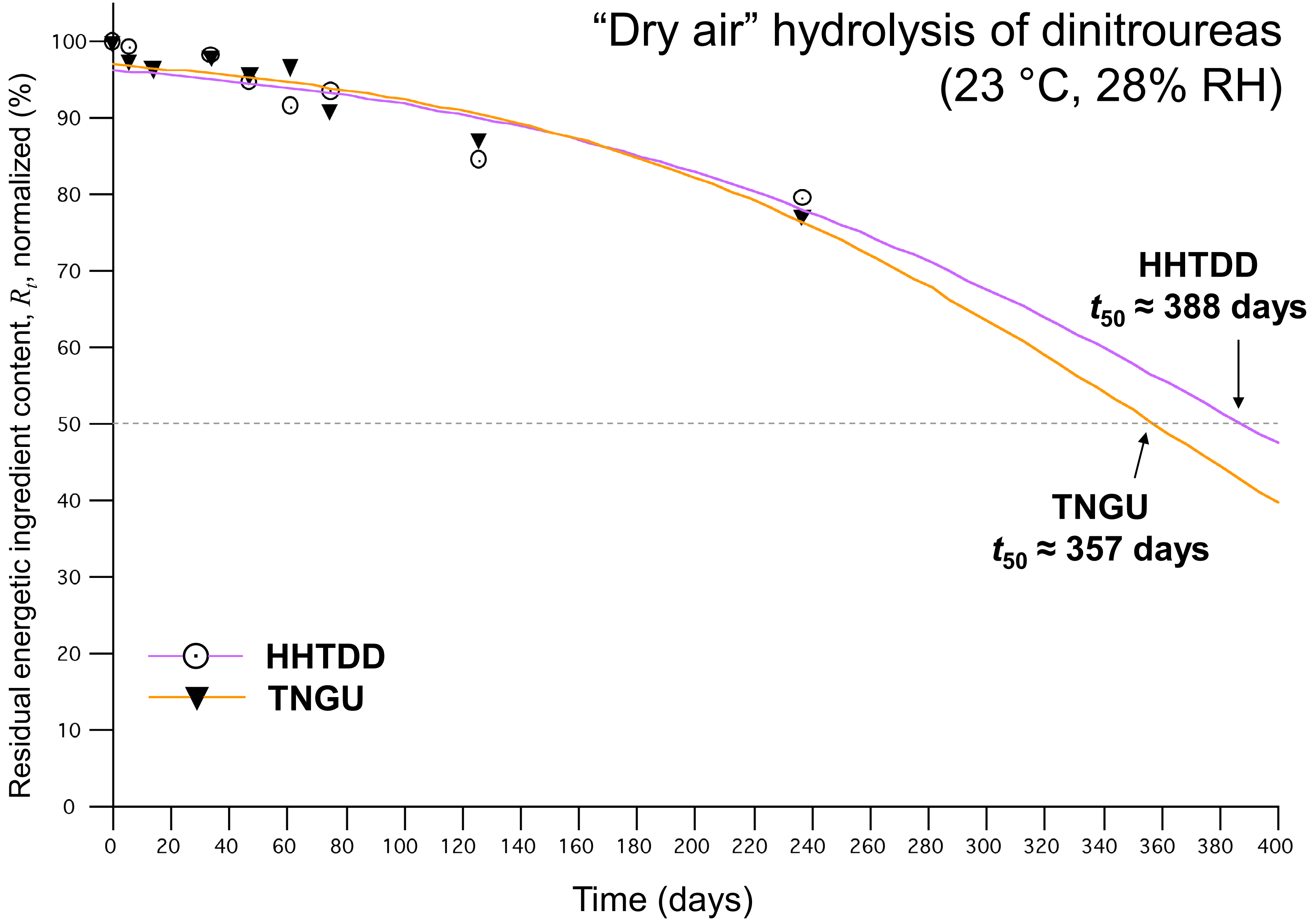
TNGU
$t_{50} \approx 3.67$ days

Hydrolyses of HHTDD and of TNGU are autocatalytic

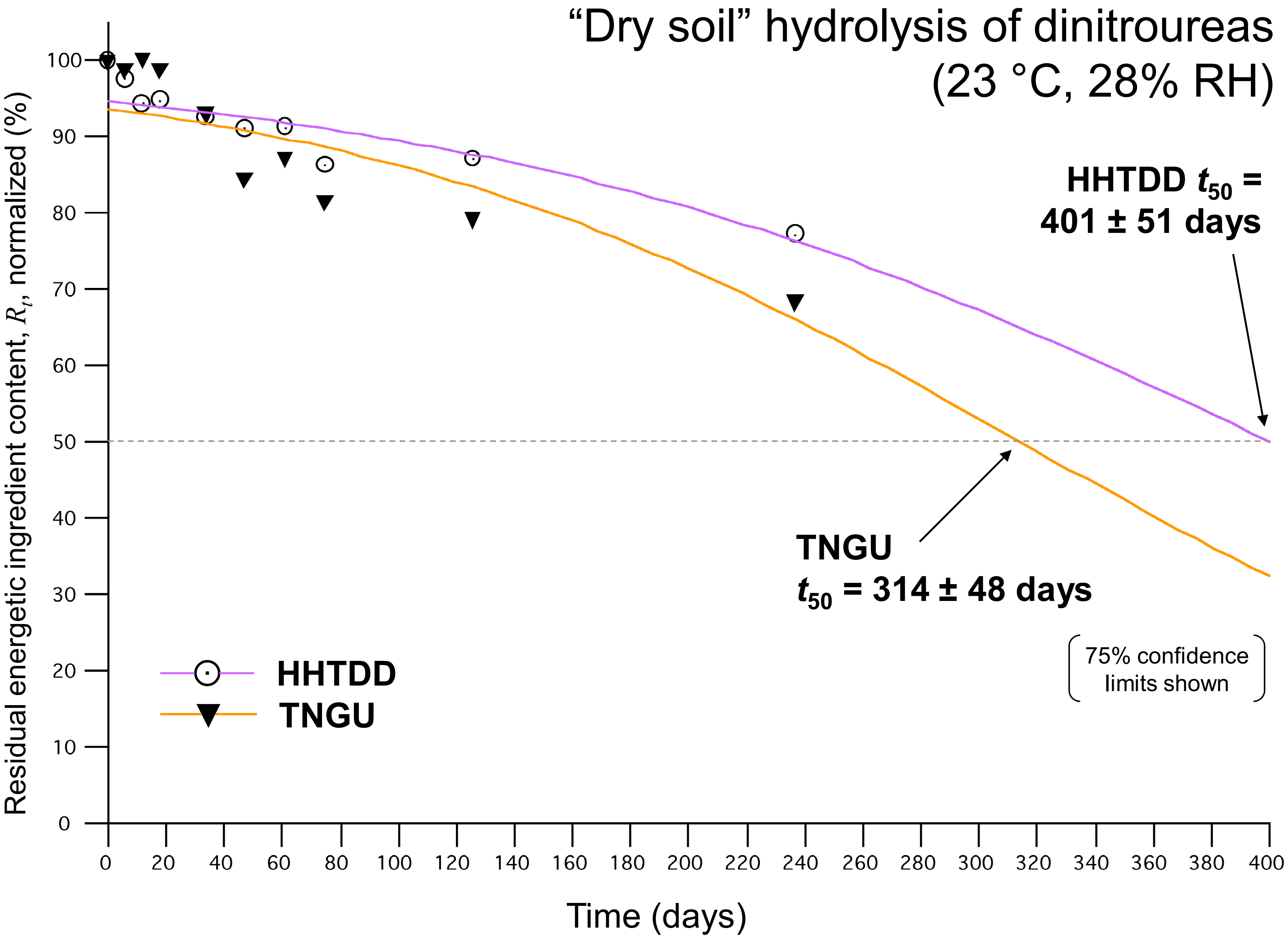
Degradation of HHTDD and TNGU in 1:4 Water:Soil



Degradation of HHTDD and TNGU in Dry Air (28% RH)



Degradation of HHTDD and TNGU in Contact with “Dry Soil”



Conclusions

- Cyclic dinitroureas TNGU and HHTDD have energetic performance comparable to HMX and CL-20, respectively, and are sufficiently stable under dry conditions to manufacture and process
- Adventitious environmental hydrolysis makes them self-remediating against long-term hazards
- Time frame of hydrolysis depends on humidity, ranging from a very few hours to months
- Rapid UXO self-remediation could be implemented by incorporating a source of water into munition design, if necessary, or external application of water

Follow-up WP-2147 Technical Approach

Task 1
Scale-up Energetic

Task 2
Develop and Scale-up PBX

Task 3
Characterize Performance and Degradation

Preliminary Feasibility/Risk Reduction

- SERDP SEED Project WP-1624:
  Demonstrated that cyclic dinitroureas
    - are stable under dry conditions enabling manufacturing and processing;
    - undergo rapid hydrolysis in high humidity to "self remediate" in the environment

Deliverables

- A viable and well characterized PBX formulation that will readily decompose into benign products when exposed to moisture (i.e., high humidity or precipitation). The new formulation will meet or exceed the performance of currently used formulations (e.g., PBXN-107).
- Interim Report (midway through project)
- Final Report
- Peer reviewed articles

Acknowledgements

- **Sponsor: SERDP** Weapons Systems & Platforms SEED Project WP-1624; full Project WP-2147

N,N-Dihaloamine Explosives to Defeat Biological Weapons

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DTRA Sponsor
Action Officer: Dr. Suhithi Peiris

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Project Background

Defense Threat Reduction Agency’s “Basic Research for Combating Weapons of Mass Destruction” program (HDTRA1-07-BRCWMD) called for novel energetic materials to produce ***biocidal*** reaction products from explosive events that destroy structures or containers

Prior Approaches

- Past agent defeat projects have used ***hydrogen chloride*** as a chemical neutralizer, e.g., Lockheed Martin's "Agent Defeat Warhead Device":

"These propellants produce gas-phase water and hydrogen chloride that combine to form very reactive hot hydrochloric acid; and as the reactants cool the cooled hydrochloric acid remains in the bunker and may act to continue neutralization of bunker contents for many days… Thus, incendiary agents based on standard composite rocket propellant technology are logical choices for the application described herein."
[Jones, J.W. *US Patent* 6382105 (2002)]

- However, it has long been known that **HCl has among the poorest bactericidal activities** of common acid species
[Paul, T. et al. *Biochemische Zeitschrift* **1911**, *29*, 202]

Proposal

- In contrast to HCl, **hydrogen fluoride** (HF) is an efficient antibacterial, antimicrobial biocide:
  - ➢ 200 ppm aq. HF destroys *Pseudomonas aeruginosa* and *Saccharomyces diastaticus* in 5 min
    [Bessems, E.; Junger, R. *GB Patent* 1584845 (1981)]
  - ➢ 1–100 ppm aq. HF sterilizes water lines
    [Tatsuno, T. et al. *US Patent* 5147605 (1992)]

- Deployed HF might be neutralized by application of gaseous ammonia, for example

Research Objectives and Approach

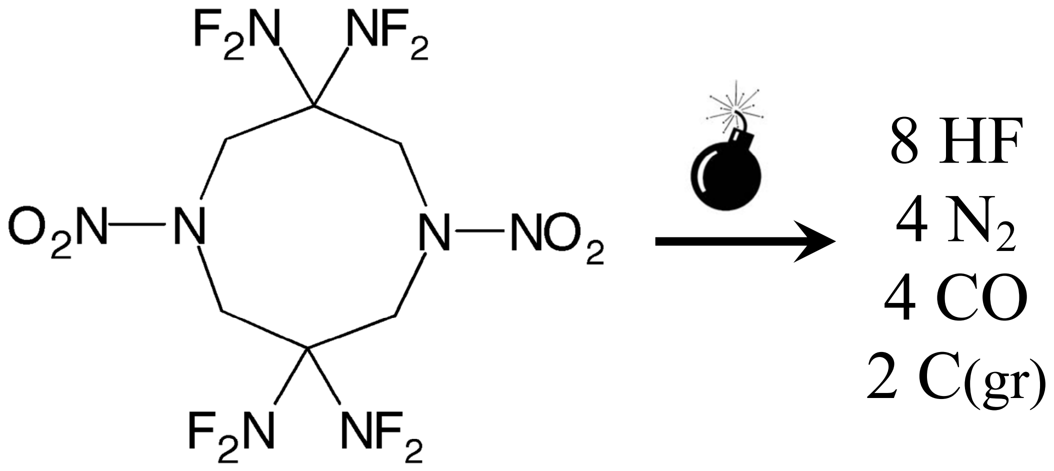
- Prepare and characterize novel chemical explosive compounds capable of producing explosion products that are efficient biocides: not hydrogen chloride but hydrogen fluoride (HF) or chlorine (Cl_2)

- Specifically, prepare certain explosive ***N,N*-dihalo-amine** compounds producing high ($\gtrsim$40 wt%) biocide content upon explosion

- Determine their potential efficacy as components of agent defeat weapons designs

Method: Energetic Difluoramines as HF Generators

- Known or new difluoramines of high biodical equivalent content:



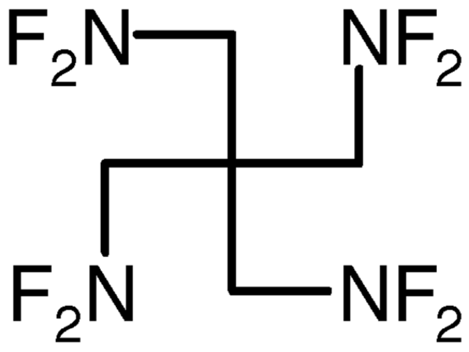
HNFX

>39% HF

7.99 moles HF

per mole HNFX (CHEETAH)

[Chapman, R.D.; Groshens, T.J.
U.S. Patent 7,632,943 (2009)]

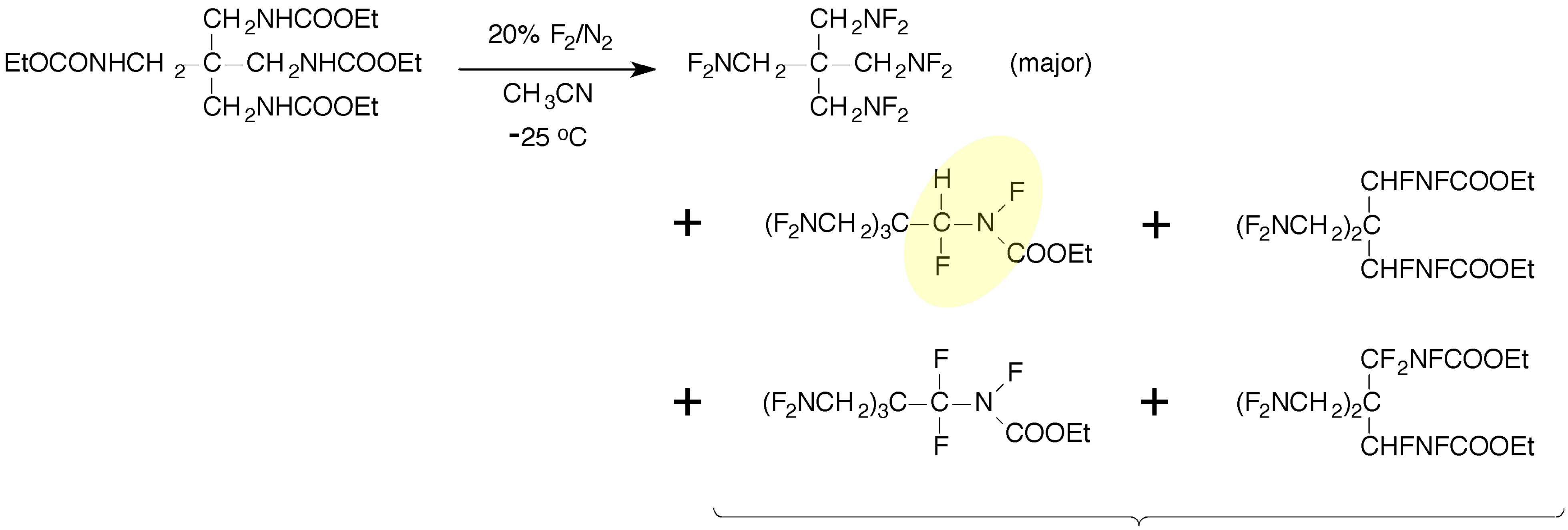


Octafluoro-
pentaerythrityl-
tetramine (F_8-PETA)

58% HF

[Chapman, R.D.; Hollins, R.A.
U.S. Patent Appl. (1 July 2009)]

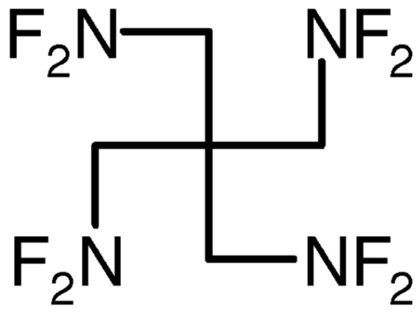
Octafluoropentaerythrityltetramine



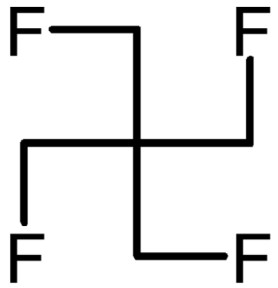
(minor, identified by ^{1}H, ^{19}F NMR)

Unprecedented linkage in chemical literature

Octafluoropentaerythrityltetramine



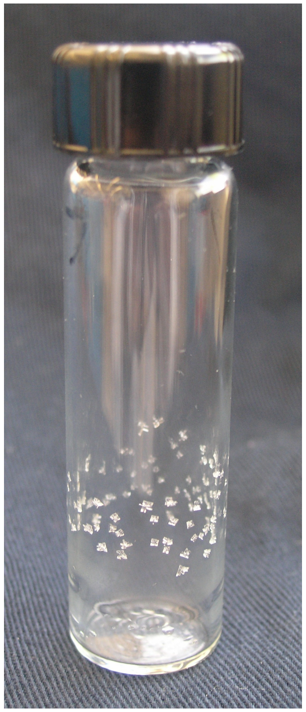
Compare to →



ρ = 1.70 g/cm^3 (X-ray, r.t.)

m.p. ≈ 40–42 °C

Volatile!
Sublimes ≤30 °C

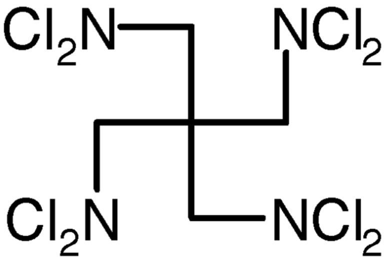


m.p. = 92 °C
b.p. = 110 °C
[Gryszkiewicz-Trochimowski, E.
Bull. Soc. Chim. France **1953**, 125]

- **F_8-PETA practicality in formulations is limited, due to volatility**

N,N-Dichloramines as Cl_2 Generators

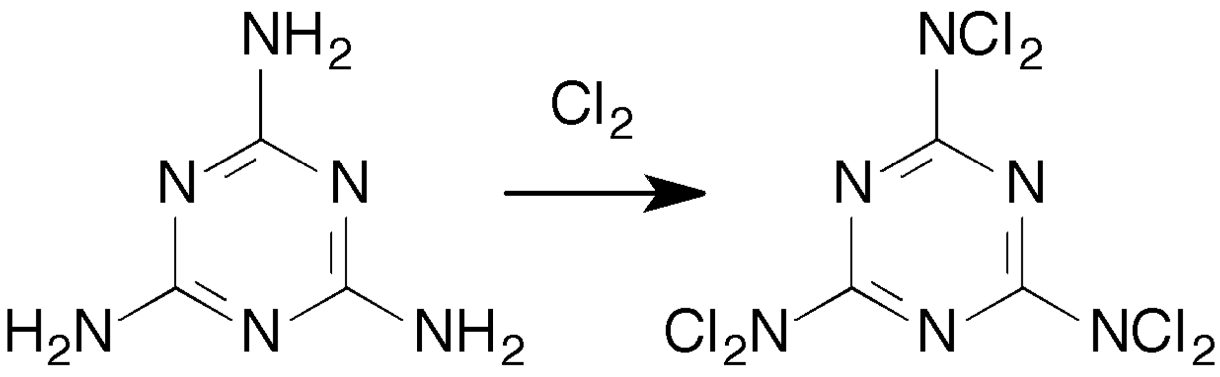
- Chlorine (Cl_2) is an effective biocide used by the Agent Defeat Warhead [http://www.globalsecurity.org/military/systems/munitions/adw.htm]
- *N,N*-Dichloramines ***can*** explosively generate Cl_2 (not HCl)
- Known dichloramine:



"octachloro-PETA"
70% Cl_2
"very powerful explosive"
[Gryszkiewicz-Trochimowski, E. et al. *Memorial des Poudres* **1958**, *40*, 109]

- but insufficient long-term stability

“Explosive” *N,N*-Dichloramine: Hexachloromelamine



hexachloromelamine

Preparation:

Hamprecht, G.; Mohr, R. *US Patent* 3364214 (1968)

(known to be biocidal)
64% Cl_2
African Explosives & Chemical Industries Ltd. *GB Patent* 1092994 (1967)

Sensitivity Results:

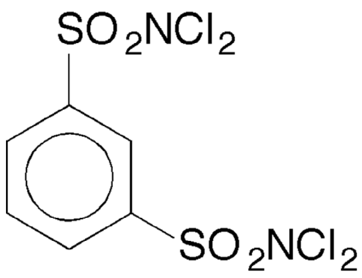
Impact: H_{50} ~ 9.2 cm
[detectable but not dramatic by sound and odor]
(PETN = 10.8 cm)

Electrostatic:
10/10 no-fires @ 0.25 J

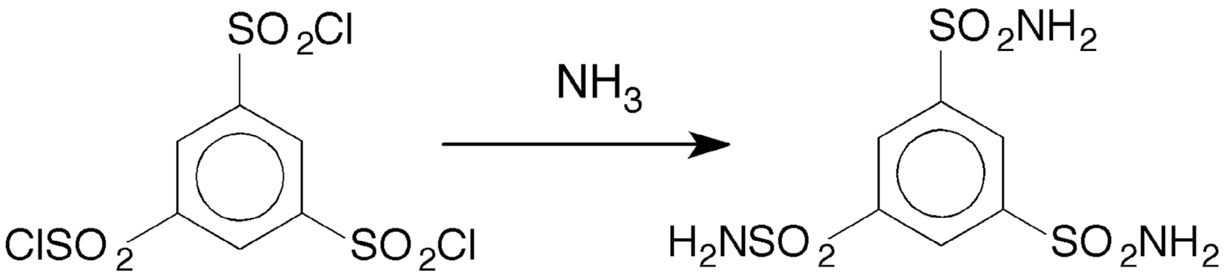
Friction (ABL):
10/10 no-fires at 1000 lbf

New *N,N*-Dichloramine Explosive

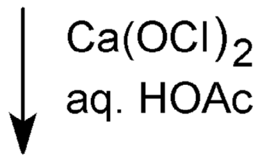
- *N,N*-Dichloramines stabilized as sulfonamides:



m.p. 128 °C
“explodes
with violence”
[Chattaway, F.D.
*J. Chem. Soc.
Trans.* **1905**, *87*, 145]



1,3,5-Benzenetrisulfonamide:
Jackson, C.L. *Am. Chem. J.* **1887**, *9*, 325

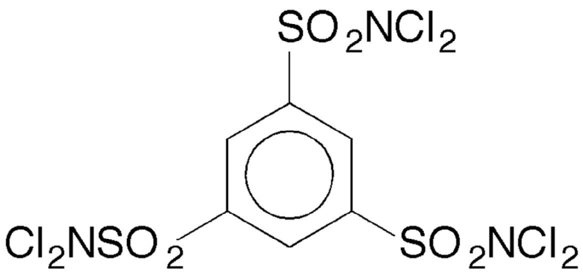


Sensitivity Results:

Impact: H_{50} ~ 7.8 cm
[sharp report;
heavy chlorine odor]
(PETN = 12.2 cm)

Electrostatic:
10/10 no-fires @ 0.25 J

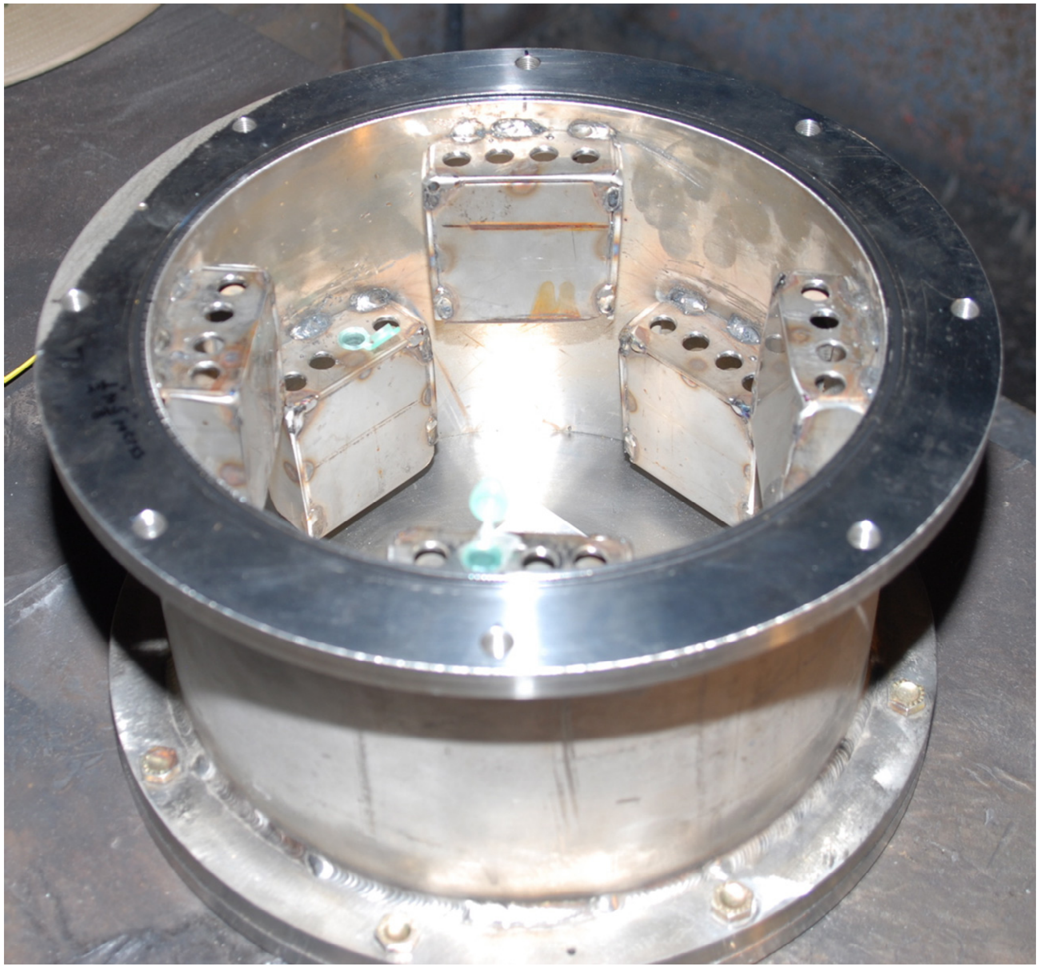
Friction (ABL):
F_{50} ~ 447 lbf
(PETN ≈ 316 lbf)



**1,3,5-Benzenetris-
(*N,N*-dichlorosulfonamide)**
m.p. 205 °C
41 wt% Cl_2

Method: Feasibility as Agent Defeat Weapons

- Detonate *N,N*-dihaloamine candidate in an 8.5-liter detonation chamber (Detonation Sciences Branch):



Agent Survivability Assessments

- Detonation chamber is set up with anthrax spore **surrogates** (*Bacillus thuringiensis* and/or *Bacillus subtilis*), prepared by Sun BioMedical Technologies (Ridgecrest, CA)

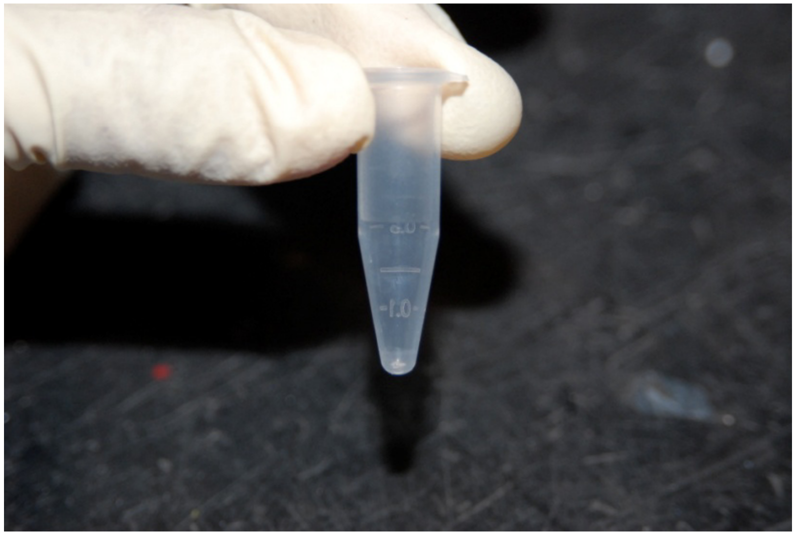
- Dried spores contained in polypropylene microcentrifuge tubes (FY 08–09) or stainless steel coupons (FY 10)
  - FY 08: $10^{7.3}$ colony forming units used in initial HNFX experiments
  - FY 09–10: $\gtrsim 10^9$ colony forming units total per detonation to demonstrate $\gtrsim 9$ orders of magnitude killing efficiency

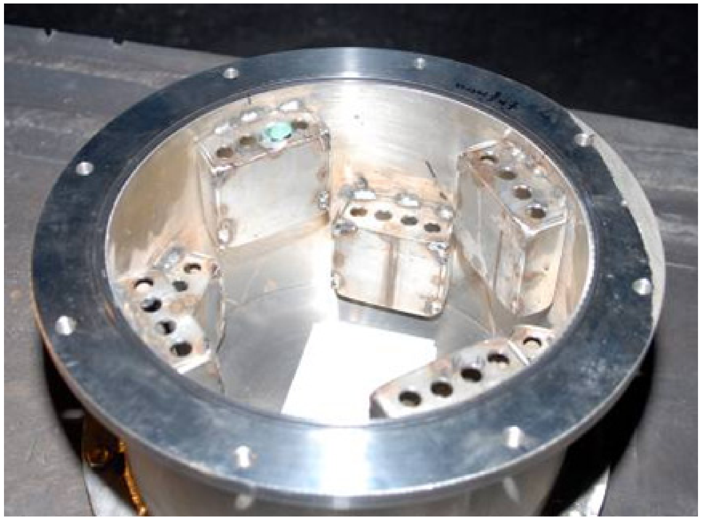
- Measure spore viability after various product-of-explosion exposure conditions
  - FY 08–09: various times following three detonations
  - FY 10: various chamber configurations, charge sizes

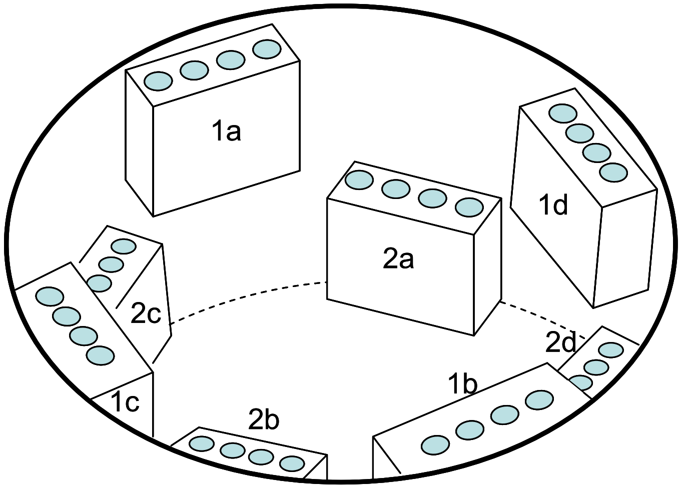
Agent Survivability Assessment: Setup (FY 08–09)

Right: Microcentrifuge tube for *Bacillus* spores

Below: (left) Detonation chamber; (right) the relative locations where *Bacillus* spore samples were placed for testing. Quadruplicate samples of two different *Bacillus* spores (*B. subtilis* and *B. thuringiensis*) at two different location levels (1 and 2) were tested during each detonation.



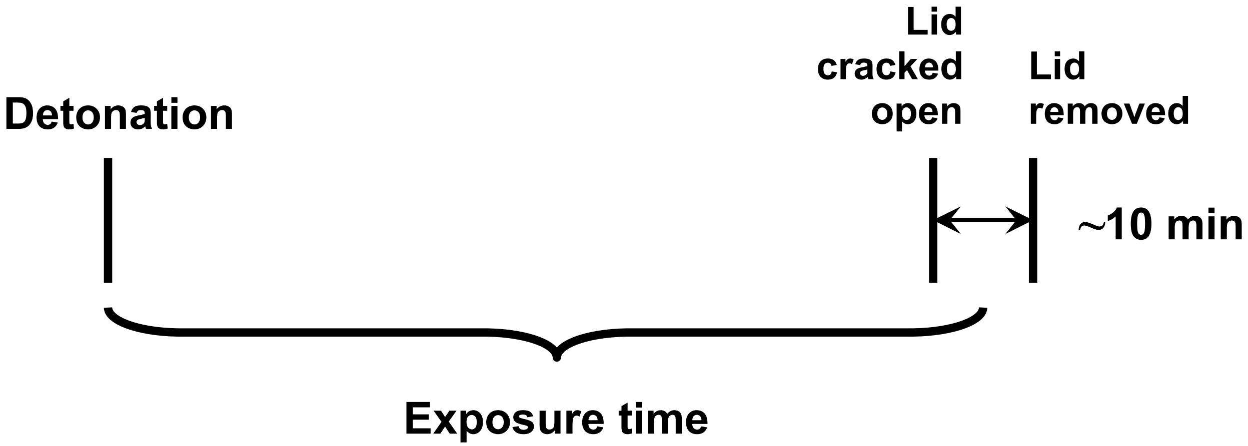




Agent Survivability Assessment (FY 08–09): Detonations

- Explosive charge contained in 4-cm^3 conductive polypropylene vial (2.7 ± 0.2 grams HNFX), initiated by an RP-3 or RP-2 miniature exploding bridgewire detonator

- Three exposure times: 0.4 hour, 3 hours, 24 hours



Detonation

Lid cracked open

Lid removed

~10 min

Exposure time

- Spore survivability analyzed at Sun BioMedical Technologies

HNFX Results (FY 08)

- Killing efficiency: log-reduction ≡ $R = \log(N_0 + 1) - \log(N_E + 1)$
  - N_0 = mean number from control (inoculated, unexposed)
  - N_E = mean number after exposure to detonation products

	Detonation #1 (0.4 h) N_E		**Detonation #2 (2.9 h) N_E**		**Detonation #3 (24.0 h) N_E**	
Sample:	***B. subtilis***	***B. thuringiensis***	***B. subtilis***	***B. thuringiensis***	***B. subtilis***	***B. thuringiensis***
Control $\overline{\log(N_0 + 1)}$	**6.956 ± 0.104**	**6.997 ± 0.067**	**6.956 ± 0.104**	**6.997 ± 0.067**	**6.956 ± 0.104**	**6.997 ± 0.067**
Exposed $\overline{\log(N_E + 1)}$	**0**	**0**	**0**	**0**	**0**	**0**
***R*(exposed)**	**≥6.96**	**≥7.00**	**≥6.96**	**≥7.00**	**≥6.96**	**≥7.00**
<*R*>(exposed)	**≥7.278 ± 0.060**		**≥7.278 ± 0.060**		**≥7.278 ± 0.060**	

The expectation value **<*R*>** treats all *Bacillus* spores collectively as surrogates of *B. anthracis* to estimate the log-reduction in survivability caused by detonation products.

HNFX Conclusions (FY 08)

- Zero *Bacillus* spores (out of $10^{7.3}$) were viable following any exposure time (0.4–24 hours)
- Spores were shielded from direct blast by steel plates; spore materials were intact, but not viable, following detonation
- *Bacillus* (*anthracis* and surrogates) spores have high dry heat resistance (minutes at 250–300 °C for $\gtrsim 4\ \log_{10}$ kill rate)
- No evidence of polypropylene melting, so chamber's contents did not exceed 160~165 °C for >1 sec
- Agent defeat not due to heat or pressure but harsh conditions of exposure to biocidal detonation products, tentatively proposed as HF

Hexachloromelamine Results

- Killing efficiency: log-reduction ≡ $R = \log(N_0 + 1) - \log(N_E + 1)$
  - N_0 = mean number from control (inoculated, unexposed)
  - N_E = mean number after exposure to detonation products

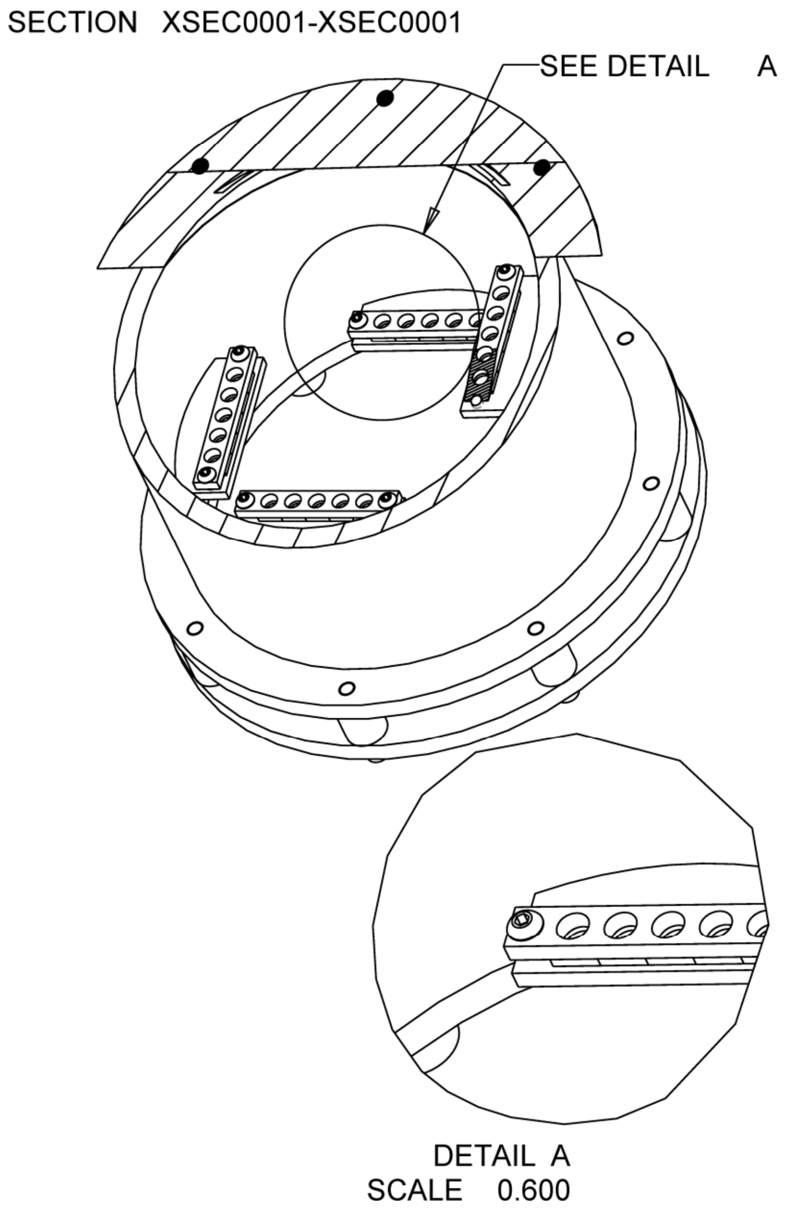
	Detonation #1 (0.4 h) N_E		Detonation #2 (3.0 h) N_E		Detonation #3 (24.0 h) N_E	
Sample:	***B. subtilis***	***B. thuringiensis***	***B. subtilis***	***B. thuringiensis***	***B. subtilis***	***B. thuringiensis***
Control $\overline{\log(N_0+1)}$	7.430 ± 0.044	8.955 ± 0.031	7.430 ± 0.044	8.955 ± 0.031	7.430 ± 0.044	8.955 ± 0.031
Uncapped $\overline{\log(N_E+1)}$	0	0	1.703 ± 1.823	0	0	0
Capped $\overline{\log(N_E+1)}$	6.806 ± 0.248	7.322 ± 0.501	3.072 ± 1.210	4.747 ± 3.362	4.482 ± 0.101	4.346 ± 0.219
R(capped)	0.624 ± 0.252	1.633 ± 0.502	4.358 ± 1.211	4.208 ± 3.362	2.948 ± 0.110	4.609 ± 0.221
R(uncapped)	>7.43	>8.95	5.727 ± 1.824	>8.95	>7.43	>8.95
<R>(uncapped)	>8.968 ± 0.054		7.265 ± 1.824		>8.968 ± 0.054	

The expectation value **<*R*>** treats all *Bacillus* spores collectively as surrogates of *B. anthracis* to estimate the log-reduction in survivability caused by detonation products.

Detonation Hardware Modification

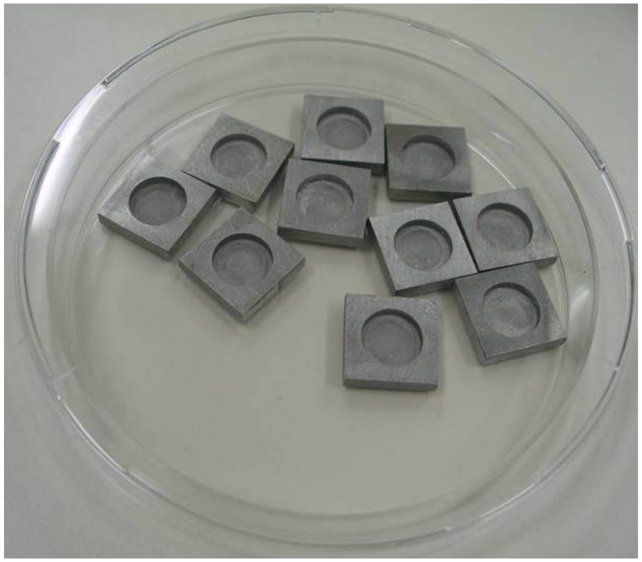
- Modification of experimental protocol in order to obtain nonzero survival of *Bacillus* spores
- Chamber hardware modified to allow variable exposure times: either chamber floor separated by spacers (very short exposures) or "sealed" chamber evacuated to vacuum at controlled times

Modified Detonation Chamber

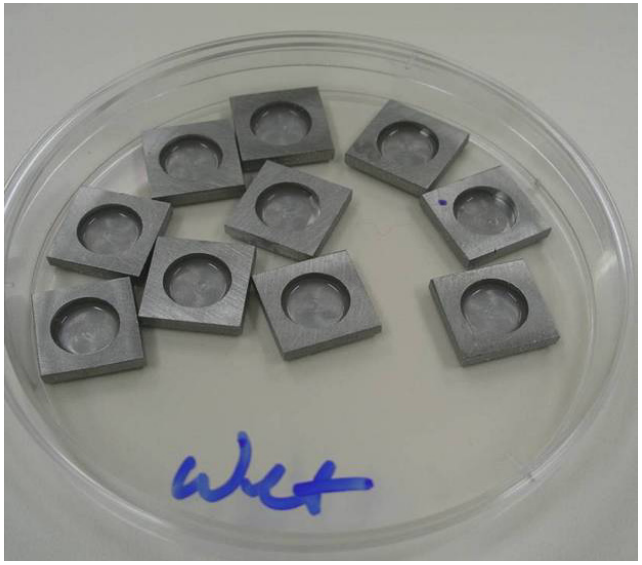


Modified Detonation Chamber

Spore Samples (Modified)



Dehydrated spore coupons

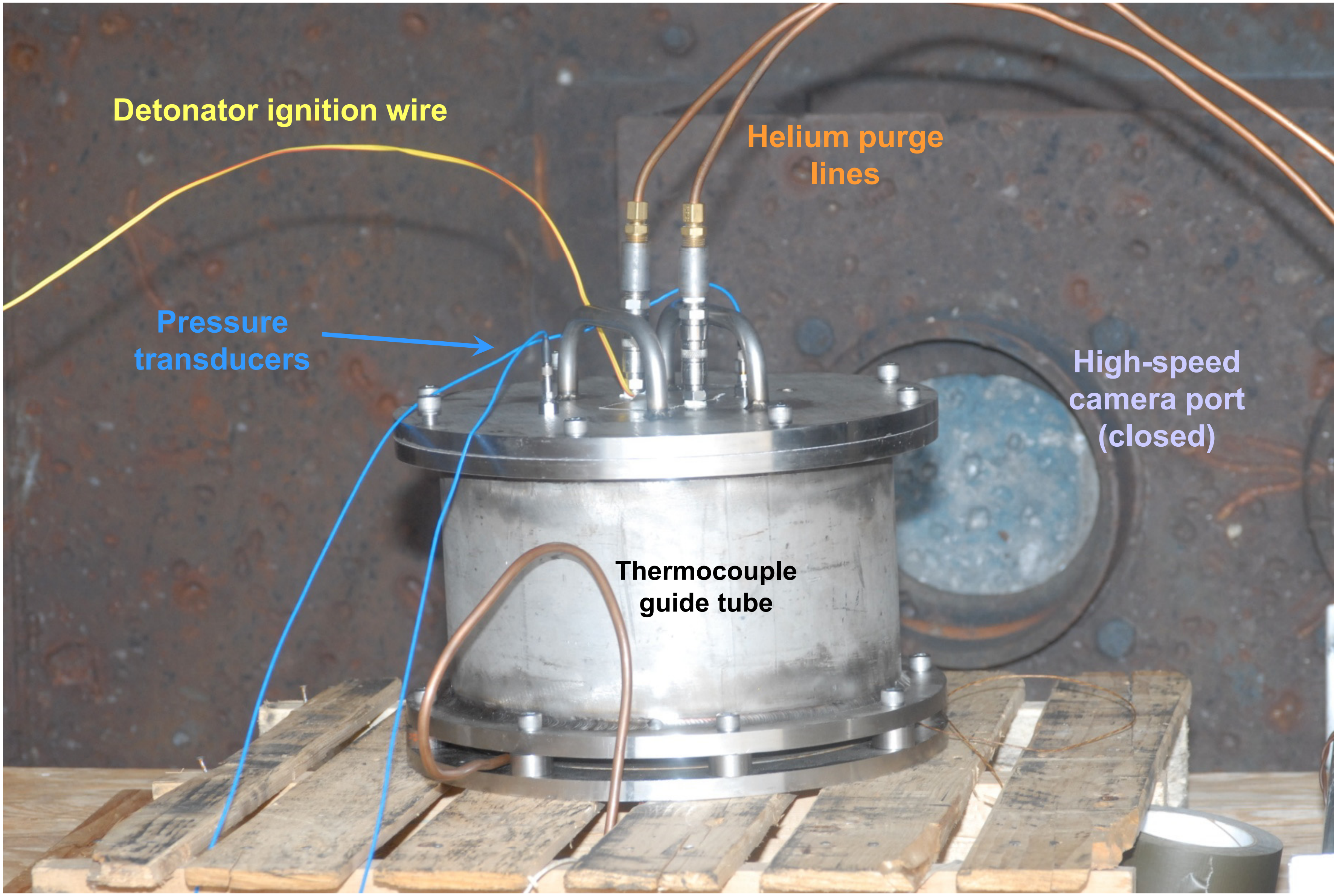


"Wet" spore coupons (10 µL glycerol in each)

Thermocouple coupon

Spore Sample Coupons in Place

Modified Detonation Chamber



HNFX Results (Modified Chamber)

- *B. thuringiensis* used both as dry spores and glycerol-wet
- Killing efficiency: log-reduction ≡ $R = \log(N_0 + 1) - \log(N_E + 1)$
  - N_0 = mean number from control (inoculated, unexposed)
  - N_E = mean number after exposure to detonation products

	HNFX Detonation #1		HNFX Detonation #2		HNFX Detonation #3		HMX Detonation	
Explosive charge size	2.64 g		2.38 g		2.09 g		2.67 g	
Chamber spacer	½″		1″		¼″		¼″	
Delay before He purge	“immediate” (but partial)		100 sec		180 sec		180 sec	
***Bt* sample state**	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
Control $\overline{\log(N_0+1)}$	9.472	9.511	9.520	9.571	9.502	9.623	9.575	9.651
Exposed $\overline{\log(N_E+1)}$	8.459	8.624	8.490	8.669	8.246	8.588	9.491	9.599
***R*(exposed)**	1.013	0.887	1.030	0.902	1.256	1.035	0.084	.052
<*R*>(exposed)	**0.945**		**0.958**		**1.109**		**0.064**	

The expectation value **<*R*>** treats all *Bt* spores collectively as surrogates of *B. anthracis* to estimate the log-reduction in survivability caused by detonation products.

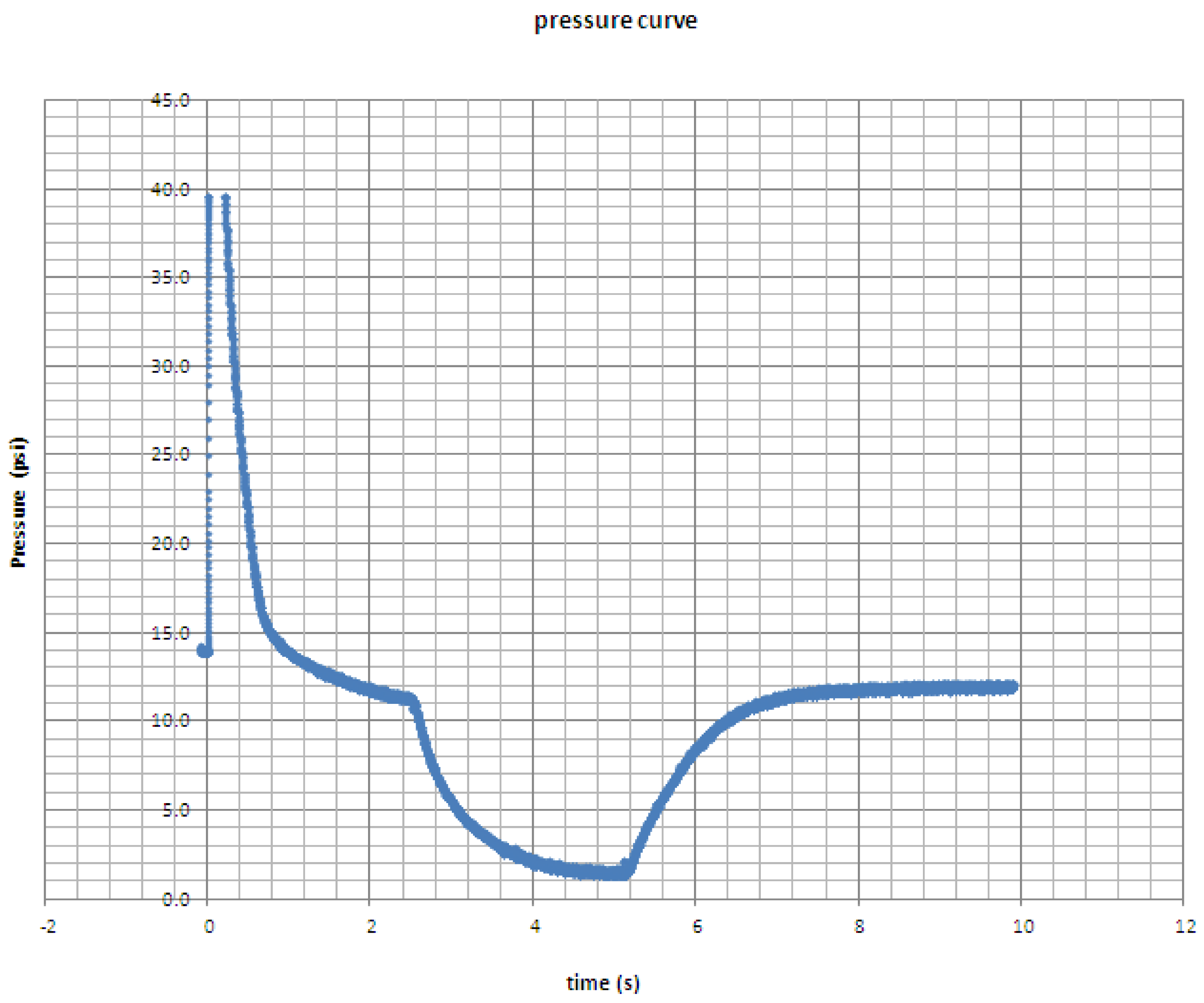
HMX “Sealed Chamber” Results

- HMX detonated in “sealed” chamber (open ignition wire port) as for HNFX in FY 08 experiments
- Killing efficiency: log-reduction ≡ *R* = $\log(N_0 + 1) - \log(N_E + 1)$
  - N_0 = mean number from control (inoculated, unexposed)
  - N_E = mean number after exposure to detonation products

	HMX Detonation	
Delay before opening	**0.4 hour**	
***Bt* sample state**	**Dry**	**Wet**
Control $\overline{\log(N_0+1)}$	**9.681**	**9.699**
Exposed $\overline{\log(N_E+1)}$	**9.487**	**9.504**
***R*(exposed)**	**0.194**	**0.195**
<*R*>(exposed)	**0.195**	
The expectation value **<*R*>** treats all *Bt* spores collectively as surrogates of *B. anthracis* to estimate the log-reduction in survivability caused by detonation products.		

Vacuum Chamber for "Convenient" Exposures (seconds)

Pressure vs. Time Profile (HNFX, 2.5 sec Exposure)

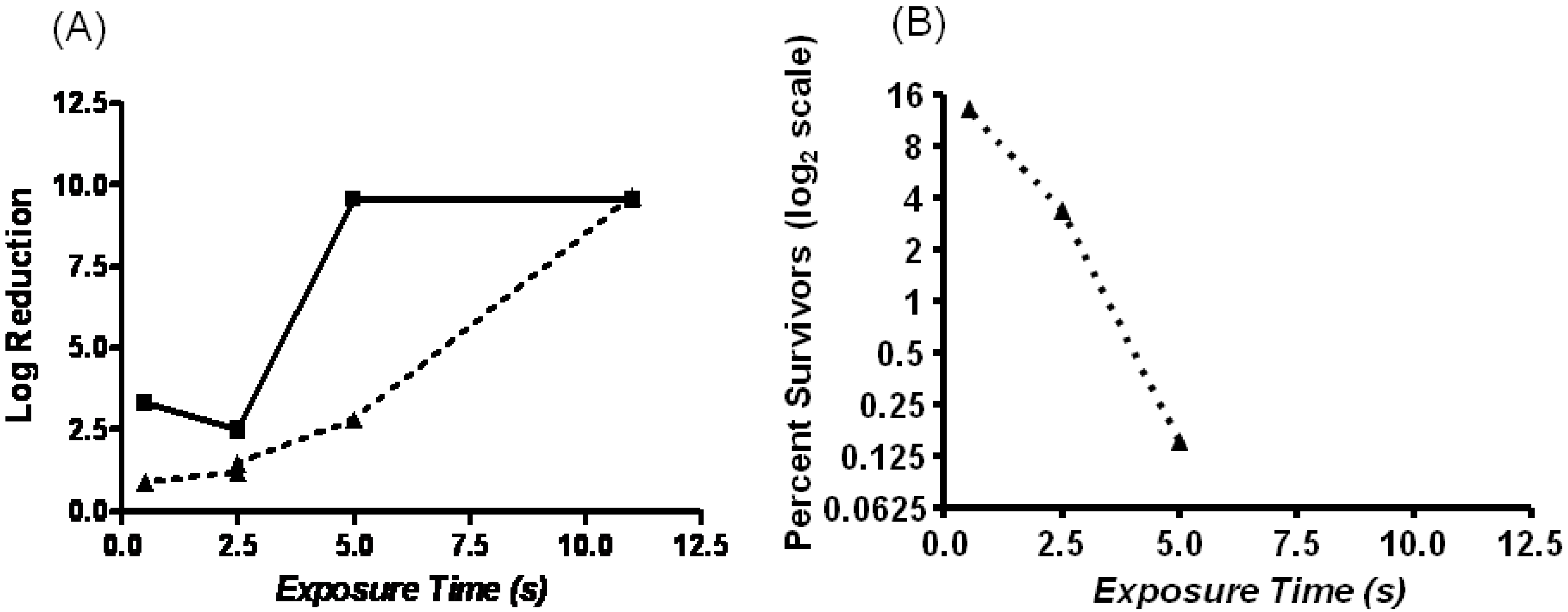


HNFX Results (Seconds Exposure)

- *B. thuringiensis* used both as dry spores and glycerol-wet
- Killing efficiency: log-reduction ≡ $R = \log(N_0 + 1) - \log(N_E + 1)$
  - N_0 = mean number from control (inoculated, unexposed)
  - N_E = mean number after exposure to detonation products

	HNFX Detonation #1		HNFX Detonation #3,4		HNFX Detonation #2	
Explosive charge size	**2.90 g**		**2.90 g**		**2.90 g**	
Delay before evacuation	**5 seconds**		**2.5 seconds**		**0.5 second**	
***Bt* sample state**	**Dry**	**Wet**	**Dry**	**Wet**	**Dry**	**Wet**
Control $\overline{\log(N_0+1)}$	**9.59**	**9.50**	**9.62 ± 0.01**	**9.50 ± 0.16**	**9.56**	**9.30**
Exposed $\overline{\log(N_E+1)}$	**0**	**6.68**	**7.14 ± 0.03**	**8.17 ± 0.03**	**6.42**	**8.42**
***R*(exposed)**	**≥9.59**	**2.82**	**2.48 ± 0.03**	**1.33 ± 0.16**	**3.14**	**0.88**

HNFX Results (Seconds Exposure)



Plot of spore kill rates vs. *Exposure Time.* (A) Graph of Log Reduction vs. Exposure Time for both dry spores (—■—) and glycerol-wetted spores (– ▲– ·). (B) Graph of Percent Survivors vs Exposure Time for glycerol-wetted spores. Extrapolation of the line to y = 0.000625 gave an Exposure Time of ~11.5 s.

Conclusions

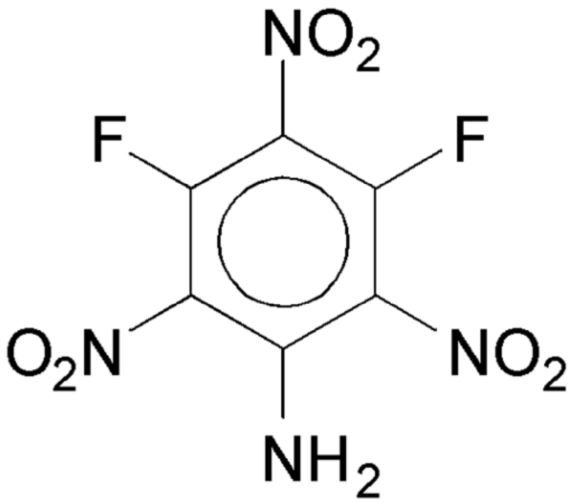
- Agent defeat not due to heat or pressure but transiently harsh conditions of exposure to biocidal detonation products (proposed conceptually to be HF or Cl_2, but other halogen species may be involved)

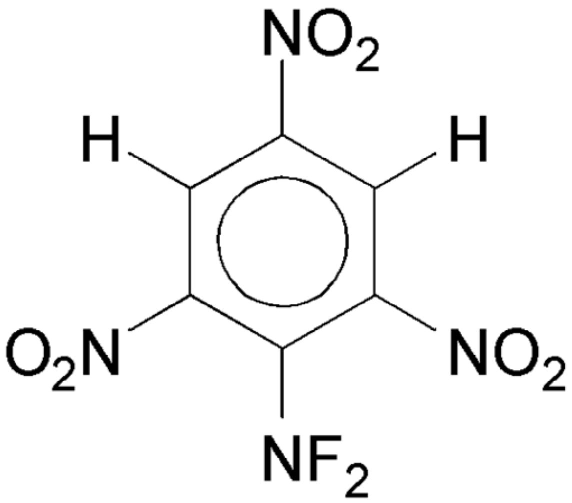
- High killing efficiency (>7.3 to ~9 $\log_{10}$-reduction) achieved by brief but "contained" exposure of spores to products from dihaloamine detonations (compared to ~0.2 $\log_{10}$ reduction from conventional explosive HMX)

- With the detonation chamber "open" (¼″–1″ spacers), exposure to highly biocidal products is too short (milliseconds?) to achieve high-orders-of-magnitude efficiency

Follow-on Project (FY 2011+)

- Test agent defeat by certain halogenated explosives on a larger scale (40~50 g) in collaboration with NSWC-IHD and NSWC-DD
- Compare C–F vs. N–F explosives as agent defeat product generators: e.g., 3,5-difluoropicramide vs. *N,N*-difluoropicramide (both known)





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