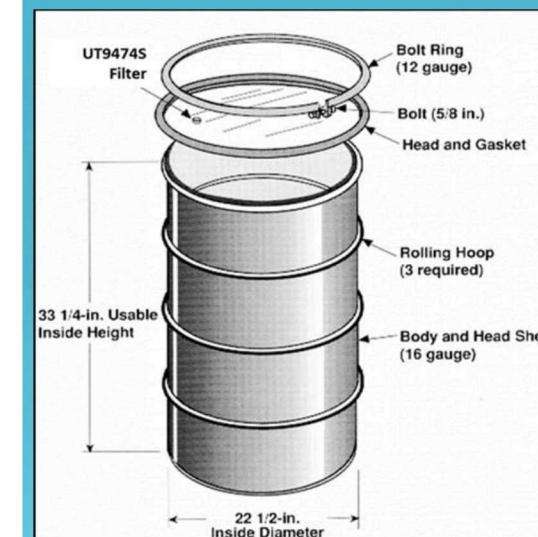




Sandia
National
Laboratories

SAND2019-15188PE

Thermal Testing to Determine ARF on 7A Drums Equipped With UT 9424S Filter



PRESENTED BY

Victor Figueroa, Hector Mendoza, Scott Sanborn 11/19/2019

SNL

SAND2019-####



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Team Members



1. Scott Sanborn (Program Manager, 8854)
2. Victor G. Figueroa (PI, 8853)
3. Hector Mendoza (Test Engineers, 8853)
4. Danielle Michel (Package Coordinator, 8854)
5. Walt Gill (Test Director, 1532)
6. Shane Adee (SNL Test Tech)
7. ECI Contractors for Setup and Test Execution

History 7A Testing at Sandia Prior 2019



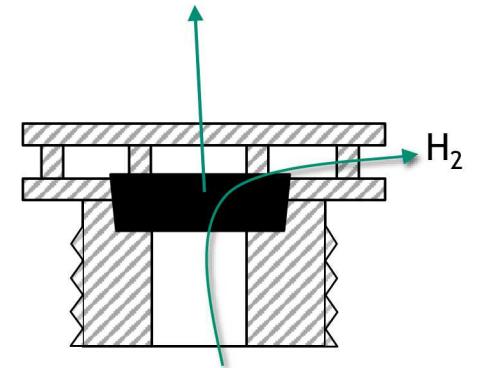
2015-2016 7A Package Fire Response Tests with Carbon Filter (NUCFIL-019DS)

- 7A filled near capacity with combustibles but with no instrumentation
- Inside the fire, demonstrated that 7A drum lid will be ejected
 - Air expansion is enough to cause lid ejection
- Outside the fire, 7A the drum lid stays on

2017 7A Package Fire Response Tests with new Plastic Sleeve Filters (UT9474S)

- 7A filled near capacity with combustibles but with no instrumentation
- Inside the fire, demonstrated that 7A drum lid will not be ejected
 - 7A drum still releases material through the filter hole in the drum lid
- However, no test conducted with 7A drums partially filled with combustibles

NUCFIL-019DS



UT9474S



2017 7A Fire Test

Gas jet
from filter
hole



Summary of 2017 Pool Fire Studies

- Documented in SAND2018-6570
- For drums with a **UT 9424S filter**
 1. The plastic filter sleeve melts/softens;
 2. The filter pops off about 1 min after fully engulfing conditions are met, opening up a $\frac{3}{4}$ -inch diameter hole;
 3. The internal drum pressure is relieved through the $\frac{3}{4}$ -inch diameter hole, and drum lid remains in place.
 4. At most 2/3 of the material remained inside the drum

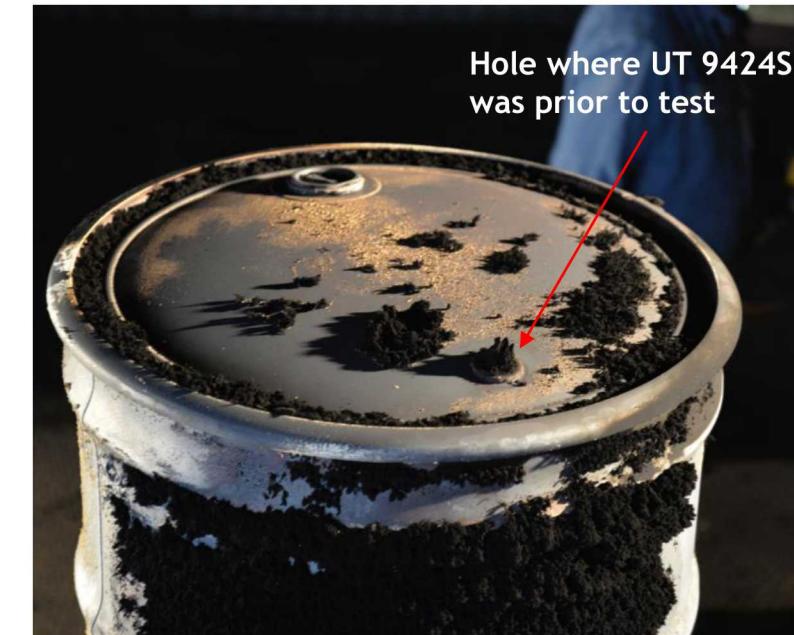
Material left inside the drum: drum outside fire (left) and inside (right)



(a)



(b)



7A drum after 30-minute fully engulfing pool fire

UT 9424S filter before [left] and after [right] pool fire



Motivation for Current 7A Test Program



All test prior to 2019, including the more resent test just presented, demonstrated that 7A drum lid will be ejected when drum is filled close to capacity

- 35-45psi pressure is required to open or eject the lid
- Typical loading (cellulose, plastic, rubber, metals, etc.)
 - Loading used were not necessarily bounding

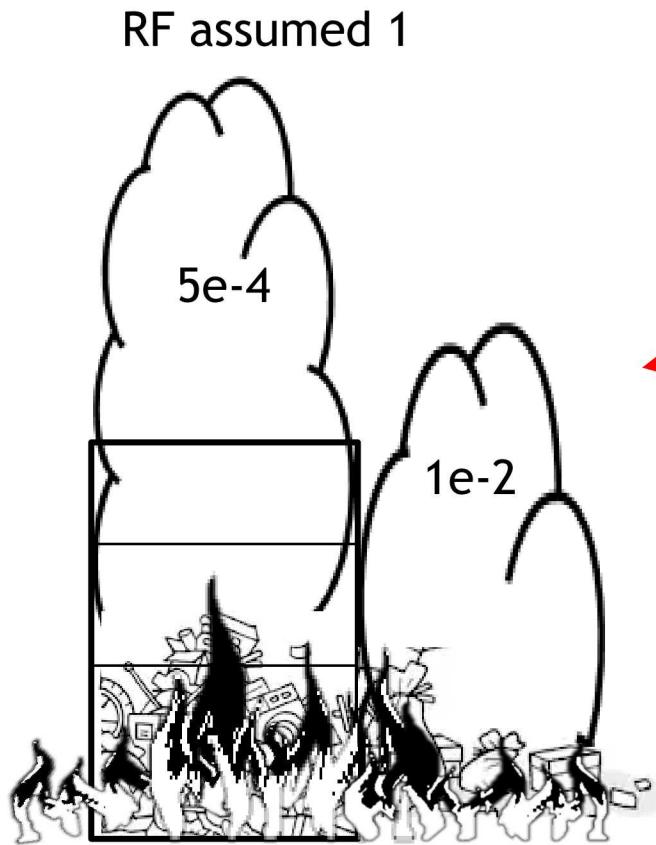
What happens when the 7A drums are loaded with bounding loads?

- Majority of the pressure built inside the drum is due to air
 - The more air volume, the faster the drum pressurizes, possibly leading to lid ejection even **with the new filter**

What is the ARF for 7A confined burning conditions?

- Not currently covered under DOE-STD-5506-2007

TABLE 4.5-1 ARF*RF Value Applicable to TRU Waste Accidents



Waste Form ¹ (surface-contaminated)		Explosion ²	Over-Pressure ³	Fire ⁴	Mechanical Insults	
					Spill ⁵	Impact ⁶
Combustible – cellulose, plastics	Ambient Atm.	(see fire) ⁷	---	1E-2 ⁷	---	---
	In container	(see fire)	1E-4	5E-4	1E-4	1E-4/2E-3
	In-flight	1E-4	---	---	---	---
Grout – cement, concrete		3E-4[ED] ^{8, 9}		<1E-6	7E-5	7E-4
Sludge or liquid slurries		MR ¹⁰	1E-4	2E-3	4E-5	MR ¹¹
Liquid		MR ¹⁰	2E-3	2E-3	1E-4	4E-5
Soil/Gravel, Powder, Granules		2E-4 ⁹	7E-2	6E-5	6E-4	1E-3
Metal, Non-Combustible materials not subject to brittle fracture		MR ¹⁰	1E-3 ¹²	6E-5 ¹²	1E-4 ¹²	1E-3 ¹²
HEPA filters	In-package	1E-2 ¹³	2E-3	1E-4	5E-4	
	Un-contained				1E-2	1E-3

¹ The event is assumed to fail any additional layers of plastic wrapping.

² Deflagration of H₂-air stoichiometric mixture that ejects lid and some fraction of the contents.

³ Internal pressure that fails the container and expels some fraction of the contents at a pressure \leq 500-psig.

⁴ Thermal stress that ejects lid and some of the contents. Some fraction of the ejected combustible contents may burn as well as the residual contents that remain in the open drum.

⁵ Some fraction of the contained powder and liquid contents are released from a location that is elevated to the equivalent of 3rd or 4th tier of stacked drums as defined in Table 4.4.4-1 and impacts a hard, unyielding surface.

⁶ The container is impacted with two possible levels of force. For lower energy impacts that do not crush the container, the "Spill" ARF*RF value of 1E-4 is applicable as discussed in Section 4.5.3.1. For impacts postulated that crush the container due to falling massive debris such as during a seismic event, or an errant blow from a high-speed vehicle crash that crushes the container, the cited value of 2E-3 is applicable as discussed in Section 4.5.3.2. The phenomena in this category are complex; and, provided a defensible technical basis is developed, other ARF & RF values are allowed.

⁷ For the fraction ignited from a container due to deflagration event or ejection from thermal effects that burns to completion.

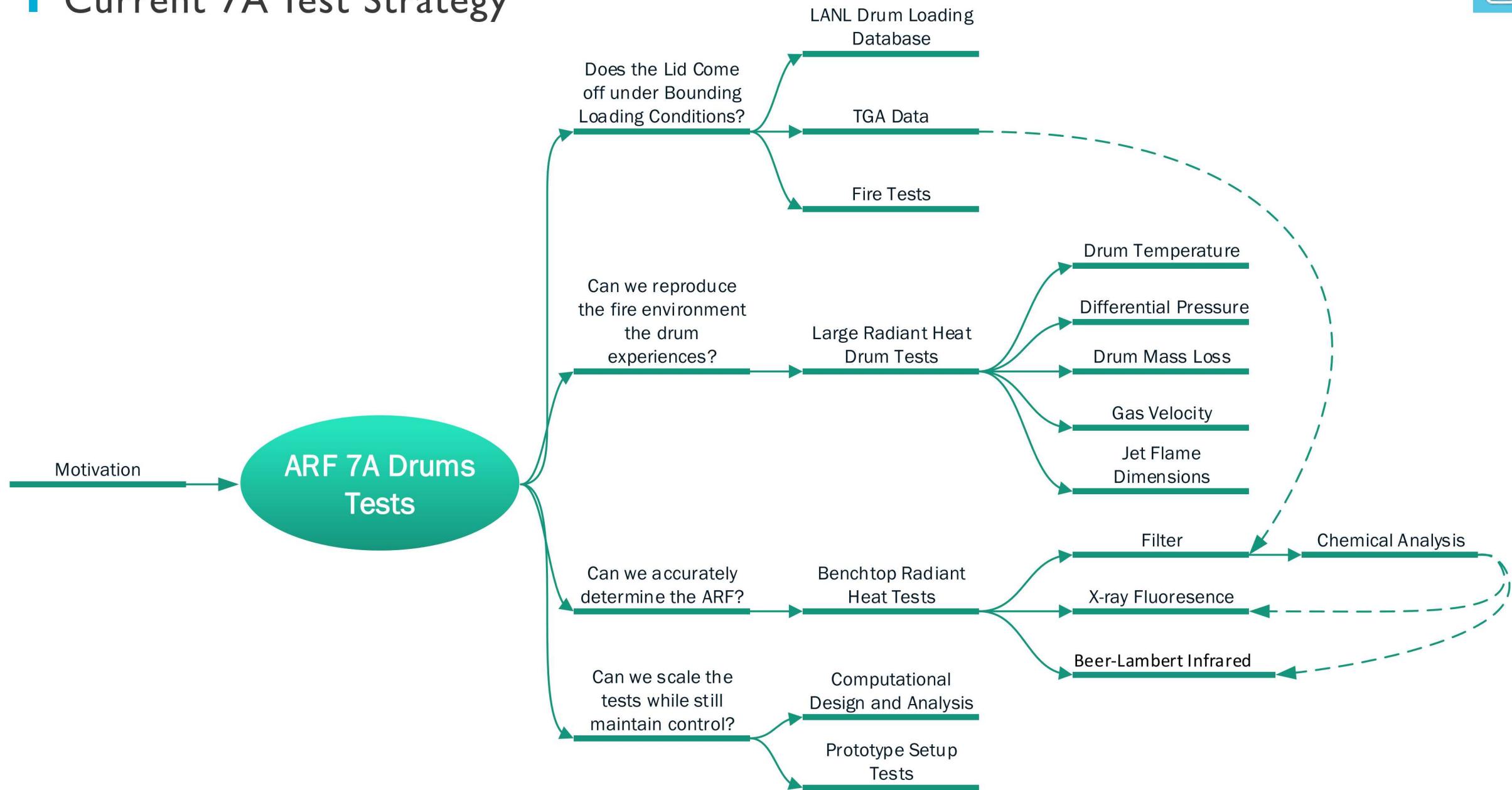
⁸ Applied to the volume of grout/cement affected, ED = Energy Density, J/cm³. Note: ARF*RF values vary according to drop height and material density. The density of concrete is used to approximate ARF/RF values. A drop height of

Opportunities and Risks



1. Dr. Robert Nelson (DOE-EM)
 - For 7A unconfined burns, ARF $\sim 1e-2$
 - ARF applicable to unconfined burns only
 - ARF estimates came from testing in the 1970s
 - Large uncertainties in ARF estimates – 20-25% of the mass unaccounted
2. Jim O’Neil (LANL-NNSA office at the time of 2017 tests)
 - Hypothesis that with the lid in-place, the ARF $< 1e-2$
 - Potential for lowering ARF estimates
 - Result in significant operational savings to LANL if this is the case
3. What if the ARF is larger than $1e-2$?
 - “Those applying the data must be aware of the range of stress represented by the measured ARFs, and seek to define the accident conditions to determine, in a gross sense, whether or not the stresses induced by the postulated events are bounded by the experimental parameters” DOE-HDBK-3010-94
4. Can we do better than the ARF tests (Jofu Mishima) conducted in the 1970s?
 - How do we collect/measure the released material from a drum inside a fire?
 - Difficult to field equipment inside a fire

Current 7A Test Strategy



Outline of Current Test Series



Green items completed
Blue is next step

1. Conduct pool fire tests to:
 - a) TGA Analysis to identify worst case scenario for material composition of drum contents
 - b) Test response of drum with worst case scenario/s identified in (1) while equipping the lid with a UT-9424S filter
 - c) Obtain temperature profile near drum to attempt to replicate with radiant heat setup
 - d) Obtain drum internal pressure profile to serve as verification for proper radiant heat setup
2. Reproduce fire environment based on data acquired in (2), but using a radiant heat setup to obtain:
 - a) Plume shape of effluent gas coming out of filter orifice on 7A drum lid for aerosol collection system design
 - b) Obtain velocity profile of effluent gas for aerosol collection system design
3. Design benchtop aerosol-release measurement system using small-scale tube furnace and debris samples contaminated with specified amounts of CeO₂
4. Using the knowledge learned in (2) and (3), perform a full-scale radiant heat test with an appropriately-sized ARF measurement system as identified by the tests in (3).

Pool Fire Tests for Fire Environment Definition (I)
(Does the lid come out of the 7A drum under bounding conditions?)

TGA Analysis (I)



LANL Database: Rubber, Cellulose, Plastic, Metals, etc.

Majority of combustibles

Format Painter

Clipboard

Font

Alignment

Number

Formatting

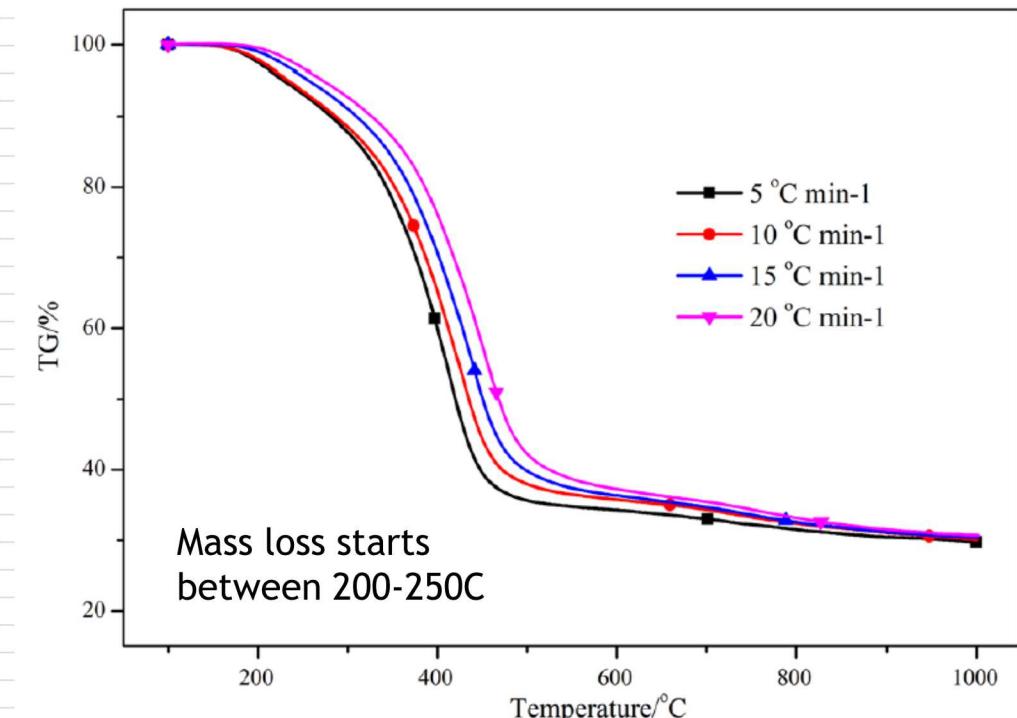
Table

Styles

E58 =AVERAGE(E7:E57)

6	A	B	C	D	E	F	G	H	I	J	K	L
	Container	WG Summary	Type	Waste Stream	Cellulosics (kg)	Plastics (kg)	Rubber (kg)	Inorganic Matrix (kg)	Iron-based Metals (kg)	Aluminum-based Metals (kg)	Other Metals (kg)	Other Inorganics (kg)
7	67727	Debris	Original	New Gen Awaiting Assignm	8.6	1.5			0.2			
8	67744	Debris	Original	New Gen Awaiting Assignm	3.4	0.4			6			
9	67748	Debris	Original	New Gen Awaiting Assignm	4.2	0.6			7			
10	67745	Debris	Original	New Gen Awaiting Assignm	3.4	0.5			6			
11	67742	Debris	Original	New Gen Awaiting Assignm	4.4	0.6			9			
12	87826	Debris	RemediationDaughter	LA-MHD01.001	24.8	0.5						
13	87827	Debris	RemediationDaughter	LA-MHD01.001	8.8	0.5						
14	67723	Debris	Original	New Gen Awaiting Assignm	6	1.5	0.5		1	1		
15	67743	Debris	Original	New Gen Awaiting Assignm	4.5	0.7			16			
16	67720	Debris	Original	New Gen Awaiting Assignm	4	1.4			2			0
17	67693	Debris	Original	New Gen Awaiting Assignm	3.5	1.4	0.1					
18	67718	Debris	Original	New Gen Awaiting Assignm	3	1.5	9.8					9.3
19	67716	Debris	Original	New Gen Awaiting Assignm	2	3						
20	67758	Debris	Original	New Gen Awaiting Assignm	1.5	2.3						
21	67697	Debris	Original	New Gen Awaiting Assignm	0	0.5	15.6					
22	67698	Debris	Original	New Gen Awaiting Assignm	0	0.5	10.1					
23	67757	Debris	Original	New Gen Awaiting Assignm	2	3.5	3.6					
24	67704	Debris	Original	New Gen Awaiting Assignm	0	2.6	25.6					
25	67713	Debris	Original	New Gen Awaiting Assignm	0	2.6	20.8					
26	67759	Debris	Original	New Gen Awaiting Assignm	3	6.2						
27	67703	Debris	Original	New Gen Awaiting Assignm	2.4	5						
28	67715	Debris	Original	New Gen Awaiting Assignm	0.1	1.5	11.3					
29	67751	Debris	Original	New Gen Awaiting Assignm	3	7			5			1.5
30	67666	Debris	Original	New Gen Awaiting Assignm	0.5	1.5			2.1			1
31	68987	Debris	RemediationDaughter	LA-MHD01.001	1	3			0.9			
32	67726	Debris	Original	New Gen Awaiting Assignm	0.8	3			0.1			0.1
33	67728	Debris	Original	New Gen Awaiting Assignm	1.6	6						
34	67717	Debris	Original	New Gen Awaiting Assignm	0.5	2						

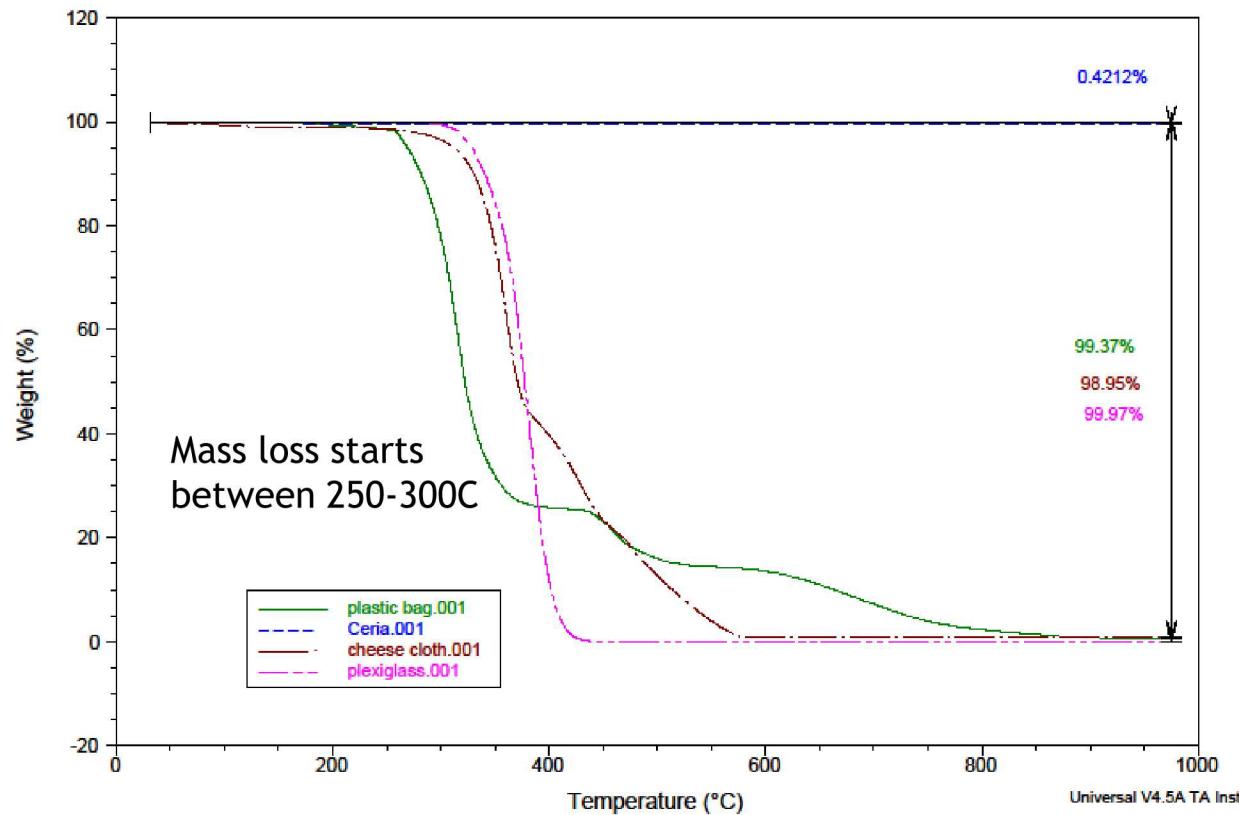
TGA analysis: Rubber Gloves



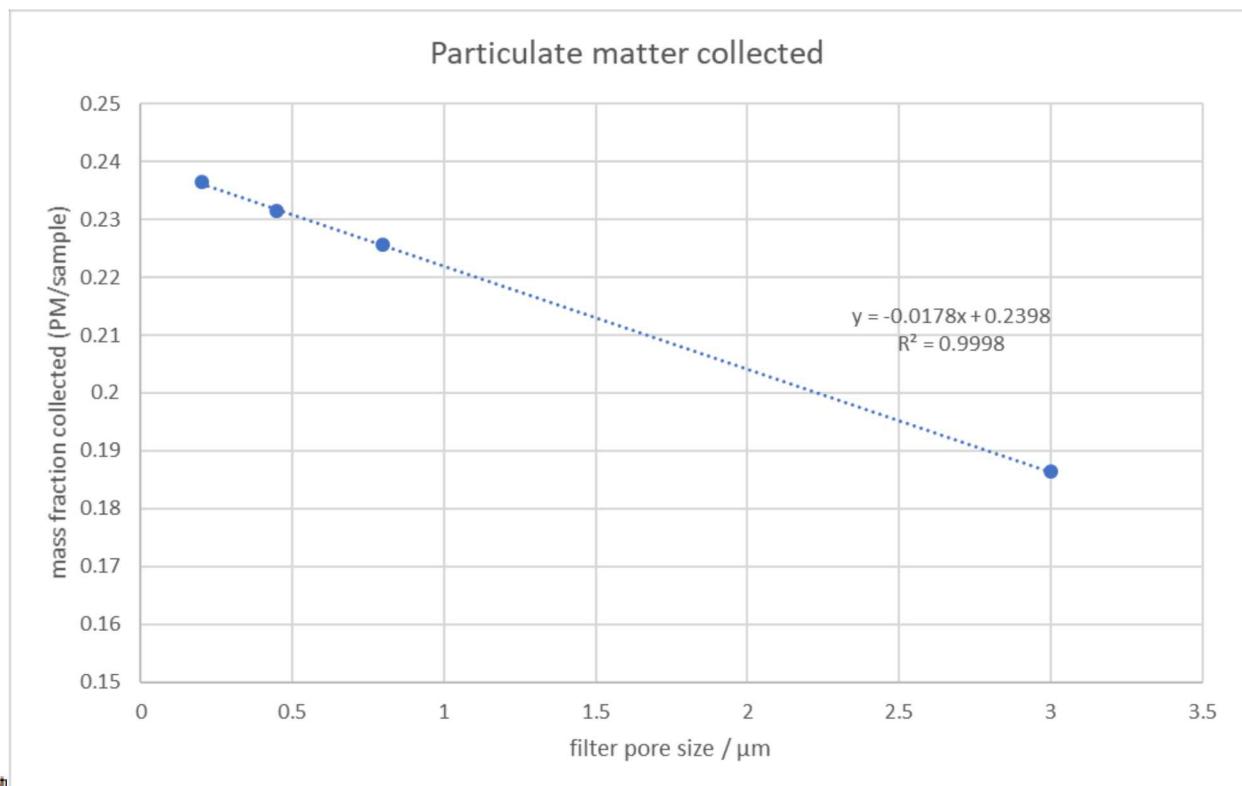
TGA Analysis (2)



TGA Cellulose, PMMA, Plastic Bag Gloves

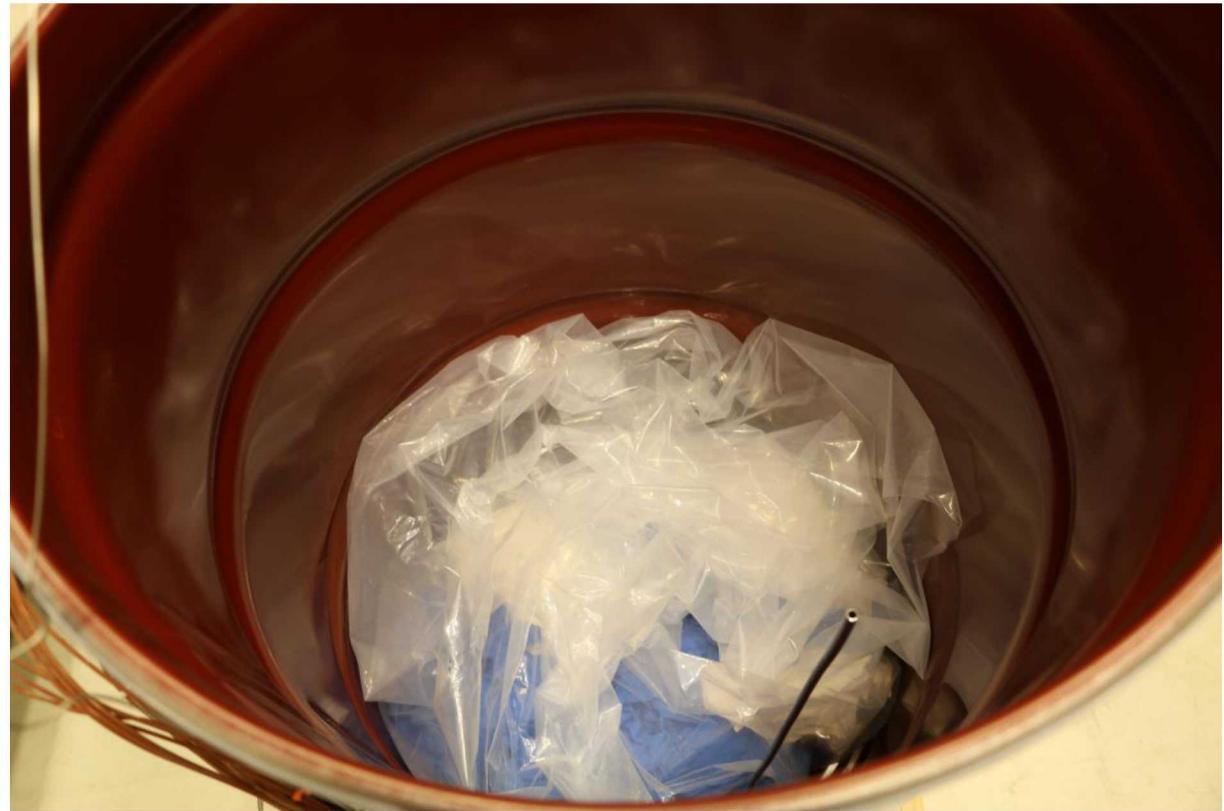


Plastic matter condensed on filter of various sizes

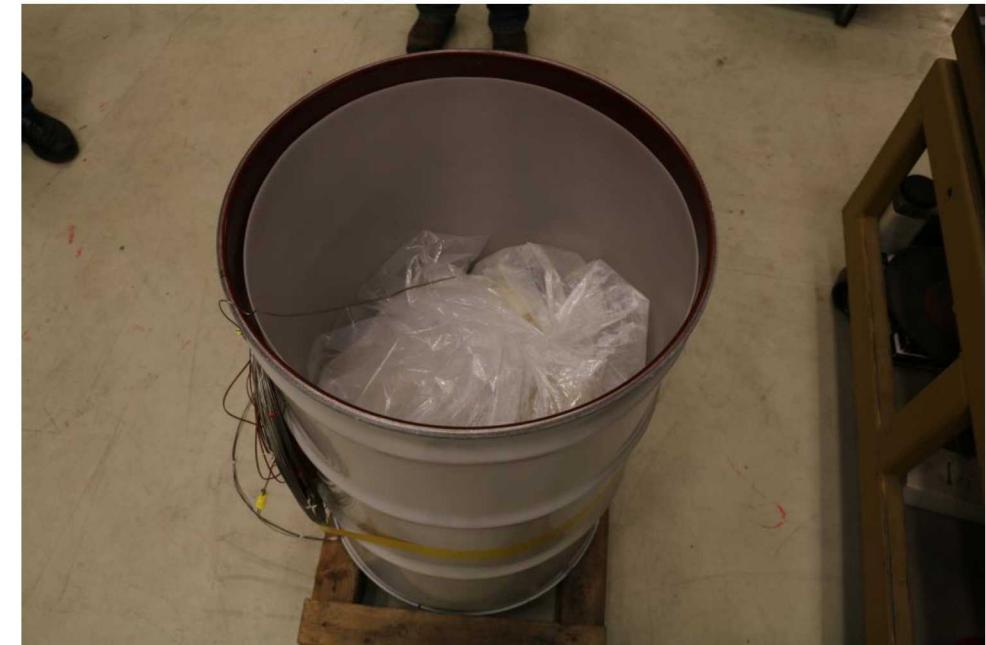
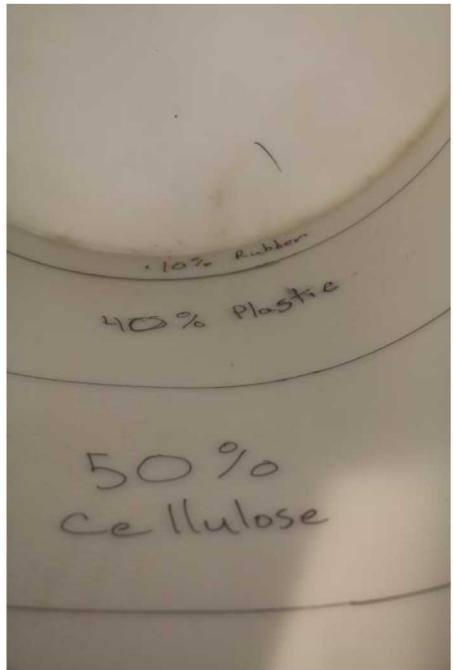


Respirable Fraction < 10-microns

Test #1 (No Rigid Plastic Liner, 20% Debris)



Test #2 (WITH Rigid Plastic Liner, 60% Debris)



Pool Fire Test Matrix



Mock fire tests demonstrated that with no material inside the drum, the lid will not be ejected with the new filter.

Test Location	Test #1				Test #2
	Center	55 kW/m ²	45 kW/m ²	35 kW/m ²	Center
% of drum volume occupied by debris	20.00%	20.00%	20.00%	20.00%	60.00%
Volumetric debris composition	85% rubber, 15% cellulose, + plastic bag	50% cellulose, 40% plastic, 10% rubber, + plastic bag. ^[1]			

^[1]Drum was equipped with rigid liner, therefore volume percentages are based on the remaining volume after liner is placed inside drum. 1290 g of CeO₂ were also added to debris

^[2] This mass includes the rigid liner and the 1290 g of CeO₂

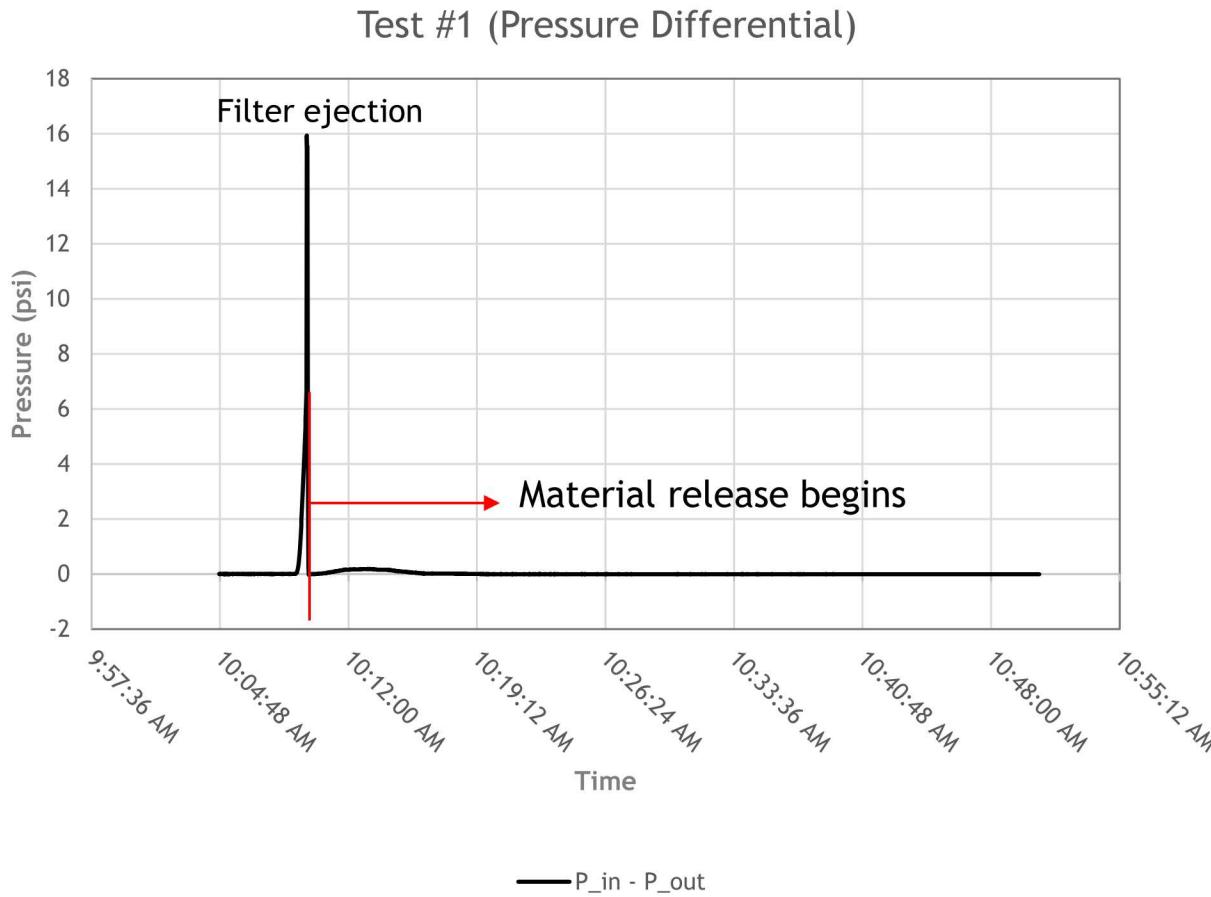
Pool Fire Test Setup



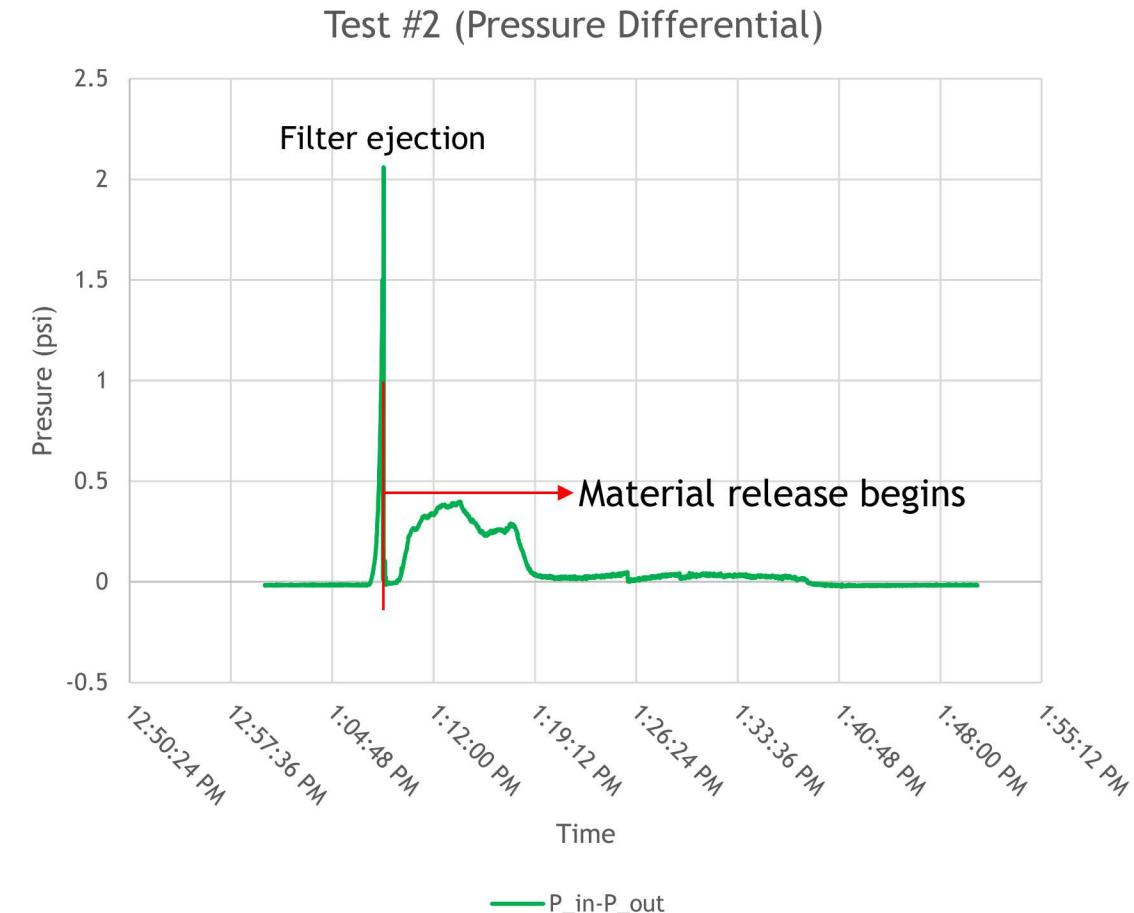
Results on Pressure Evolution



No lead ejection in either test



Higher peak pressure prior to filter release for 20% volume loading due to more air inside the drum.



Mass Loss Results



Test Location	Test #1				Test #2
	Center	55 kW/m ²	45 kW/m ²	35 kW/m ²	Center
% of drum volume occupied by debris	20.00%	20.00%	20.00%	20.00%	60.00%
Volumetric debris composition	85% rubber, 15% cellulose, + plastic bag	50% cellulose, 40% plastic, 10% rubber, + plastic bag. ^[1]			
Lid Loss?	No	No	No	No	No
Initial mass of drum contents (kg)	2.80	3.00	3.68	3.58	8.86 ^[2]
Pre-tested and fully assembled drum mass (kg)	31.18	31.20	31.90	32.10	38.60
Mass Loss (kg)	2.44	0.50	0.14	0.02	6.30
Mass Loss (% of initial contents)	87.14%	16.67%	3.80%	0.56%	71.11%
Peak Pressure differential	~16 psi	N/A	N/A	N/A	-2 psi

[¹]Drum was equipped with rigid liner, therefore volume percentages are based on the remaining volume after liner is placed inside drum.

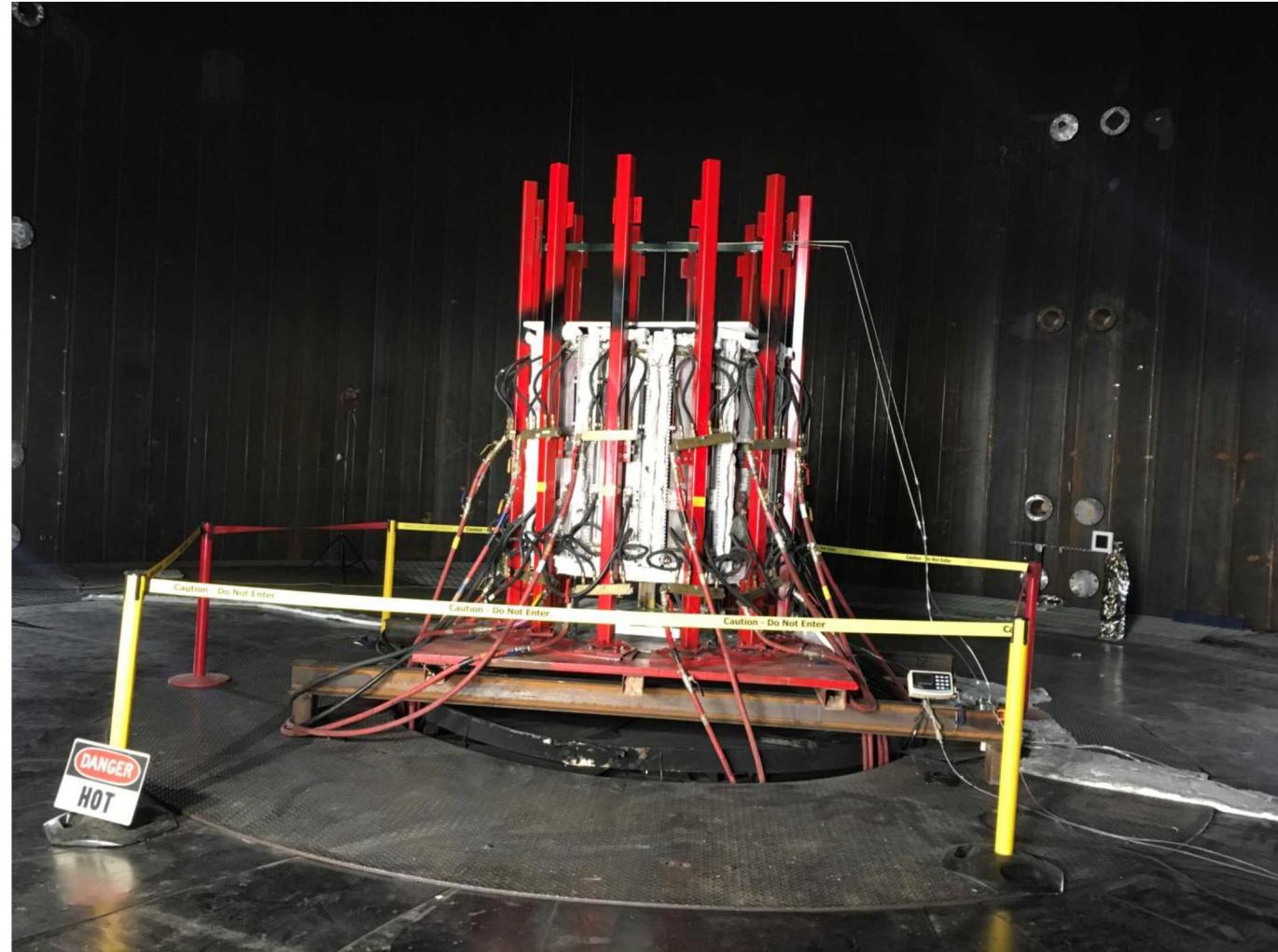
[²] This mass includes the rigid liner and the 1290 g of CeO₂

High mass loss. How much CeO₂ are we releasing in this confined burn configuration?

Process and Results of Radiant Heat Tests (2)

(Can we reproduce the response of the loaded 7A drum in an alternate environment more conducive for collecting/measuring CeO₂ release?)

Radiant Heat Test Setup



Test Matrix for Radiant Heat Tests



	Test #1	Test #2
Test Location	Center	Center
% of drum volume occupied by debris	20.00%	60.00%
Volumetric debris composition	85% rubber, 15% cellulose, + plastic bag	50% cellulose, 40% plastic, 10% rubber, + plastic bag. ^[1]
^[1] Drum was equipped with rigid liner, therefore volume percentages are based on the remaining volume after liner is placed inside drum.		
^[2] This mass includes the rigid liner		

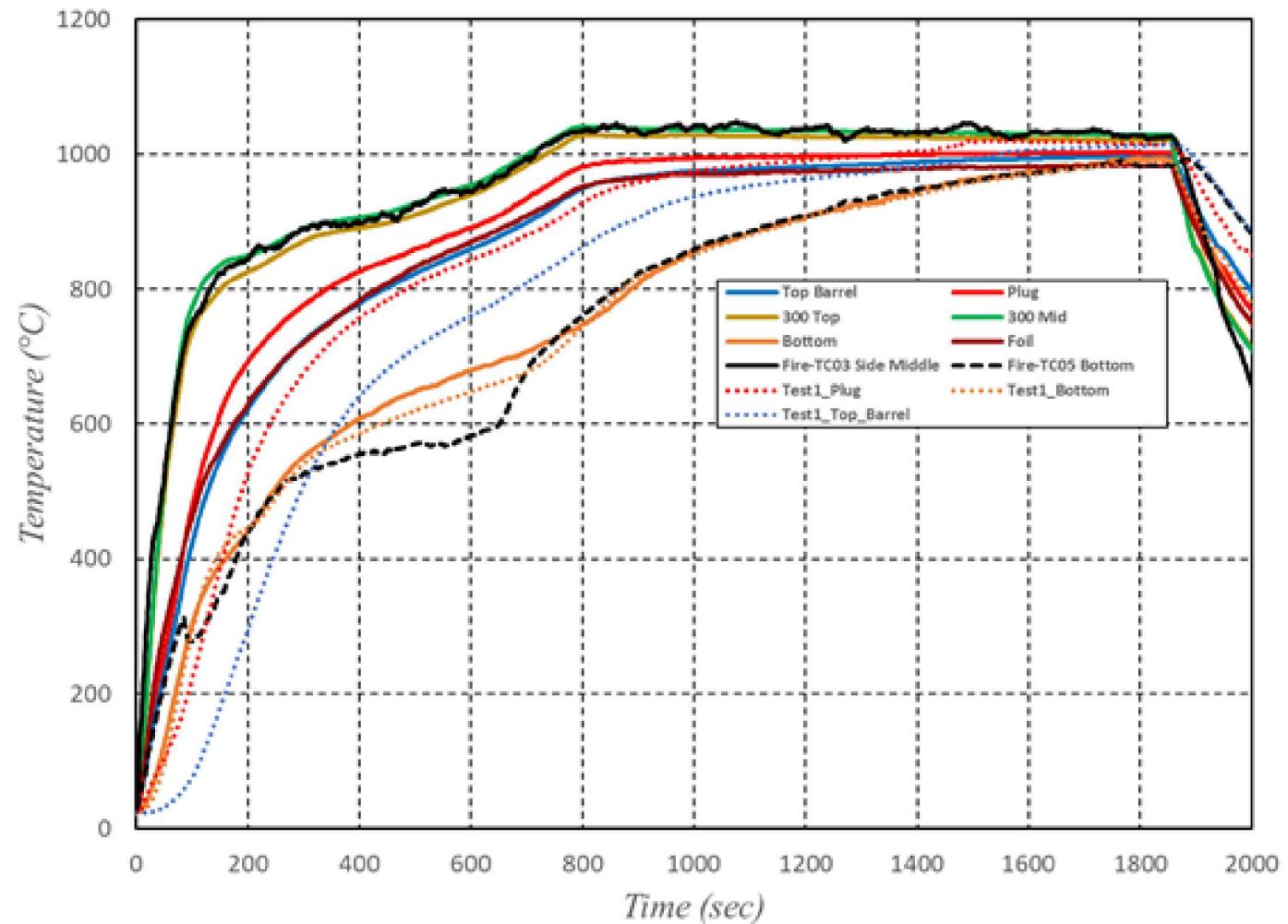
Loading is essentially the same as pool fire tests, but note that no filter was used on the drum lid on either of these radiant heat tests

Profile Matching for Radiant Heat Tests



Lines of interest:

1. Solid black (fire) and green (radiant heat) lines should match for external drum wall temperature at mid-height
2. Dashed black (fire) and solid orange (radiant heat) lines should match for external drum bottom temperature
3. Solid red line (radiant heat final setup) should rise faster than dashed red line (radiant heat preliminary setup) to show improvement in heating rates for drum-filter region

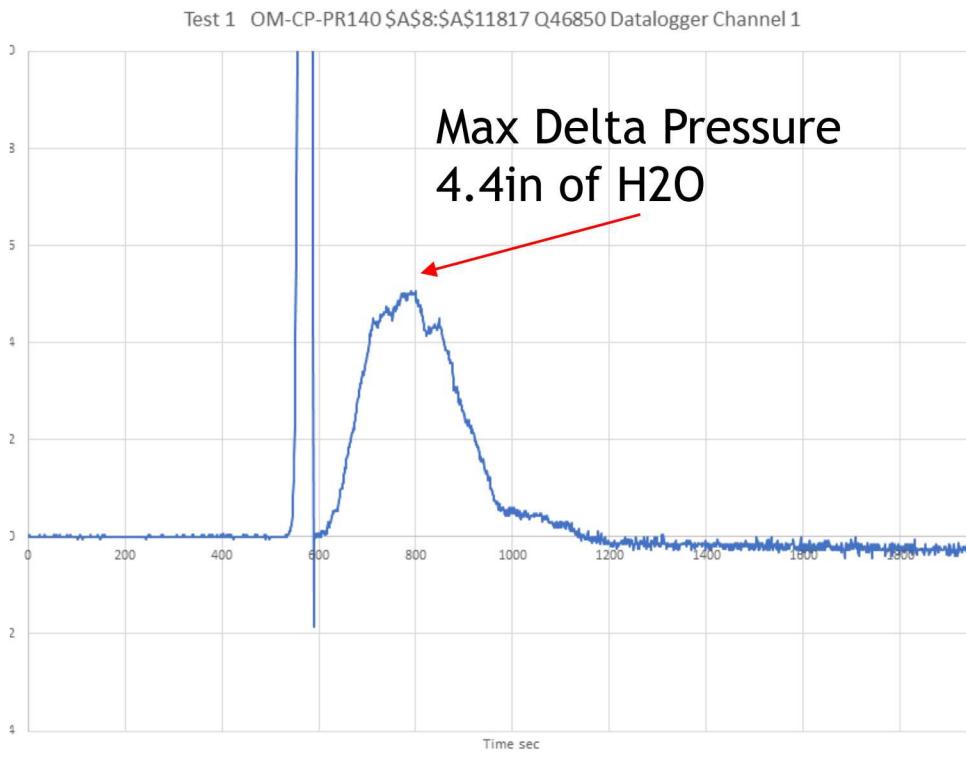


Capable of reproducing temperatures on skin of the drum

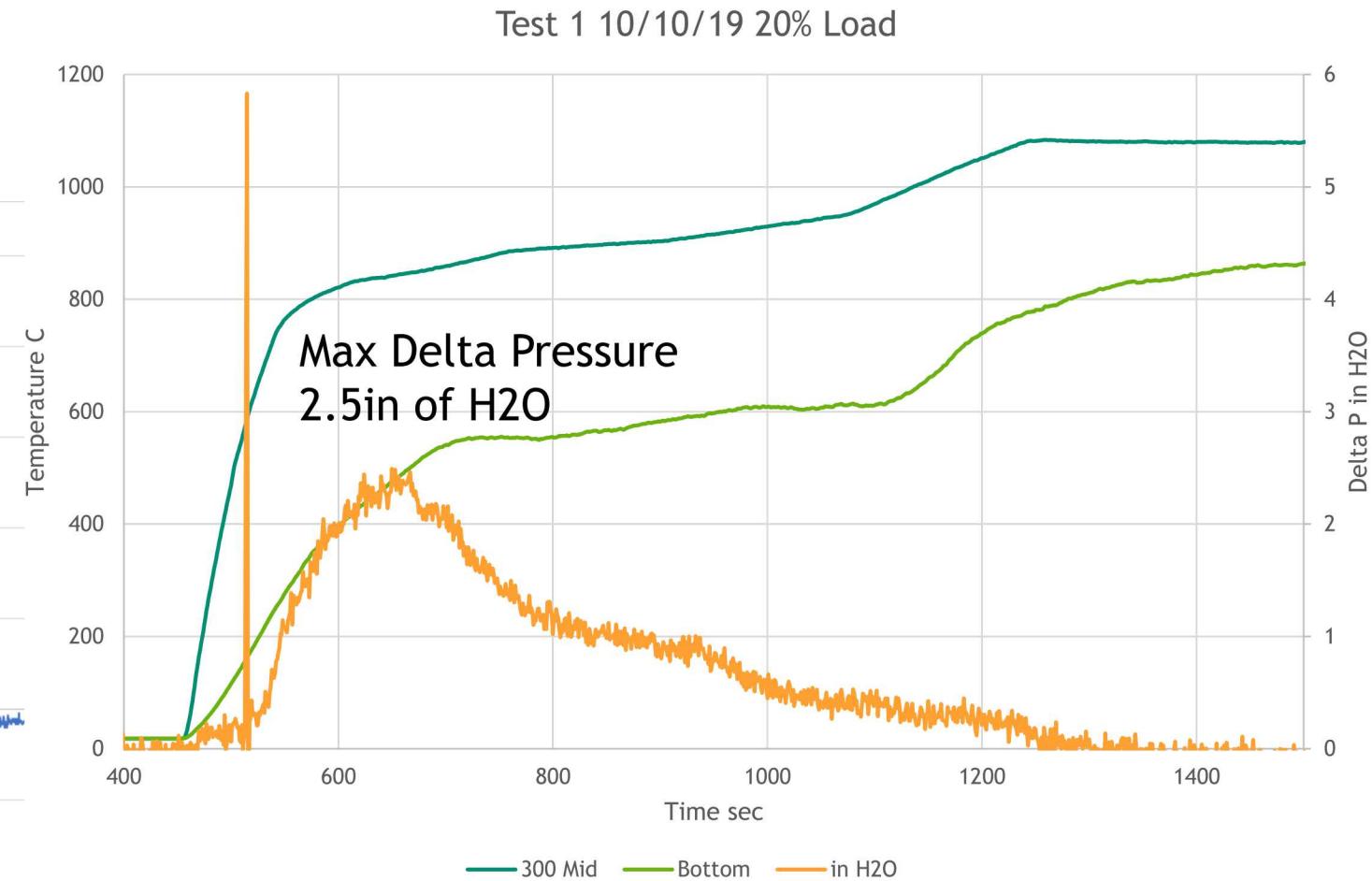
Radiant Heat Test #1 Temperatures and Pressure



- Some discrepancy observed
- Can add more power to the lamps to drive drum pressure up

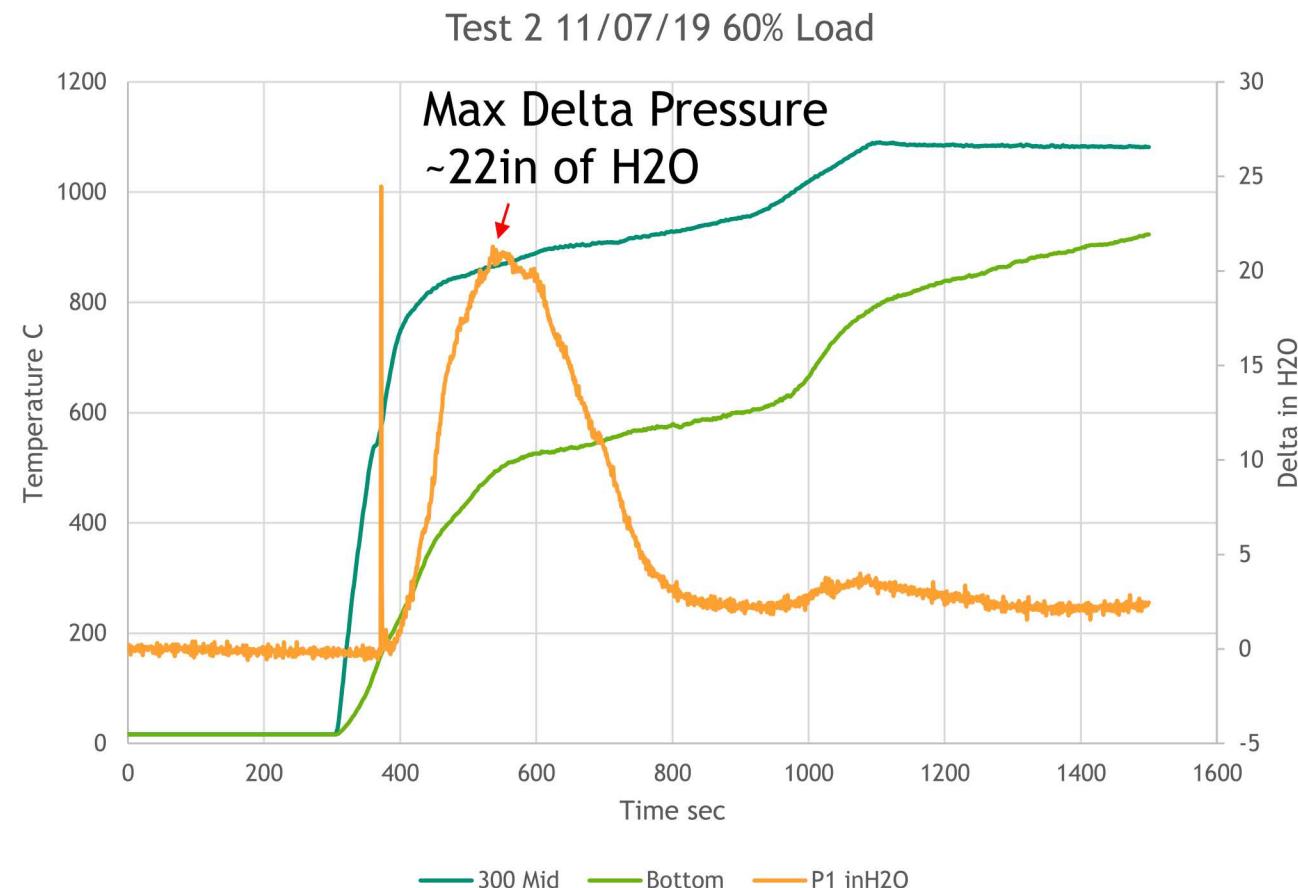
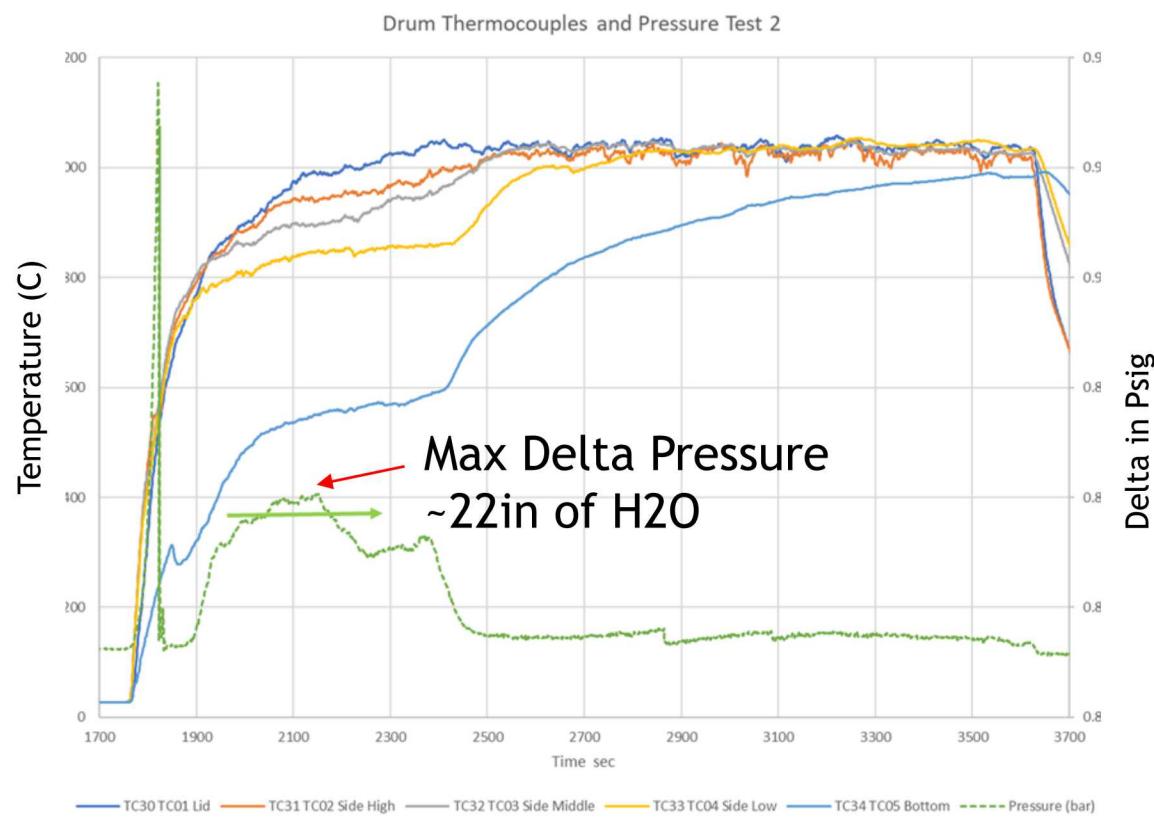


Fire Tests



Radiant Heat Test

Radiant Heat Test #2 Temperatures and Pressure



Able to reproduce peak pressure after filter release



7a Test Campaign

High soot release starting about 5 minutes into the test

Significant mass loss (~55%) in less than 10 minutes into the test



Benchtop Tests to Determine Methodology for Measuring the ARF (3) (Can we determine the ARF from a 7A drum?)

Benchtop Tests

- Small Scale Filter Collection System
 - Collect material release and left in flask to determine ARF via chemical analysis
 - May give an early indication of the ARF expected in large scale test
 - Test spectral system's ability to detect CeO₂ and measure CeO₂ concentrations of materials of interest
- X-ray Fluorescence Spectrometry
 - Huge potential as a diagnostic tool
 - Can be used to detect material in filter or in gas jet if proven to work as claimed in these papers.
 - Can be used in other release scenarios, not just drum fires
 - Some funds already available as part of another project
- Beer-Lambert Infrared Spectrometry
 - Already used at SNL to obtain Al₂O₃ particles concentrations inside a propellant fire
 - Needs accurate particle temperature measurements
 - Looking at using X-ray Fluorescence to determine particle temperatures

Pharm Res (2016) 33:816–825
DOI 10.1007/s11095-015-1828-6



RESEARCH PAPER

Temporally and Spatially Resolved x-ray Fluorescence Measurements of in-situ Drug Concentration in Metered-Dose Inhaler Sprays

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<https://doi.org/10.5194/amt-11-3541-2018>
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ABSTRACT

Purpose Drug concentration measurements are typically performed using metering. These techniques are based on the nozzle, making it difficult to control. nozzle design will affect the precision in solution-based MDIs, and the

Methods In-situ measurement

Atmospheric Measurement Techniques
Open Access

Field and laboratory evaluation of a high time resolution x-ray fluorescence instrument for determining the elemental composition of ambient aerosols

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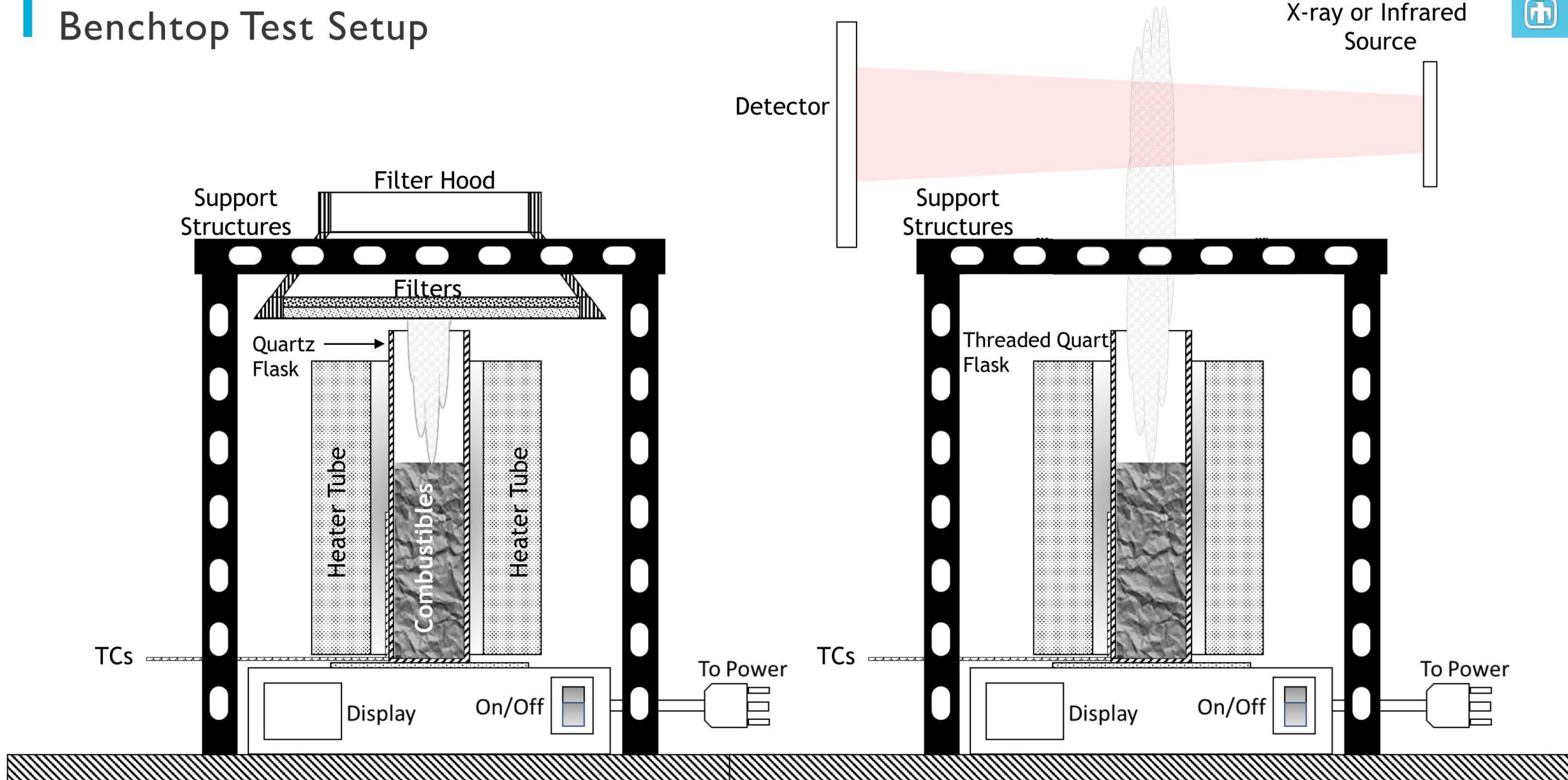
Received: 4 October 2017 – Discussion started: 14 December 2017

Revised: 22 May 2018 – Accepted: 25 May 2018 – Published: 20 June 2018

Abstract. Measuring the chemical composition of airborne particulate matter (PM) can provide valuable information on the concentration of regulated toxic metals, support modelling approaches for source detection and assist in the identification and validation of abatement techniques. Undertaking

2. The XACT was evaluated in three contrasting field deployments; a heavily trafficked roadside site (PM₁₀ and PM_{2.5}), an industrial location downwind of a nickel refinery (PM₁₀) and an urban background location influenced by nearby industries and motorways (PM₁₀). The

Benchtop Test Setup



Determine ARF from CeO₂ released and remaining in the flask → Measure concentration & compare to filter test results

Materials for Benchtop Tests



Thermo F21135 Tubular Furnace

Saint-Gobain Quartz Pre-filter

Hi-Q CFPH-810 8x10" Filter Holder

Combustibles:

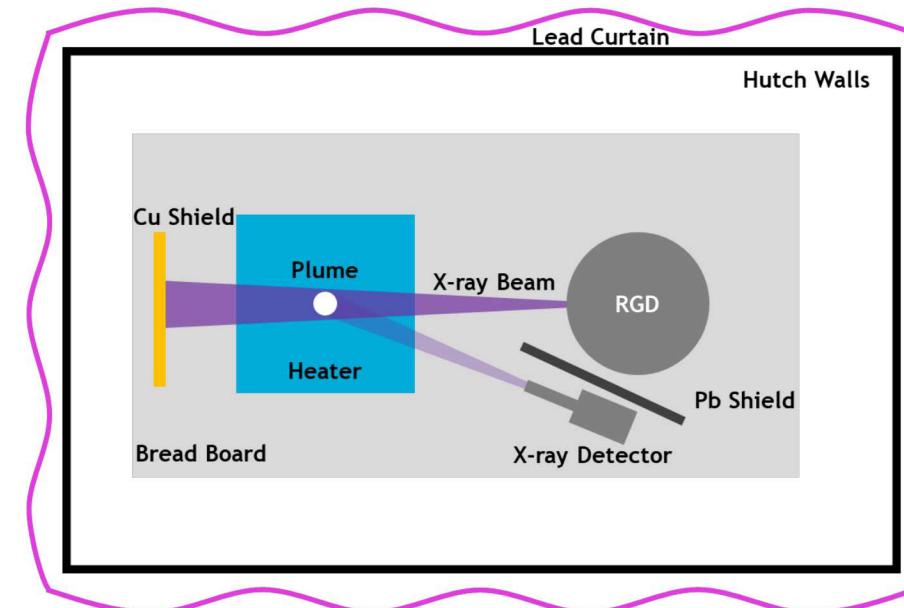
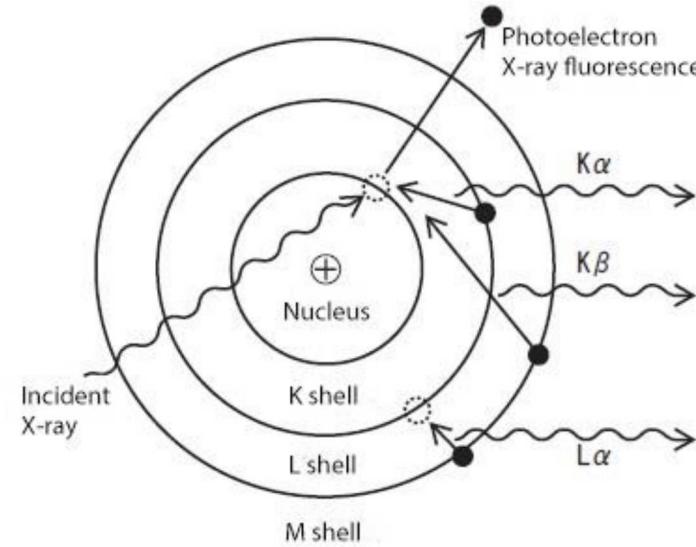
- Cellulose, Plastic, Rubber

Combustion Tube:

- Quartz (ceramic as backup)
- Threaded End for Plastic Cap or Glass cap with Plastic Clip



Can see CeO₂ through Soot with X-rays!

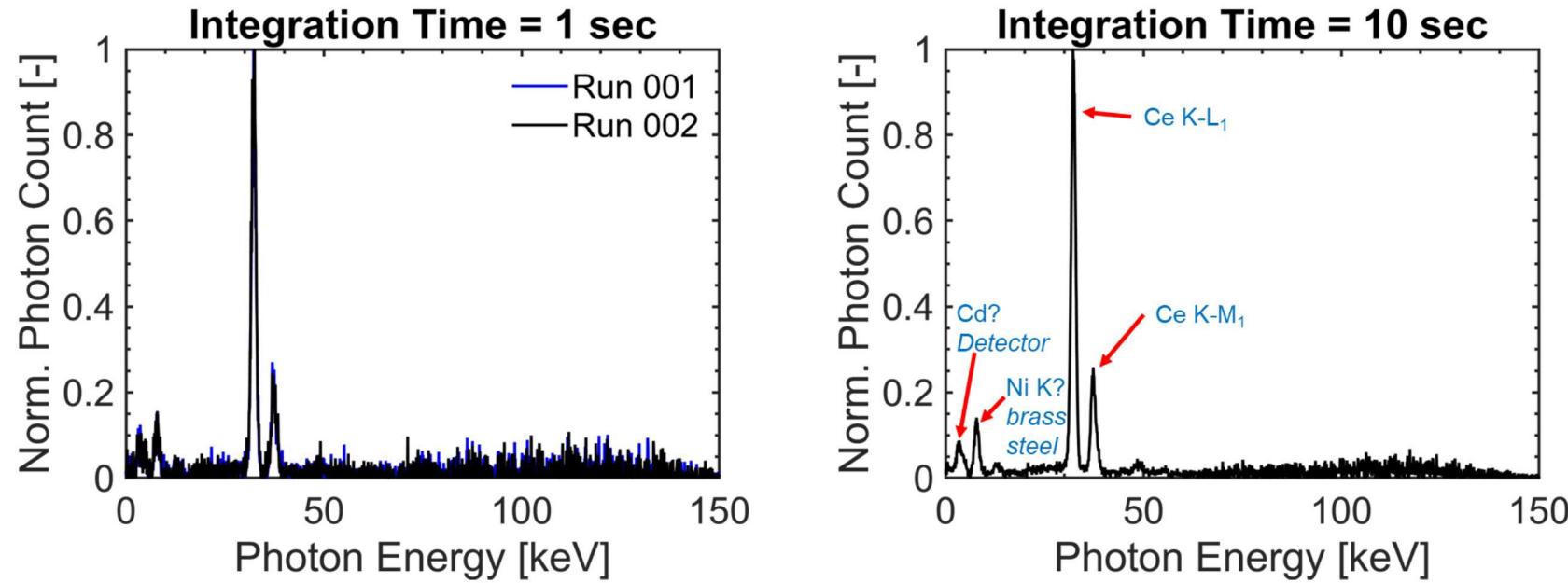


- X-ray fluorescence (XRF) has two steps:
 - An absorbed incident x-ray ejects an electron from the closest shell to the nucleus (K-shell)
 - Electrons from the L and M shells lose energy by photonic emission (fluorescence) to “fall down” into the lower shell

- X-ray source (RGD) generates a beam of x-rays with known energies over the drum, encompassing the plume
- The X-ray detector captures fluorescence from cesium throughout the full test
- Time resolution depends on achieving good Signal to Noise from Ce

Applying XRF to CeO₂

Work not done at SNL, but demonstrates potential of XRF to detect CeO₂ in a fire.



- How does XRF help us see heavy metals?
 - Fluorescence is unique to each element
 - Fluorescence wavelength relates to atomic weight → there is no signal interference from soot (C and H atoms) because they are much lighter than Ce
 - Fluorescence intensity (# of photons emitted) is linearly proportional to the mass of Ce atoms in the test volume
 - **10 second integration time is required to generate good Signal to Noise from 210 mg of Ce**



Thanks



Backup slides

Gas Speed: Test #2



Data can be used for model validation?

