PNNL-21321
WTP-RPT-218, Rev 0

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Fire Safety Tests for Spherical Resorcinol Formaldehyde Resin: Data Summary Report

D Kim
MJ Schweiger
RA Peterson

July 2012

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Fire Safety Tests for Spherical Resorcinol Formaldehyde Resin: Data Summary Report

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Test Specification: 24590 PTF-TSP-RT-09-002, Rev. 0
Test Plan: TP-WTPSP-002, Rev. 3.0
Test Exceptions: 24590-PTF-TEF-RT-11-00003, Rev. 0
R&T Focus Area: Pretreatment
Test Scoping Statement: None

Prepared for the U.S. Department of Energy
Under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by Test Specification 24590-PTF-TSP-RT-09-002, Rev 0, and Test Plan TP-WTPSP-002, Rev. 3.0. The work followed the quality assurance requirements outlined in the test specification and test plan. The descriptions provided in this report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

D E Kurath — 7/18/12

Dean E. Kurath, Manager
WTP R&T Support Project

Date

Testing Summary

A draft safety evaluation of the scenario for spherical resorcinol formaldehyde (SRF) resin fire inside the ion exchange column was performed by the Hanford Tank Waste Treatment and Immobilization Plant (WTP) Fire Safety organization. The result of this draft evaluation suggested a potential change of the fire safety classification for the Cesium Ion Exchange Process System (CXP) emergency elution vessels, equipment, and piping, which may be overly bounding based on the fire performance data from the manufacturer of the ion exchange resin selected for use at the WTP. To help resolve this question, key combustion properties of the SRF resin were measured by Southwest Research Institute (SwRI), following the American Society for Testing and Materials (ASTM) standard procedures, through a subcontract managed by Pacific Northwest National Laboratory (PNNL). For some tests, the ASTM standard procedures were not entirely appropriate or practical for the SRF resin material, so the procedures were modified and deviations from the ASTM standard procedures were noted.

This report summarizes the results of fire safety tests performed and reported by SwRI. The efforts by PNNL were limited to summarizing the test results provided by SwRI into one consolidated data report. All as-received SwRI reports are attached to this report in the Appendix. Where applicable, the precision and bias of each test method, as given by each ASTM standard procedure, are included and compared with the SwRI test results of the SRF resin.

Objectives

The objectives for the SRF resin fire safety tests were

- to determine whether the SRF resin is to ignite and burn at temperatures up to 750°C
- to determine how much soot/smoke will be generated if the SRF resin burns or smolders
- to determine the size of particulates generated if the SRF resin does generate soot/smoke.

Table S.1 provides the objectives that applied to the SRF resin fire safety tests.

Test Exceptions

The test exception in Test Plan TP-WTPSP-002, Rev 3, that was applicable to this testing is presented in Table S.2.

Results and Performance Against Success Criteria

Success criteria for achieving the test objectives are discussed in Table S.3.

Table S.1. Summary of Test Objectives and Results

Test Objective	Objective Met?	Discussion
• Determine whether the SRF resin is to ignite and burn at temperatures up to 750°C.	Yes	The flash ignition temperature (FIT) and spontaneous ignition temperature (SIT) were determined in accordance with ASTM D1929-96. The average values of FIT and SIT from duplicate tests were 595°C and 613°C, respectively. The results are summarized in Section 4.1.
• Determine how much soot/smoke will be generated if the SRF resin burns or smolders.	Yes	The specific smoke extinction area (SEA), which is a ratio of smoke production to specimen mass loss, was measured in accordance with ASTM E1354-10. The average SEA was 13 m^2/kg from duplicate runs. The specific optical density (D_s) of smoke generated from a constant irradiation of 25 kW/m^2 was determined in general accordance with ASTM E662-09 with deviations as described in Section 3.5. The average maximum D_s values were 0.65 under flaming condition and 0.35 under nonflaming condition obtained from duplicate runs. The results are summarized in Section 4.4.
• Determine the size of particulates generated if the SRF resin does generate soot/smoke.	Yes	The particle size distribution of smoke generated from a constant irradiation of 25 kW/m^2 was determined during smoke evaluation tests that were performed in general accordance with ASTM E662-09 (no applicable standard procedures for smoke particle size determination). The median diameter (d_{50}) was 2.4 µm under nonflaming condition and 4.0 µm under flaming condition. Under nonflaming condition, the largest particle was 7.2 µm and about 1.5% of the particles were smaller than 0.52 µm. Under flaming condition, the largest particle was 11.1 µm and only about 0.3% of the particles were smaller than 0.52 µm.

Table S.2. Test Exception

Test Exception Number	Description of Test Exception
24590-PTF-TEF-RT-11-00003, Rev 0	This test exception was received from Bechtel National, Inc. (BNI) on September 9, 2011. It incorporates the approved Request for Technology Development (RTD), 24590-WTP-RTD-RT-11-0001, Rev 0, which requests data on the fire safety limits for soot/particulate generation and ignition temperature for the SRF ion exchange resin.

Table S.3. Success Criteria for Achieving Test Objectives

List Success Criteria	Explain How the Tests Did or Did Not Meet the Success Criteria
1. Provide test results for high-temperature analysis of resin to determine flash ignition/spontaneous ignition temperatures.	This success criterion was met. The flash ignition and spontaneous ignition temperatures were measured in accordance with ASTM D1929-96. The flash ignition temperature was 595°C, and spontaneous ignition temperature was 613°C.
2. Provide test results that include heat release rates and mass/soot/smoke yield if SRF resin were to burn or smolder.	This success criterion was met. The heat release rates and specific smoke extinction area were measured in accordance with ASTM E1354-10 and ASTM D7309-07A. The peak heat release rate was 52 kW/m^2, and the maximum specific heat release rate was 21.6 W/g in an oxygen-free environment and 124.6 W/g in an oxidizing environment. The specific smoke extinction area was 13 m^2/kg. The specific optical density of smoke generated from a constant irradiation of 25 kW/m^2 was determined in general accordance with ASTM E662-09 with deviations as described in Section 3.5. The average maximum specific optical density was 0.65 under a flaming condition and 0.35 under a nonflaming condition.
3. Provide particle size distribution analysis of soot generated by SRF resin.	This success criterion was met. The particle size distribution of the smoke was determined in general accordance with ASTM E662-09 (no applicable standard procedures for smoke particle size determination). The median diameter (d_{50}) was 2.4 µm under nonflaming condition and 4.0 µm under flaming condition. Under nonflaming condition, the largest particle was 7.2 µm and about 1.5% of the particles were smaller than 0.52 µm. Under flaming condition, the largest particle was 11.1 µm and only about 0.3% of the particles were smaller than 0.52 µm.

Quality Requirements

The PNNL Quality Assurance (QA) Program is based on the requirements defined in U.S. Department of Energy Order 414.1D, *Quality Assurance,* and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A – Quality Assurance Requirements (a.k.a. the Quality Rule). PNNL has chosen to implement the following consensus standards in a graded approach:

- ASME NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, Part I, Requirements for Quality Assurance Programs for Nuclear Facilities
- ASME NQA-1-2000, Part II, Subpart 2.7, Quality Assurance Requirements for Computer Software for Nuclear Facility Applications
- ASME NQA-1-2000, Part IV, Subpart 4.2, Graded Approach Application of Quality Assurance Requirements for Research and Development.

The Waste Treatment Plant Support Project (WTPSP) implements an NQA-1-2000 Quality Assurance Program, graded on the approach presented in NQA-1-2000, Part IV, Subpart 4.2. The WTPSP Quality Assurance Manual (QA-WTPSP-0002) describes the technology life-cycle stages under the WTPSP Quality Assurance Plan (QA-WTPSP-0001). The technology life cycle includes the progression of technology development, commercialization, and retirement in process phases of basic and applied research and development (R&D), engineering and production, and operation until process completion. The life cycle is characterized by flexible and informal quality assurance activities in basic research that become more structured and formalized through the applied R&D stages.

The work described in this report has been completed under the QA technology level of Applied Research. The WTPSP addresses internal verification and validation activities by conducting an independent technical review of the final data report in accordance with WTPSP procedure QA-WTPSP-601, Document Preparation and Change. This review verifies that the reported results are traceable, that inferences and conclusions are soundly based, and that the reported work satisfies the test plan objectives.

The Southwest Research Institute, who performed the tests described in this report, is listed on the PNNL Approved Suppliers List.

Test Conditions

This report summarizes the fire test results performed by SwRI and submitted to PNNL.

All test conditions delineated by the test plan and test exceptions were met. A summary of test conditions is provided in Table S.4.

Simulant Use

Simulant was not developed for the tests summarized in this report. The SRF resin was manufactured by Microbeads AS, a Norwegian company (www.micro-beads.com), and shipped in an approximately100-L steel drum. WTP provided the entire drum to PNNL. About 20 L of the resin were shipped to SwRI in a 20-L carboy.

Table S.4. Test-Condition Summary

Test Conditions	Were Test Conditions Followed?
1. Fire safety testing will also be performed to obtain data on potential soot and combustion byproducts generated by an ion exchange resin fire to assist the WTP Project in assessing off-gas filtration requirements. An independent laboratory will conduct tests using WTP-supplied SRF resin. The testing will include standard small-scale fire behavior characterization testing according to ASTM E1354-10, *Standard Test Method for Heat and Visible Smoke Release Rates Using an Oxygen Consumption Calorimeter*; ASTM D1929-96, *Standard Test Method for Determining Ignition Temperature;* ASTM D7309-07A, *Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry*; ASTM E800-07, *Standard Measurement of Gasses Present Or Generated During Fires Using Gas Sampling Protocol*; and ASTM E662-09, *Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials*.	Fire safety testing was performed by an independent laboratory, Southwest Research Institute. The WTP-supplied SRF resin was filtered and then dried for 2 to 12 h depending on the amount of material required for each test at 70°C before testing. All tests were performed in general accordance with the standard procedures: ASTM E1354-10, ASTM D1929-96, ASTM D7309-07A, ASTM E800-07, and ASTM E662-09, except for the deviations from ASTM E662-09 in the following items: 1) furnace, 2) pilot burner, and 3) drying condition (see Section 3.5 for detailed descriptions and Table 3.1 for a summary).

Discrepancies and Follow-On Tests

Large differences in CO and CO_2 concentrations were observed between duplicate tests (Table 4.8). SwRI performed careful review of the raw data files to investigate the potential cause of these large differences and did not find any indication that the tests were done incorrectly. It is suspected that the large differences are likely due to the characteristics inherent to the present SRF resin material. It is suggested that the gas analyses be repeated so that the assumption on the characteristics of the SRF resin may be checked.

Acknowledgments

The authors thank the Southwest Research Institute (Eugene Horton and SwRI staff, San Antonio, Texas) for test support. The authors also thank Jennifer Meehan and David Sherwood of the Hanford Tank Waste Treatment and Immobilization Plant project for their technical insights and much helpful discussion and support. The authors are grateful for the assistance of Renee Russell for her technical review. In addition, the authors thank Casey D. Emery, PNNL Contracts Department, for his diligent effort in negotiating this contract.

Acronyms and Abbreviations

ASTM	American Society for Testing and Materials
BNI	Bechtel National, Inc.
CHF	critical heat flux
CXP	Cesium Ion Exchange Process System
DOE	U.S. Department of Energy
EHC	effective heat of combustion
FIT	flash ignition temperature
FPI	fire propagation index
FTIR	Fourier transform infrared
GGRF	ground gel resorcinol-formaldehyde
HRR	heat release rate
HRR_{peak}	peak heat release rate
ISO	International Organization for Standardization
MLR	mass loss rate
ORP	Office of River Protection
PMMA	poly methyl methacrylate
PNNL	Pacific Northwest National Laboratory
PTF	pretreatment facility
QA	quality assurance
R&D	research and development
RF	resorcinol formaldehyde
RTD	request for technology development
SEA	specific smoke extinction area
SIT	spontaneous ignition temperature
SRF	spherical resorcinol formaldehyde
SwRI	Southwest Research Institute
THR	total heat release
TRP	thermal response parameter
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WTPSP	Waste Treatment Plant Support Project

Symbols

A	exposed area of test sample
a, b	linear regression coefficients for repeatability or reproducibility
d_{50}	median diameter
D_m	mass optical density of smoke
D_s	specific optical density of smoke
h_c	specific heat release
$h_{c,gas}$	specific heat of combustion of specimen gases
I	intensity of light transmitted through smoke
I^0	intensity of incident light
K	light extinction coefficient
k_c	convection coefficient
L	light path length
Δm	mass loss
Δm_s	mass loss per unit sample surface area
Q_{max}	maximum specific heat release rate
r	repeatability limit
R	reproducibility limit
S_A	total smoke production per unit sample area
s_r	standard deviation for repeatability limit (r)
s_R	standard deviation for reproducibility limit (R)
T_{ig}	surface temperature at ignition
t_{ig}	time to ignition
t_T	total test duration
T_{max}	heat release temperature
T_∞	ambient temperature
V	volume of test chamber
$\dot{V}$	volume flow rate of exhaust at the location of the light extinction measurement
Y_p	pyrolysis residue
ε	surface emissivity
η	heat release capacity
σ	Stefan–Boltzmann constant

Contents

Figures

Tables

1.0 Introduction

Ion exchange using the spherical resorcinol-formaldehyde (SRF) resin has been selected by Bechtel National, Inc. (BNI) and approved by the U.S. Department of Energy (DOE) Office of River Protection (ORP) for use in the Pretreatment Facility (PTF) of the Hanford Tank Waste Treatment and Immobilization Plant (WTP). SRF is an engineered spherical form of the older ground gel resorcinol-formaldehyde (GGRF) resin, also termed resorcinol-formaldehyde (RF).

A draft safety evaluation of the scenario for resin fire inside the ion exchange column was performed by the WTP Fire Safety organization. The result of this draft evaluation suggested a potential change of the fire safety classification for the Cesium Ion Exchange Process System (CXP) emergency elution vessels, equipment, and piping. To help resolve this question, key combustion properties of the SRF resin were measured by Southwest Research Institute (SwRI), following the standard procedures, through a subcontract managed by Pacific Northwest National Laboratory (PNNL).

Section 2.0 details the basis of the PNNL Quality Assurance (QA) Program as applied to the WTP quality requirements. PNNL QA program was flowed down to SwRI. Section 3.0 describes the test methods and ASTM standard procedures used in this testing. Section 4.0 summarizes the results of the experimental tests performed by SwRI. Section 5.0 provides all test results summarized in one table. Section 6.0 provides a list of pertinent references. All as-received SwRI reports are included in the Appendix. Some errors found in the as-received SwRI reports were corrected and additional data that were missing in the as-received reports were added to this summary report.

2.0 Quality Assurance

The PNNL Quality Assurance (QA) Program is based on the requirements defined in U.S. Department of Energy Order 414.1D, *Quality Assurance,* and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A – Quality Assurance Requirements (a.k.a. the Quality Rule). PNNL has chosen to implement the following consensus standards in a graded approach:

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The Southwest Research Institute, who performed the tests described in this report, is listed on the PNNL Approved Suppliers List.

3.0 Experimental

This section describes the test methods and procedures used by SwRI to perform various fire safety tests on the SRF resin provided by PNNL. Table 3.1, presented at the end of this section, summarizes the ASTM standard test procedures used by SwRI for this testing.

All tests were performed in duplicate. For cone calorimeter, microscale combustion calorimeter, and smoke density tests, each run consisted of three tests (total six tests) as required by ASTM standards.

3.1 Sample Preparation

PNNL provided SwRI with one 20-L carboy of SRF resin that consisted of a micro bead resin material dispersed in water. For all tests described in this report, the sample was filtered and then dried for 2 to 12 h depending on the amount of material required for each test in an oven at 70°C prior to testing. This temperature was chosen not to over-dry the resin. The resin was constantly monitored to achieve a consistency in drying condition gauged by the color. After drying, the sample consisted of only the micro bead material. The beads became statically charged in the absence of the water medium, so they were placed in a closed container after drying in an effort not to lose any beads. Each sample was then stored in a controlled environment maintained at 23°C ± 3°C and 50% ± 5% relative humidity until just prior to testing.

3.2 Flash Ignition Temperature and Spontaneous Ignition Temperature Using Hot-Air Furnace

The flash ignition temperature (FIT) and spontaneous ignition temperature (SIT) of SRF resin were determined using a hot-air furnace according to ASTM D1929-96, *Standard Test Method for Determining Ignition Temperature*. The hot-air ignition furnace consists primarily of an electrical heating unit and specimen holder. The furnace tube is a vertical tube with an inside diameter of 100 ± 5 mm and a length of 230 ± 20 mm, made of ceramic that will withstand at least 750°C. The inner ceramic tube, with an inside diameter of 75 ± 5 mm, a length of 230 ± 20 mm, and a thickness of approximately 3 mm, is placed inside the furnace tube and positioned 20 ± 2 mm above the furnace floor on spacer blocks. The pilot flame is located immediately above the opening. The test apparatus is shown in Figure 3.1.

The FIT is the minimum temperature at which, under specified test conditions, sufficient flammable gases are emitted to ignite momentarily upon application of a small external pilot flame. The lowest air temperature at which a flash is observed during a 10-min period is recorded as the FIT.

The SIT is the minimum temperature at which the self-heating properties of the specimen lead to ignition or ignition occurs spontaneously, under specified test conditions, in the absence of any additional flame ignition source. The lowest air temperature at which the specimen ignites during a 10-min period is recorded as the SIT.

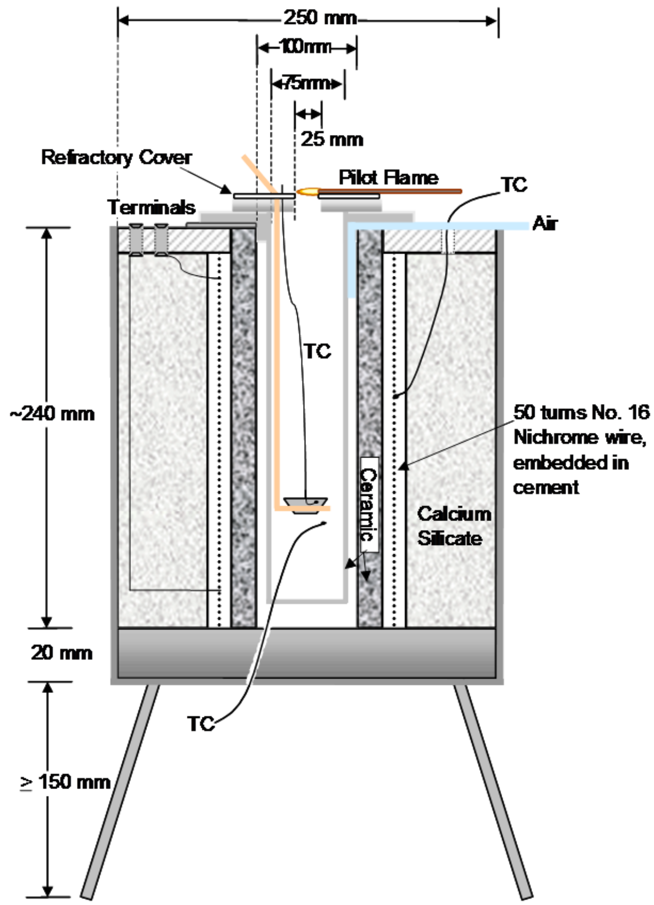

Figure 3.1. Schematic of Southwest Research Institute Hot-Air Furnace (from SwRI report for R13295.12.008a)

3.3 Cone Calorimeter Tests

Heat release rate (HRR, kW/m^2), mass loss rate (MLR, g/[m^2s]), effective heat of combustion (EHC, MJ/kg), and specific smoke extinction area (SEA, m^2/kg) of SRF resin were determined using a cone calorimeter according to ASTM E1354-10, *Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter.*

The cone calorimeter is a small-scale test apparatus that measures primarily the rate of heat release of materials and products under a wide range of conditions using the oxygen consumption technique. A schematic of the cone calorimeter apparatus is provided in Figure 3.2. The cone calorimeter used for tests at SwRI is shown in Figure 3.3.

For organic solids, liquids, and gases, a nearly constant net amount of heat is released per unit mass of oxygen consumed for complete combustion. An average value for this constant of 13.1 MJ/kg of oxygen can be used for practical applications and is accurate with very few exceptions to within ± 5%. Therefore, measurements of the oxygen consumed in a combustion system can be used to determine the net heat released. This technique, generally referred to as the "oxygen consumption technique," is now the most widely used and accurate method for measuring heat release rate in experimental fires.

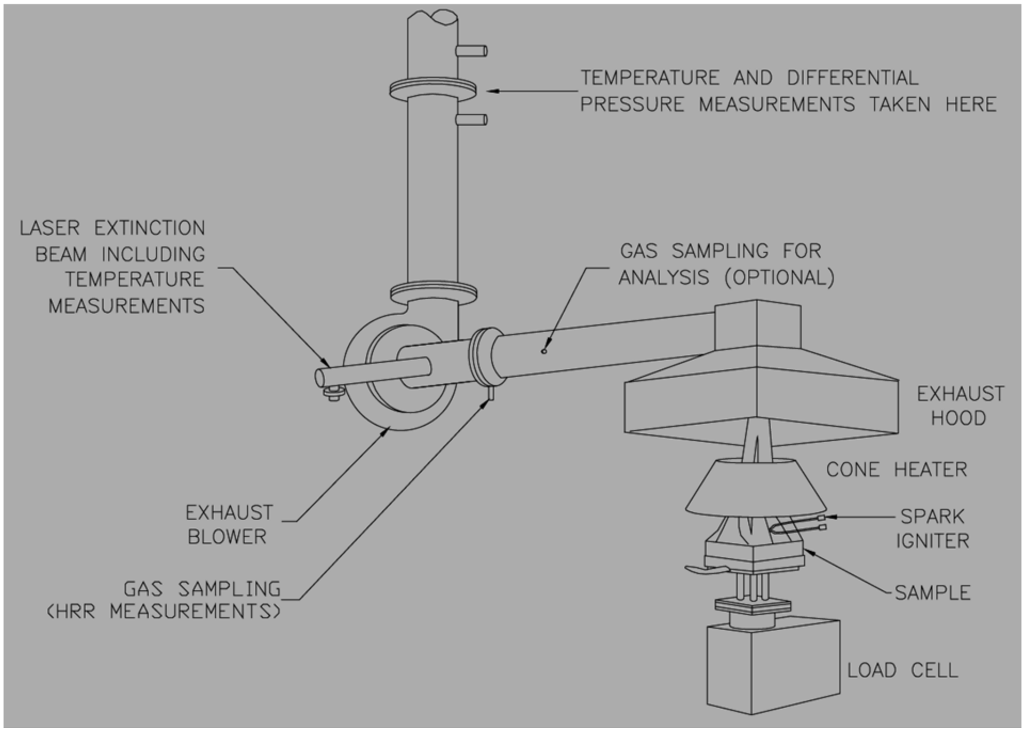

Figure 3.2. Schematic of Cone Calorimeter Apparatus (from SwRI report for R13295.12.008b)

Figure 3.3. Cone Calorimeter Apparatus at SwRI

In the cone calorimeter, a square sample 100 × 100 mm (4 × 4 in.) is exposed to the radiant flux of an electric heater. The heater has the shape of a truncated cone (hence the name of the instrument) and is capable of providing heat fluxes to the specimen in the range of 0–110 kW/m^2. An electric spark igniter is used for piloted ignition of the pyrolysis gases produced by the radiant heater.

Prior to testing, the test specimen is wrapped in aluminum foil, backed with a layer of low-density refractory fiber blanket, and placed in a standard specimen holder. At the start of a test, the specimen in the appropriate holder is placed on the load cell located below the heater. The top edge of the specimen is positioned 25 mm below the base plate of the heater. The electric spark igniter is located 13 mm above the center of the specimen. The electric spark igniter is removed 4 s after the pyrolysis gases are released by the specimen ignition.

The products of combustion and entrained air are collected in a hood and extracted through an exhaust duct by a fan. A gas sample is drawn from the exhaust duct and analyzed for oxygen concentration. The gas temperature and differential pressure across an orifice plate are used for calculating the mass flow rate of the exhaust gases. Smoke production is determined based on the measured light obscuration in the duct using a laser photometer located close to the gas sampling point.

3.4 Microscale Combustion Calorimeter

Specific heat release rate of the SRF resin was determined using a microscale combustion calorimeter according to ASTM D7309-07A, *Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry*.

Similar to a cone calorimeter, a microscale calorimeter is a small-scale test apparatus that also uses the oxygen consumption technique to measure the rate of heat release of materials and products. The ASTM D7309-07A apparatus (see Figure 3.4) consists primarily of two heating chambers and a specimen holder (a part of the specimen thermocouple post). The heating chambers are two sections of a vertical tube. The sample sits in the pyrolysing chamber, and the pyrolysis gases move up toward the combustor furnace.

The combustor furnace was set at 900°C. The pyrolysis chamber was set at 75°C and was ramped up to 850°C at a specified heating rate of 1 K/s. The ASTM D7309-07A standard specifies two different test methods. Method A is controlled thermal decomposition, and Method B is controlled thermal oxidative decomposition. The primary difference between the two methods is the point at which the oxygen is introduced. In Method A, the purge gas nitrogen flows through the pyrolyser section, then the nitrogen/oxygen mixture, along with any pyrolysis gases emitted from the sample, travel through the combustor furnace. In this method, the heat of combustion of the volatile components (gases) is measured, but the heat of combustion of any solid residue is not. In Method B, the nitrogen/oxygen mixture is introduced into the pyrolyser section. In this method, the net calorific value of the specimen gases and solid residue are measured. Because Method A can be used to obtain the heat release capacity η_c (J/g-K), which is a flammability parameter unique to this test method, Method A is typically used. The heat release rate is calculated based on oxygen consumption, and the specimen mass loss is determined based on weight measurements before and after testing.

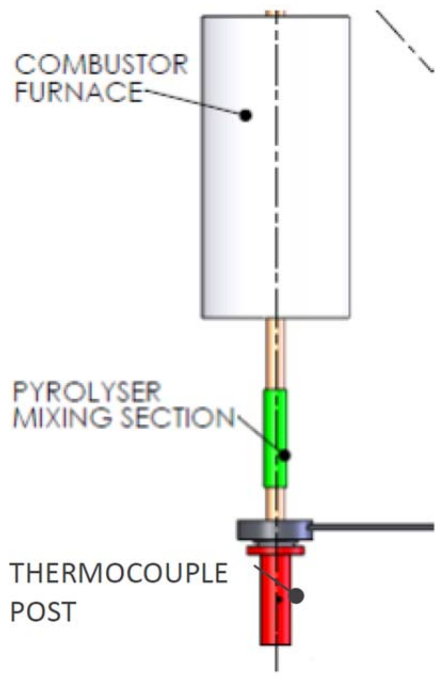

Figure 3.4. Schematic of Microscale Combustion Calorimeter (from SwRI report for R13295.12.008d)

3.5 Smoke Evaluation and Gas Analysis

Specific optical density of smoke was determined in general accordance with ASTM E662-09, *Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials*. The gas analysis was performed using the Fourier transform infrared (FTIR) spectroscopy according to ASTM E800-07, *Standard Guide for Measurement of Gases Present or Generated During Fires*. Because there is no applicable standard procedure, the particle size distribution was measured following the same procedure used for specific optical density determination.

A radiant heat furnace was used to provide a constant irradiance of 25 kW/m^2 in two exposures, flaming and nonflaming. A nonflaming exposure was used to determine smoke particle size distribution. Specific optical density measurement of smoke was performed under both flaming and nonflaming exposures. The furnace was not the same as that prescribed in procedure ASTM E662. Because the material could not sit in the sample holder in a vertical orientation, the International Organization for Standardization (ISO) 5659 furnace (a radiator cone) was used. The ISO 5659 is a similar smoke generation test which is typically run in the horizontal orientation. A pilot flame is used during flaming exposure and is centered in front of and parallel to the specimen holder. The pilot flame is also different from that prescribed in the ASTM E662-09 procedure, which is a six-flamed pilot burner. Instead, the ISO 5659 single-flame pilot burner was used because this ISO 5659 burner is more appropriate for the present tests using the ISO 5659 furnace.

ASTM E662-09 standard requires samples to be dried for 24 h at 60°C ± 3°C (140°F ± 5°F) prior to testing. However, the pre-drying condition described in Section 3.1 was already selected as the best condition for the present material and therefore it was determined that a 24 h drying is not necessary. Test specimens measuring 3 × 3 in., +0, −0.03 in. (76.2 × 76.2 mm, +0, −0.8 mm) were covered across the back and along the edges with a single sheet of aluminum foil with the dull side in contact with the specimen.

Specific optical density measurement was conducted for 10 min unless a minimum light transmittance value had not yet occurred, in which case the test was continued for an additional 10 min. Notes were made relevant to any burning characteristics such as delamination, intumescence, shrinkage, and ignition. Test runs were carried out with a chamber wall temperature of 35°C ± 2°C (95°F ± 4°F). The tests for smoke particle size distribution were conducted for 20 min.

Specific optical density measurement was made based on the attenuation of a vertically oriented light beam through smoke accumulating within the chamber. Results are expressed in terms of the dimensionless specific optical density, D_s. A clear beam value was recorded upon evacuation of smoke from the box to determine a correction factor. The maximum corrected specific optical density value from each of the runs at a single exposure condition were averaged and reported. Per the standard procedure, smoke density values were rounded to two significant figures.

Gas analysis was performed during the third measurement for specific optical density in flaming and nonflaming exposures using an FTIR spectrometer. The sample was drawn through a preheated line to prevent condensation and adsorption and then filtered into a preheated gas cell. Analysis was conducted for CO, CO_2, HCl, HCN, HF, HBr, SO_2, and NOx.

As described above, for specific optical density measurement and gas analysis it was necessary to modify the ASTM E662-09 standard and follow the ISO 5659 standard for the furnace and pilot burner because of the nature of the present SRF resin material. However, these deviations are expected to have little impacts on the test results because both are in general similar test methods.

Table 3.1. Standard Procedures Used by Southwest Research Institute

Main Analysis/Property	Test Equipment(s)	Standard Procedures used by SwRI	Deviation from Standard Procedure?	SWRI Report No.
Flash ignition temperature (FIT) and spontaneous ignition temperature (SIT)	Hot-air ignition furnace	ASTM D1929-96, *Standard Test Method for Determining Ignition Temperature of Plastics*	No	R13295.12.008a
Heat release rates (kW/m^2), mass loss rate ($g/[m^2s]$), effective heat of combustion (MJ/kg), specific smoke extinction area (m^2/kg) critical heat flux (kW/m^2) for ignition[a] and fire propagation index (FPI, $m^{5/3}/[kW^{2/3}s^{1/2}]$)[a]	Cone calorimeter using oxygen consumption technique	ASTM E1354-10, *Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter*	No	R13295.12.008b R13295.12.008c
Heat release rate given by maximum specific heat release rate (Q_{max}, W/g) and effective heat of combustion given by specific heat release of sample (h_c, J/g)	Microscale combustion calorimeter using oxygen consumption technique	ASTM D7309-07A, *Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry*	No	R13295.12.008d
Maximum specific optical density (D_s, dimensionless), and maximum gas concentrations (ppm)	Radiant cone heat furnace Fourier transform infrared spectrometer	ASTM E662-09[b], *Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials* ASTM E800-07[b], *Standard Guide for Measurement of Gases Present or Generated During Fires*	Furnace[c] Pilot burner[d] Pre-drying[e]	R13295.12.008f
Particle size distribution of generated smoke	Radiant cone heat furnace	Not applicable[f]	Not applicable	R13295.12.008e

(a) Calculated from data given in R13295.12.008b and R13295.12.008c.
(b) In general accordance with the standard procedures. Any changes made in a standard testing procedure are done to customize a test to the type of material being tested in unusual circumstances. These changes are done in a manner that would not impact the results of a test in an unanticipated way.
(c) ISO 5659 furnace run in the horizontal orientation was used because the material could not sit in the sample holder in a vertical orientation of the ASTM E662-09 furnace.
(d) ISO 5659 single-flame pilot burner was used instead of ASTM E662-09 six-flamed pilot burner because this ISO 5659 burner is more appropriate for the present tests using the ISO 5659 furnace.
(e) Pre-dried for 2 to 12 h at 70°C prior to testing instead of ASTM E662-09 standard of 24 h pre-drying at 60°C because the 24 h pre-drying would be an over drying for this material, which will adversely affect the test results. The 2 to 12 h drying at 70°C was already selected as the best condition for this material.
(f) There is no applicable standard procedure.

4.0 Results and Discussion

The results of the SRF resin fire tests reported by SwRI are summarized in this section. Where applicable, the precision and bias of each test method as given by each ASTM procedure are included. The precision and bias measures used in ASTM standards are defined as follows, per ASTM E177-10, *Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods*:

- *precision*: the closeness of agreement between independent test results obtained under stipulated conditions
- *bias*: the difference between the expectation of the test results and an accepted reference value
- *repeatability*: precision under repeatability conditions
- *repeatability conditions*: conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time
- *repeatability limit (r)*: the value below which the absolute difference between two individual test results obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95%)
- *reproducibility*: precision under reproducibility conditions
- *reproducibility conditions*: conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment
- *reproducibility limit (R)*: the value below which the absolute difference between two test results obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95%).

4.1 Flash Ignition Temperature and Spontaneous Ignition Temperature

The results of FIT and SIT measurements of the SRF resin and negative and positive standards are summarized in Table 4.1. The negative test was performed on marinite, which is a non-combustible material and positive test was performed on poly methyl methacrylate (PMMA). Table 4.1 also includes the repeatability and reproducibility ranges determined from interlaboratory tests involving seven laboratories, on six polymeric materials, with triplicate tests of each material (ASTM D1929-96). The difference between two tests in this study (10°C for FIT and 5°C for SIT) is comparable to the repeatability limit for FIT and significantly smaller than the repeatability limit for SIT. It should be noted that the ASTM precision estimate is for the difference between averages determined from triplicate tests, whereas the difference reported in this study is based on one test per each run. Information on bias of this test method is not yet available.

Table 4.1. Flash Ignition Temperature and Spontaneous Ignition Temperature of SRF Resin

Property	FIT (°C)	SIT (°C)
Initial Run	600	615
Duplicate Run	590	610
Average	595	613
Difference	10	5
Precision estimates[(a)]		
r	8–13	11–31
R	27–117	47–103
Negative and positive standards		
Marinite (negative standard)	NM	No ignition
PMMA (positive standard)	NM	420

(a) Determined from interlaboratory tests involving seven laboratories on six polymeric materials with triplicate tests (ASTM D1929-96).
NM: not measured.

4.2 Cone Calorimeter Results

Table 4.2 shows the results of cone calorimeter tests on SRF resin and negative (marinite) and positive (PMMA) standards according to standard procedure ASTM E1354-10 for the following properties:

- peak heat release rate (HRRpeak, kW/m^2): the maximum value of the heat release rate per unit sample area
- time to ignition (t_{ig}, s): time to ignition/sustained flaming (flame over specimen surface for at least 4 s)
- total heat release (THR, MJ/m^2): total amount of heat released per unit sample area
- mass loss (g/m^2): total mass loss per unit sample area
- mass loss rate (MLR, $g/[m^2s]$): average specimen mass loss rate per unit sample area computed over the test duration
- effective heat of combustion (EHC, MJ/kg): effective heat of combustion (the ratio of heat release rate to mass loss rate) averaged over the test duration or the entire test if ignition does not occur
- total smoke production (S_A, m^2/m^2): total smoke production per unit sample area during the test duration
- smoke extinction area (SEA, m^2/kg): specific smoke extinction area (the ratio of smoke production to specimen mass loss) averaged over the test duration.

Table 4.2. Results from Cone Calorimeter Tests of SRF Resin Performed at 100 kW/m^2 Heat Flux

Property	HRR_{peak} (kW/m^2)	t_{ig} (s)	THR (MJ/m^2)	Mass loss (g/m^2)	MLR (g/[m^{2}s])	EHC (MJ/kg)
Initial run[(a)]	50	42	122.6	9810	2.7	12.5
Duplicate run[(a)]	53	28	124.1	8782	2.5	14.1
Average	51	35	123.4	9296	2.6	13.3
Difference	3	14	1.5	1028	0.2	1.6
Precision estimates[(b)]						
r	20.0	8.5	15.8	NA	NA	1.9
R	67.7	15.1	22.7	NA	NA	3.2
Negative and positive standards[(c)]						
Marinite (negative standard)	5	NI	0.2	465	0.2	0.3
PMMA (positive standard)	819	23	414.1	19131	30.7	21.6

(a) Average of triplicate results.
(b) Based on interlaboratory tests involving six laboratories on six different materials (ASTM E1354-10).
(c) Tested at 50 kW/m^2 heat flux.
NA not available.
NI no ignition.

Property	S_A (m^2/m^2)	SEA (m^2/kg)
Initial run[(a)]	76	8
Duplicate run[(a)]	159	18
Average	118	13
Difference	83	10
Precision estimates[(b)]		
r	NA	60.0
R	NA	65.8
Negative and positive standards[(c)]		
Marinite (negative standard)	42	91
PMMA (positive standard)	1377	72

(a) Average of triplicate results.
(b) Based on interlaboratory tests involving six laboratories on six different materials (ASTM E1354-10).
(c) Tested at 50 kW/m^2 heat flux.
NA not available.

Table 4.2 also includes the repeatability limit and reproducibility limit values related to the precision of the cone calorimeter test method. The ASTM E1354-10 procedure was conducted in interlaboratory trials involving six laboratories on six different materials. The *r* and *R* values were calculated from sample-based standard deviation estimates for repeatability (s_r) and for reproducibility (s_R) using the relations:

$$r = 2.8 s_r \quad (1)$$

$$R = 2.8 s_R \quad (2)$$

The resulting r or R values then were used to fit linear regression models in the form

$$y = a + bx \quad (3)$$

where y is r or R for each property, x is the average of measured value for each property, and a and b are linear regression coefficients. The values for each property were calculated from the coefficient values given in ASTM E1354-10, and the average measured values in this study are given in Table 4.2.

The differences from duplicate tests in this study are, in general, comparable to or significantly smaller than the corresponding repeatability limits except for the time to ignition (t_{ig}). The difference from duplicate tests for t_{ig} (14 s) in this study is larger than the repeatability limit (8.5 s), which is understandable considering the difference in the heat flux values used: this study used 100 kW/m^2 whereas the ASTM interlaboratory studies used 25 and 50 kW/m^2.

For an estimated bias, ASTM E1354-10 states that the use of the oxygen consumption technique results in an expected error band of ± 5% compared to the true value.

Additional tests were performed at varied heat fluxes from 40 kW/m^2 through 80 kW/m^2, in addition to the tests at 100 kW/m^2 summarized in Table 4.2, to measure the surface temperature at ignition (T_{ig}) needed to calculate the critical heat flux (CHF) using

$$CHF = \frac{k_c}{\varepsilon}(T_{ig} - T_\infty) + \sigma(T_{ig}^4 - T_\infty^4) \quad (4)$$

where k_c is convection coefficient (0.012 $kW/[m^2K]$), ε is surface emissivity (estimated at 0.9), T_{ig} is surface temperature at ignition (K), T_∞ is ambient temperature (293 K), and σ is Stefan–Boltzmann constant (5.67×10^{-11} $kW/[m^2K^4]$). The average value of T_{ig}, along with extrapolated and bracketed CHF values are summarized in Table 4.3.

Table 4.3. Average Surface Temperature at Ignition and Extrapolated and Bracketed Critical Heat Flux for Ignition

Average Surface Temperature at Ignition, T_{ig} (K)	CHF for Ignition - Extrapolated (kW/m^2)	CHF for Ignition - Bracketed (kW/m^2)
873	40.3	42.0

The t_{ig} values were also obtained from tests at varied heat fluxes from 40 kW/m^2 through 100 kW/m^2. Figure 4.1 shows the plot of $t_{ig}^{-1/2}$ versus external heat flux, which was used to calculate the thermal response parameter (TRP). The TRP is a measure of the thermal inertia of a material (product of thermal conductivity, density, and specific heat). It is defined as the inverse of the slope of the linear fit obtained from the $t_{ig}^{-1/2}$ versus external heat flux data, which is given as 465 ± 22 $kWs^{1/2}/m^2$ in Figure 4.1.

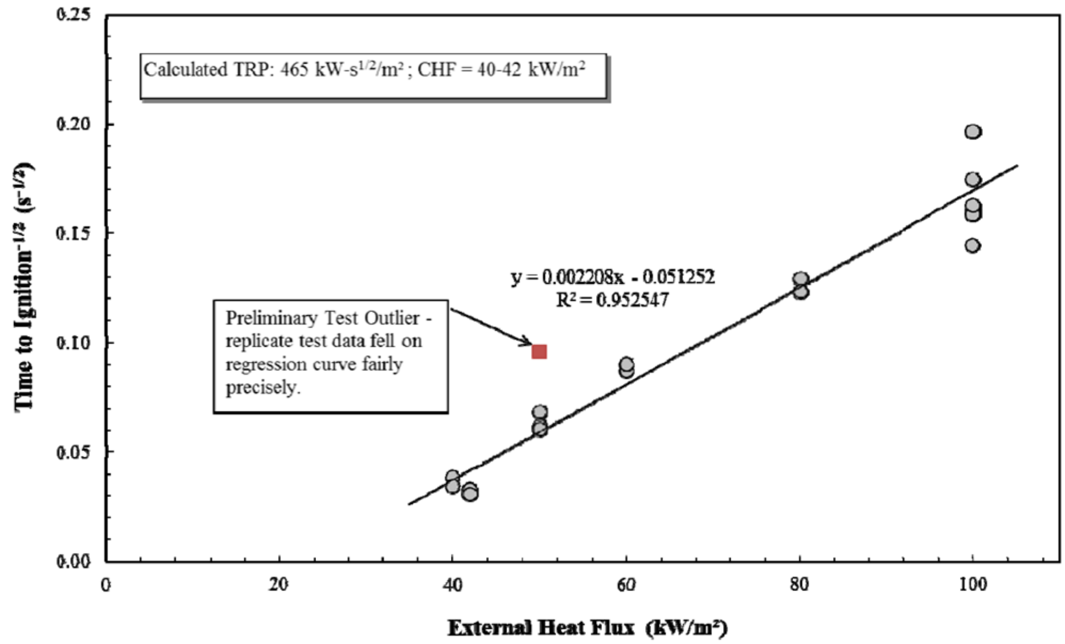

Figure 4.1. Time to Ignition as a Function of External Heat Flux to Determine the TRP (from SwRI report for R13295.12.008c)

The fire propagation index (FPI) is a measure of full-scale flame spread propensity and is calculated based on the TRP and peak HRR data. Typically, the FPI is computed with peak HRR measured at 50 kW/m^2 exposure irradiance of the test specimen. However, because the WTP was interested in HRR data at a heat flux of 100 kW/m^2, values of FPI were calculated for both heat fluxes and reported in Table 4.4. Along with the HRR and additional data obtained from tests at 50 kW/m^2, the FPI is calculated from

$$FPI = \frac{1000(0.042 HRR_{peak})^{1/3}}{TRP} \quad (5)$$

Table 4.4. Summary of Cone Calorimeter Test Results and Calculated Fire Propagation Index (FPI) at 50 and 100 kW/m^2

Heat Flux	HRRpeak (kW/m^2)	THR (MJ/m^2)	EHC (MJ/kg)	SEA (m^2/kg)	FPI m$^{5/3}$/[kW$^{2/3}$s$^{1/2}$]
50 kW/m$^{2(a)}$	44	116	13.7$^{(b)}$	8.0$^{(c)}$	2.64
100 kW/m$^{2(d)}$	51	123	13.3	12.5$^{(e)}$	2.77

(a) Average of triplicate test results.
(b) Average EHC for two of three runs was 12.2 MJ/kg (one run was 16.7 MJ/kg, which skews the average).
(c) Average SEA for two of three runs was 1.5 m^2/kg (one run was 22 m^2/kg, which skews the average).
(d) Average of six test results.
(e) There is a fair amount of scatter in this measured parameter, likely due to exactly how a given batch of samples was dried prior to testing.

4.3 Microscale Calorimeter Results

Table 4.5 shows the results of flammability characteristics following the standard procedure ASTM D7309-07A Method A between 75°C and 750°C at a heating rate of 1 K/s. The negative test was performed on silver and positive test was performed on PMMA. In Table 4.5, results are listed for the following properties:

- maximum specific heat release rate (Q_{max}, W/g): the maximum value of the specific heat release rate recorded during the test
- heat release temperature (T_{max}, °C): the specimen temperature at which the specific heat release rate is a maximum during controlled thermal decomposition
- heat release capacity (η, J/[gK]): the maximum specific heat release rate during controlled thermal decomposition divided by the heating rate of the test
- specific heat release (h_c, J/g): the net heat of complete combustion of the volatiles liberated during controlled thermal decomposition per unit initial specimen mass
- pyrolysis residue (Y_p, g/g): the fraction of the initial specimen mass remaining after controlled anaerobic thermal decomposition (Method A only)
- specific heat of combustion for specimen gases ($h_{c,gas}$, J/g): the net heat of complete combustion of the volatiles liberated during controlled thermal decomposition per unit mass of volatiles, i.e., $h_{c,gas} = h_c/(1 - Y_p)$.

Table 4.5also includes the repeatability limit and reproducibility limit values related to the precision of the microscale calorimeter test method based on interlaboratory trials involving twelve laboratories on five different solid materials.

The differences from duplicate tests in this study are, in general, comparable to or significantly smaller than the corresponding repeatability limits except for the heat release temperature (T_{max}). The large difference for T_{max} (14°C) compared to the repeatability limit (5.1–6.6°C) is likely due to the characteristics of the present SRF resin material different from those common materials used in the interlaboratory studies.

Table 4.5. Micro-Calorimeter Test Results of SRF Resin, Method A, 75–750°C, 1 K/s

Property	Q_{max} (W/g)	T_{max} (°C)	η (J/[gK])	h_c (kJ/g)	Y_p (g/g)	$h_{c,gas}$ (kJ/g)
Initial run[(a)]	22.4	641	22.4	5.171	0.5754	12.296
Duplicate run[(a)]	20.8	655	20.8	4.237	0.5853	10.139
Average	21.6	648	21.6	4.704	0.5804	11.218
Difference	1.6	14	1.6	0.934	0.0099	2.157
Precision estimates[(b)]						
r	18.5–70.0	5.1–6.6	20.8–91.0	0.6–2.3	0.0053–0.0380	NA
R	53.3–250.1	23.0–42.3	50.5–242.0	2.8–6.4	0.0091–0.0712	NA
Negative and positive standards						
Silver (negative standard)	9.7	1010	9.7	3.350	1	0
PMMA (positive standard)	383.8	674	383.8	27.653	0	27.653

(a) Average of triplicate results.
(b) Based on interlaboratory tests involving twelve laboratories on five different solid materials (ASTM D7309-07A).
NA not available.

The results of the similar tests performed per ASTM D7309-07A Method B are summarized in Table 4.6. The η value is the same as Q_{max} because the heating rate used in all tests was 1 K/s for both Methods A and B. For Method B, the test measures net heat of combustion of both the specimen gases and solid residue, i.e., $Y_p = 0$, and therefore $h_{c,gas} = h_c$. The precision data for Method B were not available. There was no statement on the bias for the ASTM D7309-07A method.

Table 4.6. Micro-Calorimeter Test Results of SRF Resin, Method B, 75–750°C, 1 K/s

Property	Q_{max} (W/g)	T_{max} (°C)	η (J/[gK])	h_c (kJ/g)
Initial run[(a)]	127.6	566	127.6	13.374
Duplicate run[(a)]	121.5	568	121.5	12.703
Average	124.6	567	124.6	13.039
Difference	6.1	2	6.1	0.671

(a) Average of triplicate results.

4.4 Smoke Particle Size and Specific Optical Density

Figure 4.2 and Figure 4.3 show the particle size distribution of smoke generated from the tests performed under nonflaming condition and under flaming condition, respectively. The median diameter (d_{50}) was 2.4 µm under nonflaming condition and 4.0 µm (average from duplicate tests) under flaming condition. Under nonflaming condition, the largest particle was 7.2 µm and about 1.5% of the particles were smaller than 0.52 µm. Under flaming condition, the largest particle was 11.1 µm and only about 0.3% of the particles were smaller than 0.52 µm.

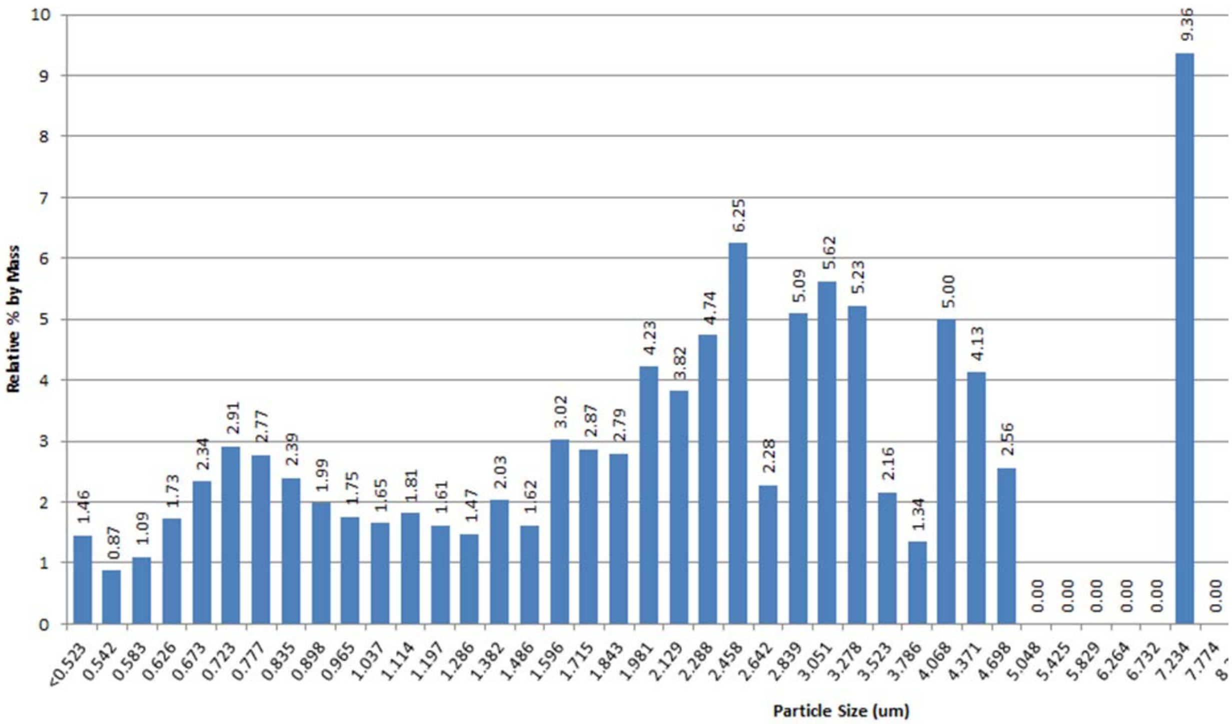

Figure 4.2. Smoke Particle Size Distribution of SRF Resin Under Nonflaming Condition

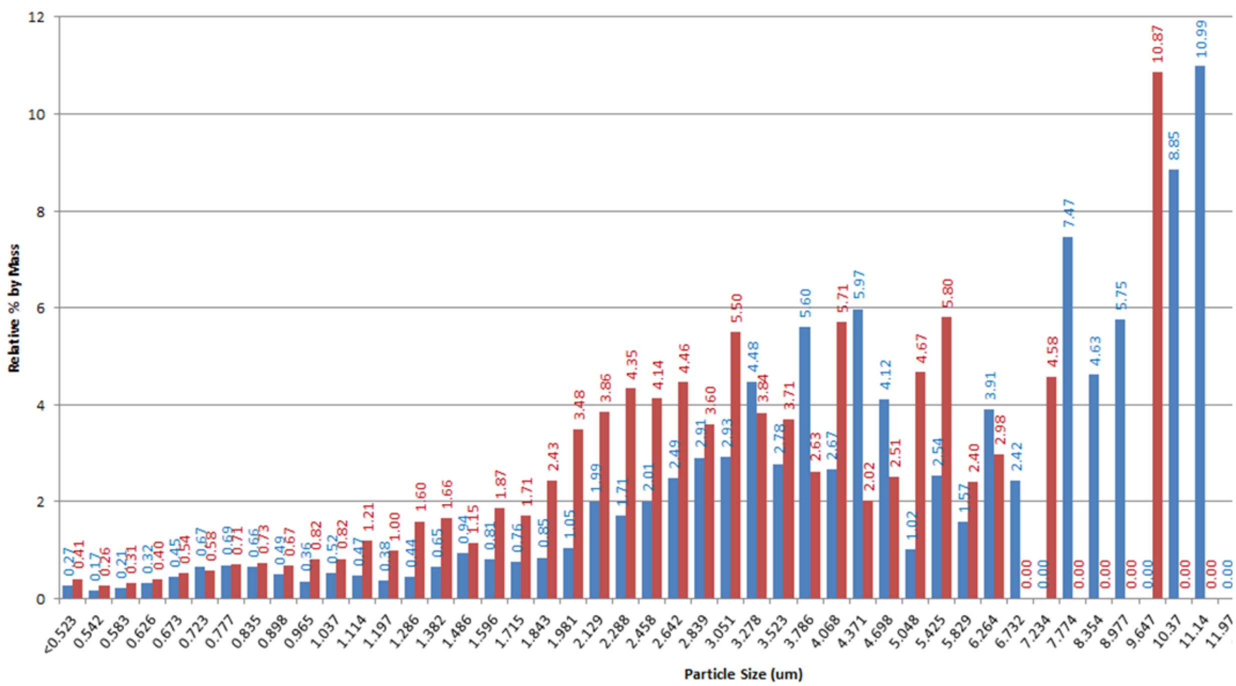

Figure 4.3. Smoke Particle Size Distribution of SRF Resin Under Flaming Condition (blue = initial test, red = duplicate test)

Figure 4.4 and Figure 4.5 show the particle size distribution of smoke generated from the negative standard (calcium silicate) and positive standard (Schneller core), respectively. The d_{50} was 3.2 µm for negative standard and 1.8 µm for positive standard.

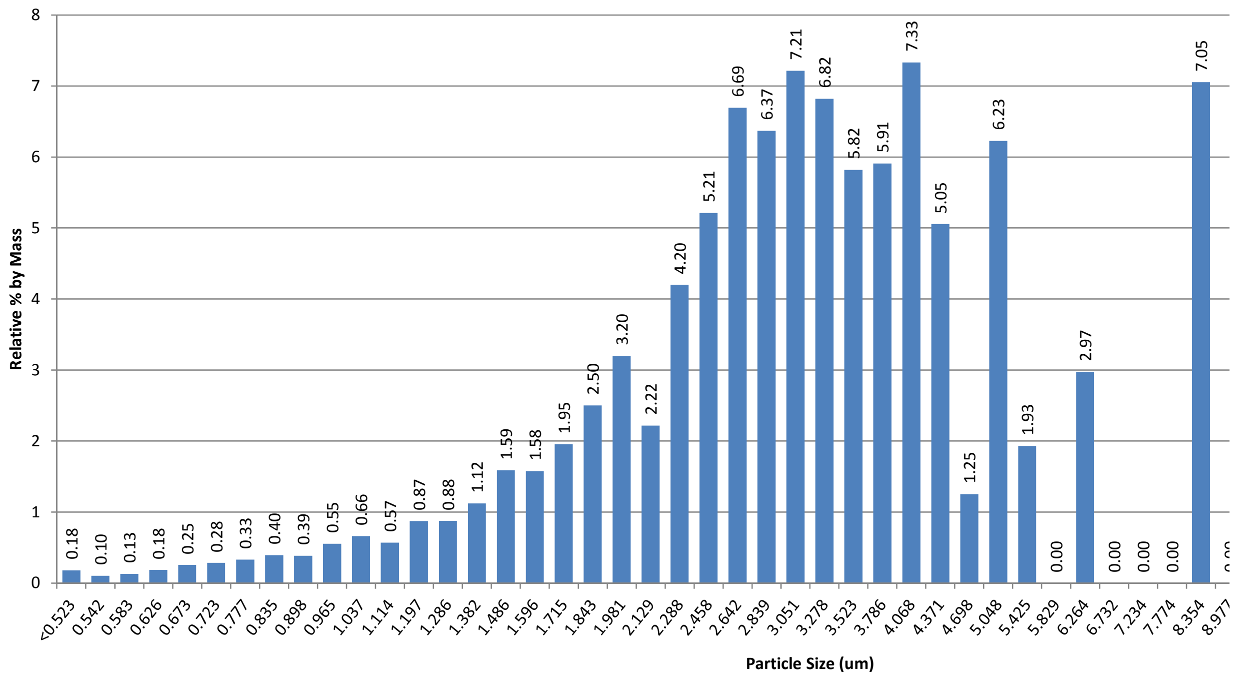

Figure 4.4. Smoke Particle Size Distribution of Negative Standard Under Nonflaming Condition

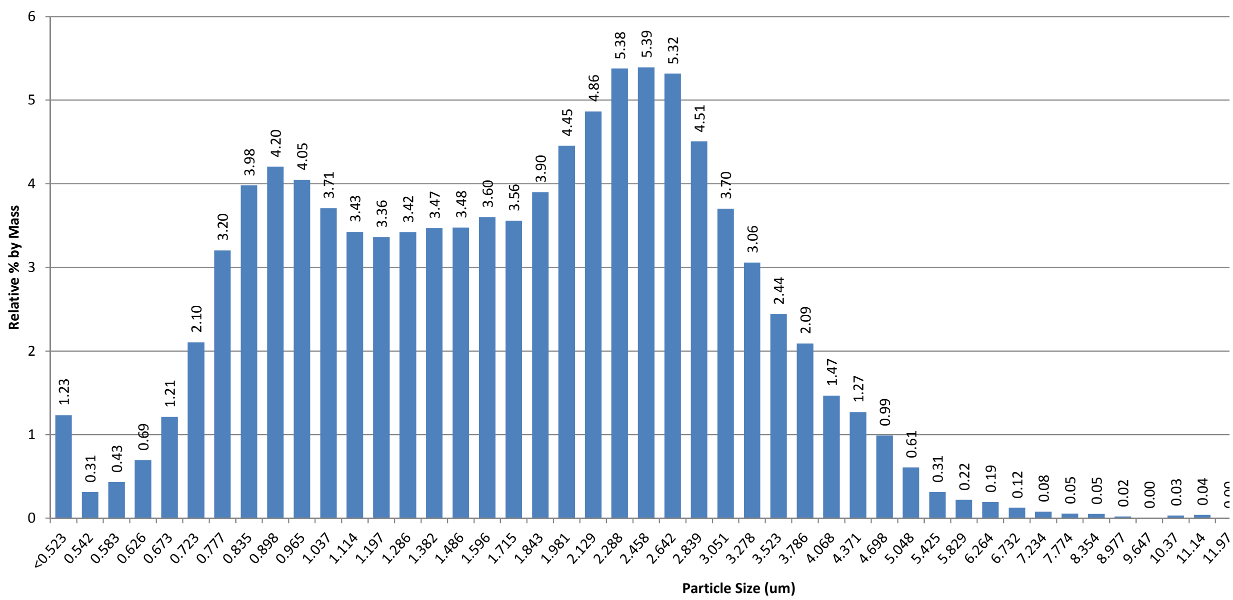

Figure 4.5. Smoke Particle Size Distribution of Positive Standard Under Flaming Condition

Table 4.7 summarizes the results for specific optical density of smoke (D_s) determined in general accordance with standard procedure ASTM E662-09 but with some deviations as described in Section 3.5. The negative test was performed on calcium silicate, which is non-combustible material and positive tests were performed on α-cellulose material under the nonflaming condition and on Schneller core panel under the flaming condition. The results from negative and positive tests are given in Table 4.7. Table 4.7 also includes the precision estimates obtained from interlaboratory trials. For standard procedure ASTM E662-09, the precision estimates are given as relative *r* and *R* calculated by dividing the r and R by average optical density, which need to be compared with relative difference that is also included in Table 4.7. The difference from duplicate runs in this study is generally comparable to the relative repeatability limits although there were deviations from the ASTM standards as described in Section 3.5. Standard procedure ASTM E662-09 states that the bias of this test method is unknown because the value of specific optical density obtained in this procedure is defined only in terms of this test method.

The maximum gas concentrations measured using an FTIR spectrometer are in Table 4.8. The other gases that were also measured—HCl, HCN, HF, HBr, and NOx—were not detected in the SRF resin sample. Table 4.8 also includes maximum gas concentration results from negative and positive tests. There are large differences in CO and CO_2 concentrations between duplicate results. SwRI performed careful review of the raw data files to investigate the potential cause of these large differences and did not find any indication that the tests were done incorrectly. It is suspected that the large differences are likely due to the characteristics inherent to the present SRF resin material.

Table 4.7. Maximum Specific Optical Density (D_s)

Test Run	Flaming Condition		Nonflaming Condition	
	Maximum D_s	Maximum D_s (corrected)	Maximum D_s	Maximum D_s (corrected)
Initial run[(a)]	0.60	0.56	0.44	0.30
Duplicate run[(a)]	0.70	0.41	0.26	0.21
Average	0.65	0.49	0.35	0.26
Difference	0.10	0.15	0.18	0.09
Relative difference	15.4%	30.9%	51.4%	35.3%
Precision estimates[(b)]				
Relative r	5.7%–47.5%	NA	5.0%–51.4%	NA
Relative R	23.0%–117.7%	NA	16.0%–91.5%	NA
Negative and positive standards				
Calcium silicate (negative standard)	0.50	0.50	0.47	0.00
α-cellulose (positive standard)	NM	NM	178.69	150.14
Schneller core (positive standard)	107.65	91.98	NM	NM

(a) Average of triplicate results.
(b) Based on interlaboratory tests (ASTM E662-09).
NA not available.
NM not measured.

Table 4.8. Maximum Gas Concentrations

Exposure Mode	CO (ppm)	CO_2 (ppm)	SO_2 (ppm)	HCN (ppm)
Nonflaming	803 ± 6	834 ± 41	3 ± 1	ND
Flaming	1681 ± 18	1628 ± 63	7 ± 2	ND
Nonflaming, duplicate	88 ± 1	3421 ± 123	2.3 ± 1.5	ND
Flaming, duplicate	762 ± 55	1057 ± 43	7 ± 3	ND
Negative and positive standards				
negative, nonflaming	ND	116 ± 21	ND	ND
negative, flaming	ND	1113 ± 38	ND	10 ± 2
positive, nonflaming	332 ± 2	820 ± 37	3 ± 1	ND
positive, flaming	20 ± 1	501 ± 7	ND	ND

± represents the 90% confidence level in the concentration measurement.
ND not detected.

4.5 Discussion on Smoke Data

Smoke is defined as the airborne solid and liquid particulates and gases evolved when a material undergoes pyrolysis or combustion (ASTM E176-10). The property most widely used to characterize the smoke is the light extinction coefficient. The physical basis for light extinction measurements is Bouguer's law (also known Lambert's law) (Mulholland 1995):

$$\frac{I}{I^0} = e^{-KL} \tag{6}$$

where I^0 is intensity of the incident light, I is the intensity of the light transmitted through the smoke, L is the light path length (m), and K is the light extinction coefficient (m^{-1}).

Per ASTM E662-09 standard, the specific optical density, D_s (dimensionless), is calculated by the equation:

$$D_s = \frac{V}{AL}\log_{10}(\frac{I^0}{I}) = \frac{KV}{2.303A} \tag{7}$$

where V is the volume of test chamber (m^3) and A is the exposed area of test sample (m^2). The D_s represents the amount of visible smoke accumulated in a test chamber measured over unit path length within a chamber of unit volume produced from a sample of unit surface area. When the mass loss of the sample is measured, the mass optical density, D_m, is the appropriate measure of visible smoke:

$$D_m = \frac{D_s A}{\Delta m} \tag{8}$$

where Δm is the mass loss (kg). Table 4.9 summarizes the maximum D_s (from Table 4.7, average value for SRF resin), the mass loss, and resulting maximum mass optical density values of the SRF resin compared with those of the negative and positive standard materials. Table 4.9 shows that the SRF resin has the maximum D_s and D_m comparable to or lower than the negative standard, calcium silicate, which is a non-combustible material.

Table 4.9. Maximum Mass Optical Density (D_m)

Material	Flaming Condition			Nonflaming Condition		
	Max D_s (corrected)	Mass loss (g)	Max D_m (m^2/kg)	Max D_s (corrected)	Mass loss (g)	Max D_m (m^2/kg)
SRF resin	0.49	17.54	0.12	0.26	17.73	0.06
Calcium silicate (negative standard)	0.50	1.80	1.18	0.00	2.00	0.00
α-cellulose (positive standard)	NM	NM	NM	150.1	3.20	198.8
Schneller core (positive standard)	91.98	3.26	119.6	NM	NM	NM

NM not measured.

The smoke data from cone calorimetry per ASTM E1354-10 standard are also obtained from the light extinction measurements based on Equation (6). However, the cone calorimetry is a dynamic test method that measures the smoke in the flowing exhaust generated at 50 or 100 kW/m^2 heat flux compared to the static method of ASTM E662-09 standard that measures the smoke accumulated in a closed system (smoke chamber) generated at 25 kW/m^2 heat flux. Per ASTM E1354-10 standard, the total smoke production per unit sample area, S_A, (m^2/m^2) is calculated by:

$$S_A = \frac{K\dot{V}t_T}{A} = \frac{\dot{V}t_T}{AL}\ln(\frac{I^0}{I}) \qquad (9)$$

where $\dot{V}$ is the volume flow rate of exhaust at the location of the light extinction measurement (m^3/s) and t_T is the total test duration. Then, the specific extinction area, SEA (kg/m^2), is calculated by:

$$\text{SEA} = \frac{S_A}{\Delta m_s} \qquad (10)$$

where Δm_s is the mass loss per unit sample surface area (kg/m^2). The SEA is a measure of the instantaneous amount of smoke being produced per unit mass of sample burnt, which is equivalent to the D_m from the smoke chamber test, although they are obtained under different test conditions mentioned above. As was summarized in Table 4.2, the total smoke production (S_A) of SRF resin is slightly larger but the SEA of SRF resin is significantly smaller than the negative standard, marinite board, which is also a non-combustible material.

Table 4.10 summarizes the d_{50} and estimated concentration of smoke used in particle size distribution for the SRF resin compared with those for the negative and positive standard materials. Table 4.10 also notes that the estimated concentrations for the SRF resin and negative standard material are indistinguishable from background, which supports the results obtained from light extinction measurements discussed above.

Table 4.10. Summary of Particle Size Distribution Measurement Results

Material	d_{50}, µm	Estimated concentration, µg/m^3	Note
SRF resin, flaming	4.0	10.9	indistinguishable from background
SRF resin, nonflaming	2.4	4.2	indistinguishable from background
Calcium silicate (negative standard), flaming	3.2	8.7	indistinguishable from background
Schneller core (positive standard), flaming	1.8	3589	

5.0 Summary

Table 5.1 summarizes the mean, difference, and repeatability limit presented in Section 4. The differences from duplicate tests in this study are, in general, comparable to or significantly smaller than the corresponding repeatability limits, suggesting general satisfactory precision of the test results. Exceptions were noted for the time to ignition (t_{ig}, ASTM E1354-10) and heat release temperature (T_{max}, ASTM D7309-07A) of which potential explanations were provided in Section 4.

Table 5.1. Summary of Fire Test Results on SRF Resin

ASTM Procedure	Analysis/Property	Mean	Difference	Repeatability Limit
ASTM D1929-96	Flash ignition temperature, FIT (°C)	595	10	8–13
	Spontaneous ignition temperature, SIT (°C)	613	5	11–31
ASTM E1354-10	Heat release rate, HRR_{peak} (kW/m^2)	51	3	20.0
	Time to ignition, t_{ig} (s)	35	14	8.5
	Total heat release, THR (MJ/m^2)	123.4	1.5	15.8
	Mass loss (g/m^2)	9296	1028	NA
	Mass loss rate, MLR (g/[m^{2}s])	2.6	0.2	NA
	Effective heat of combustion, EHC (MJ/kg)	13.3	1.6	1.9
	Total smoke production, S_A (m^2/ m^2)	118	83	NA
	Specific smoke extinction area, SEA (m^2/kg)	13	10	60.0
	Critical heat flux for ignition – extrapolated (kW/m^2)	40.3	--	NA
	Critical heat flux for ignition –bracketed (kW/m^2)	42.0	--	NA
	FPI (m$^{5/3}$/[kW$^{2/3}$s$^{1/2}$]) at 50 kW/m^2	2.64	--	NA
	FPI (m$^{5/3}$/[kW$^{2/3}$s$^{1/2}$]) at 100 kW/m^2	2.77	--	NA
ASTM D7309-07A, Method A	Maximum specific heat release rate, Q_{max} (W/g)	21.6	1.6	18.5–70.0
	Heat release temperature, T_{max} (°C)	648	14	5.1–6.6
	Heat release capacity, η (J/gK)	21.6	1.6	20.8–91.0
	Specific heat release, h_c (kJ/g)	4.704	0.934	0.6–2.3
	Pyrolysis residue, Y_p (g/g)	0.5804	0.0099	0.0053–0.0380
	Specific heat of combustion, $h_{c,gas}$ (kJ/g)	11.218	2.157	NA
ASTM D7309-07A, Method B	Maximum specific heat release rate, Q_{max} (W/g)	124.6	6.1	NA
	Heat release temperature, T_{max} (°C)	567	2	NA
	Heat release capacity, η (J/gK)	124.6	6.1	NA
	Specific heat release, h_c (kJ/g)	13.039	0.671	NA
ASTM E662-09[(a)]	Maximum specific optical density, D_s (flaming)	0.65	0.10	0.04–0.31[(b)]
	Corrected maximum specific optical density, D_s(corr) (flaming)	0.49	0.15	NA
	Maximum specific optical density, D_s (nonflaming)	0.35	0.18	0.02–0.18[(b)]
	Corrected maximum specific optical density, D_s(corr) (nonflaming)	0.26	0.09	NA
None	Maximum concentration of CO (ppm) (flaming)	1681 ± 18; 762 ± 55	--	NA
	Maximum concentration of CO (ppm) (nonflaming)	803 ± 6; 88 ± 1	--	NA
	Maximum concentration of CO_2 (ppm) (flaming)	1628 ± 63; 1057 ± 43	--	NA
	Maximum concentration of CO_2 (ppm) (nonflaming)	834 ± 41; 3421 ± 123	--	NA
	Maximum concentration of SO_2 (ppm) (flaming)	7 ± 2; 7 ± 3	--	NA
	Maximum concentration of SO_2 (ppm) (nonflaming)	3 ± 1; 2.3 ± 1.5	--	NA

(a) With deviations described in Section 3.5.
(b) Calculated by multiplying the average value with the relative repeatability limit given in Table 4.7.
-- Not applicable.
NA Not available.

It is suggested that the gas analyses be repeated so that the large differences in CO and CO_2 concentrations between duplicate results may be better understood.

From the smoke tests based on the light extinction measurements under dynamic (ASTM E1354-10) and static (ASTM E662-09) conditions, it was shown that the SRF resin had smoke production comparable to or slightly higher than that of negative standard materials that are non-combustible.

6.0 References

ASTM D1929-96. 1996. *Standard Test Method for Determining Ignition Temperature of Plastics*, ASTM International, West Conshohocken, PA.

ASTM D7309-07A. 2007. *Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry*, ASTM International, West Conshohocken, PA.

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Mulholland. G.W. 1995. *Smoke Production and Properties*, in SFPE Handbook of Fire Protection Engineering, 2nd Edition, Chapter 15, Section 2, 2/217-2/227.

Not Publically Available

24590-PTF-TSP-RT-09-002, Rev 0. 2009. *RF Resin Cesium Removal with Expanded Load and Elution Conditions*, River Protection Project Waste Treatment Plant, Richland, WA.

24590-PTF-TEF-RT-11-00003, Rev 0. 2011. *Test Exception to 24590-QL-HC9-WA49-00001-02-00014, Rev 00C (TP-WTPSP-002, Rev 2.0)*, River Protection Project Waste Treatment Plant, Richland, WA.

TP-WTPSP-002, Rev 3.0. 2011. *Cesium Ion Exchange Simulant Testing in Support of M-6*, Pacific Northwest National Laboratory, Richland, WA.

Appendix A

SwRI Reports

SOUTHWEST RESEARCH INSTITUTE®

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Chemistry and Chemical Engineering
Department of Analytical and Environmental Chemistry

December 16, 2011

Battelle Memorial Institute - PNNL
MSIN K6-24
790 6th Street
Richland, Washington 99354

Attention: Renee Russell

Subject:	Purchase Order No.:	167930
	SDG Number:	479235
	SwRI Project No.:	13295.12.00X
	SwRI Task Order No.:	111111-12
	SwRI Sample Receipt No.:	46069
	Samples Received:	11/11/11
	Required Analysis:	Various / See RFP 190709

Dear Ms. Russell,

Please find the enclosed results for the one overall sample received on the above referenced date. Should you have any questions, please feel free to contact me at (210) 522-5428, michael.dammann@swri.org.

Best Regards,

Michael J. Dammann
Director

SOUTHWEST RESEARCH INSTITUTE

CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008a
(ASTM D 1929 Testing)

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CHEMISTRY AND CHEMICAL ENGINEERING DIVISION
FIRE TECHNOLOGY DEPARTMENT
WWW.FIRE.SWRI.ORG
FAX (210) 522-3377

ASTM D 1929 – 96 (RE-APPROVED 2001), *STANDARD TEST METHOD FOR DETERMINING IGNITION TEMPERATURE OF PLASTICS*

MATERIAL ID: ***RESORCINOL-FORMALDEHYDE POLYMER***

FINAL REPORT
Consisting of 10 Pages

SwRI® Project No. 01.13295.12.008a
Test Dates: November 29, and December 1–3 and 5, 2011
Report Date: December 16, 2011

Prepared for:

Battelle Memorial Institute
902 Battelle Blvd, K6-79
P.O. Box 999
Richland, WA 99352

Submitted by:

Eugene F. Horton
Senior Engineering Technologist
Material Flammability Section

Approved by:

For: Matthew S. Blais, Ph.D.
Director
Fire Technology Department

1.0 INTRODUCTION

ASTM D 1929, *Standard Test Method for Determining Ignition Temperature of Plastics,* covers a laboratory determination of the spontaneous ignition temperature (SIT) and flash ignition temperature (FIT) of plastics using a hot-air furnace. The hot-air ignition furnace consists primarily of an electrical heating unit and specimen holder. The furnace tube is a vertical tube with an inside diameter of 100 ± 5 mm and a length of 230 ± 20 mm, made of ceramic that will withstand at least 750 °C. The inner ceramic tube, with an inside diameter of 75 ± 5 mm, a length of 230 ± 20 mm, and a thickness of approximately 3 mm, is placed inside the furnace tube and positioned 20 ± 2 mm above the furnace floor on spacer blocks. The pilot flame is located immediately above the opening. The test apparatus is shown in Figure 1.

Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative, and duplicate tests were run, in addition to the one standard test. The positive tests were performed on PMMA (Poly methyl methacrylate). This is a know standard used in testing and it was selected as the positive standard. The negative tests were performed on marinate board, which is a non-combustible material. This material is often used as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture.

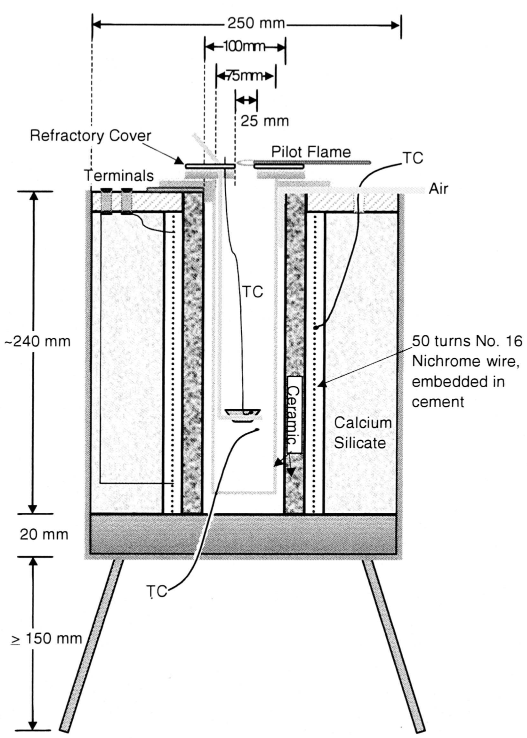


Figure 1. Schematic of Southwest Research Institute (SwRI) Hot-Air Furnace.

SIT is the minimum temperature at which the self-heating properties of the specimen lead to ignition or ignition occurs of itself, under specified test conditions, in the absence of any additional flame ignition source. The lowest air temperature at which the specimen ignites during a 10-min period is recorded as the spontaneous ignition temperature.

FIT is the minimum temperature at which, under specified test conditions, sufficient flammable gases are emitted to ignite momentarily upon application of a small external pilot flame. The lowest air temperature at which a flash is observed during a 10-min period is recorded as the flash ignition temperature.

2.0 SAMPLE IDENTIFICATION AND PREPARATION

Battelle Memorial Institute, located in Richland, Washington, provided a material identified as *Resorcinol-Formaldehyde Polymer* for testing in accordance with ASTM D 1929. A description of the material provided by the client can be found in Table 1. On November 11, 2011, SwRI received one 20-L bottle of specimen described in the table below.

TABLE 1. SAMPLE DESCRIPTION PROVIDED BY BATTELLE MEMORIAL INSTITUTE.

Material ID	Description	Color
Resorcinol-Formaldehyde Polymer	Micro Bead	Red

The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material, and then drying it in an oven. The sample was dried for a minimum of 10 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. After drying, the sample consisted of only the micro bead material; the beads became statically charged in the absence of the water medium and; therefore, they were placed in a closed container after drying, in an effort not to lose any beads. Each sample was then conditioned in a controlled environment maintained at 23 °C ± 3 °C and 50% ± 5% relative humidity until just prior to testing, but for not less than 40 h prior to testing.

3.0 RESULTS

Testing was conducted November 29, and December 1–3 and 5, 2011. Table 2 contains the results for the material provided by Battelle Memorial Institute. Test results are accurate to ± 5 °C, and are presented in detail at the end of this report. These test results relate only to the behavior of test specimens under the particular conditions of the test. They are not intended to be used, and shall not be used, to assess the potential fire hazards of a material in use.

TABLE 2. IGNITION TEMPERATURE DATA.

Material ID	SIT	FIT
Resorcinol-Formaldehyde Polymer	615 °C	600 °C
Resorcinol-Formaldehyde Polymer (Duplicate Run)	610 °C	590 °C
Marinite, Negative Standard	NA	NA
PMMA, Positive Standard	420 °C	NA

APPENDIX A

ASTM D 1929 TEST DATA SHEETS

(CONSISTING OF 6 PAGES)

SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 2, 2011	*Date Prepared by SwRI:*	Cut to size on test date
Material ID:*	Marinite, Negative Standard	*Color:*	White
Description:*	Solid White Composite	*Average Sample Mass:*	2.89 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : NA

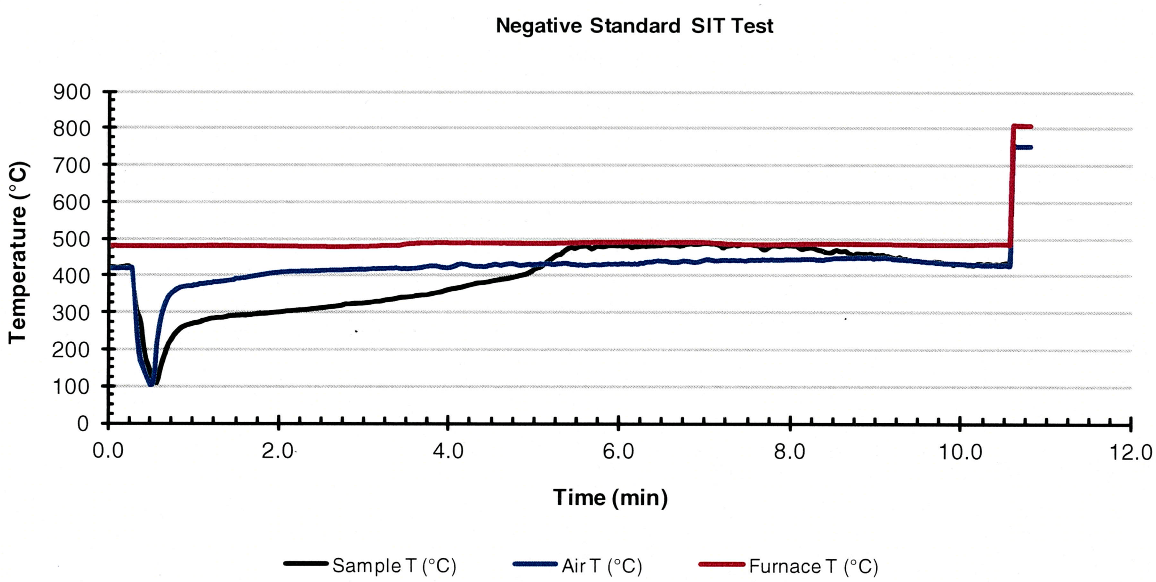
** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN008E15	2.89	2.62	0.27	750	750	809	751	753	809	No

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN008E15	0:47	NA	NA	None	None	None	None	10:48



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 3 and 5, 2011	*Date Prepared by SwRI:*	Cut to size on test date
Material ID:*	PMMA, Positive Standard	*Color:*	Black
Description:*	Solid Black Composite	*Average Sample Mass:*	3.01 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 420

** Information/instructions provided by the Client*

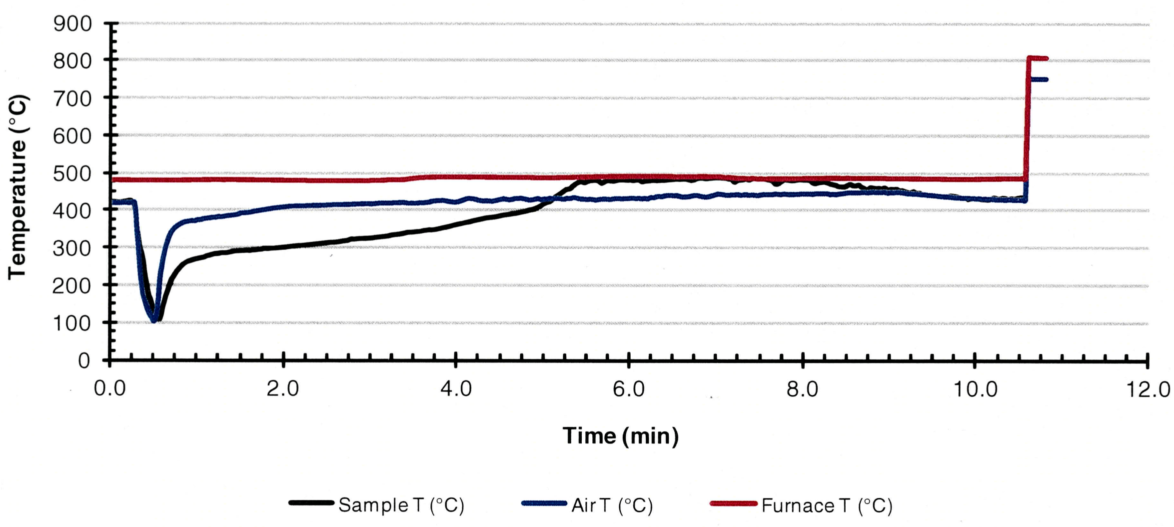
RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C)			Final Temperature (°C)			Ignition
				Sample	Air	Furnace	Sample	Air	Furnace	
11-337PNN008F65	3.15	0.75	2.40	417	410	467	422	407	474	No
11-337PNN008F45	2.87	0.00	2.87	423	420	479	434	429	484	Yes

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-337PNN008F65	0:36	NA	NA	None	Yes	None	Yes	10:36
11-337PNN008F45	0:34	NA	5:15	None	Yes	None	None	10:34

Positive Standard SIT Test



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	Nathaniel Ramos	*Receipt Date:*	November 11, 2011
Test Date(s):	November 29, 2011	*Date Prepared by SwRI:*	Cut to size on test date
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 615

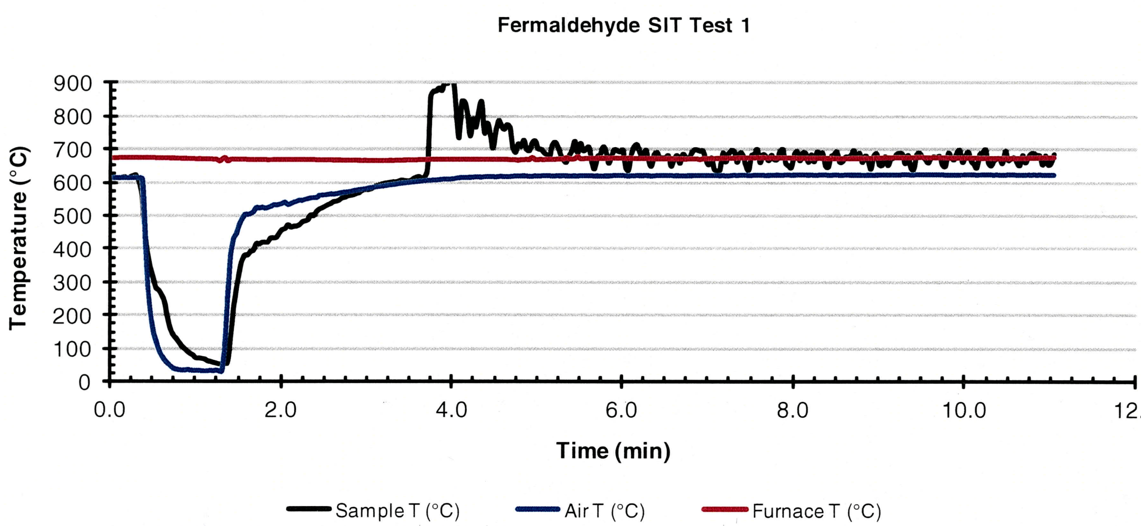
** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-333PNN00A75	3.00	1.31	1.69	609	605	603	633	614	662	No
11-333PNN00A65	3.00	1.24	1.76	615	**615**	675	670	626	678	Yes
11-333PNN00A55	3.00	1.25	1.75	630	625	684	676	635	687	Yes

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-333PNN00A75	1:56	NA	0:00	None	Yes	None	None	10:00
11-333PNN00A65	1:23	NA	2:21	None	Yes	None	None	10:00
11-333PNN00A55	1:42	NA	2:35	None	Yes	None	None	10:00



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	Nathaniel Ramos	*Receipt Date:*	November 11, 2011
Test Date(s):	December 1, 2011	*Date Prepared by SwRI:*	Tested as received
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.14 g

** Information/instructions provided by the Client*

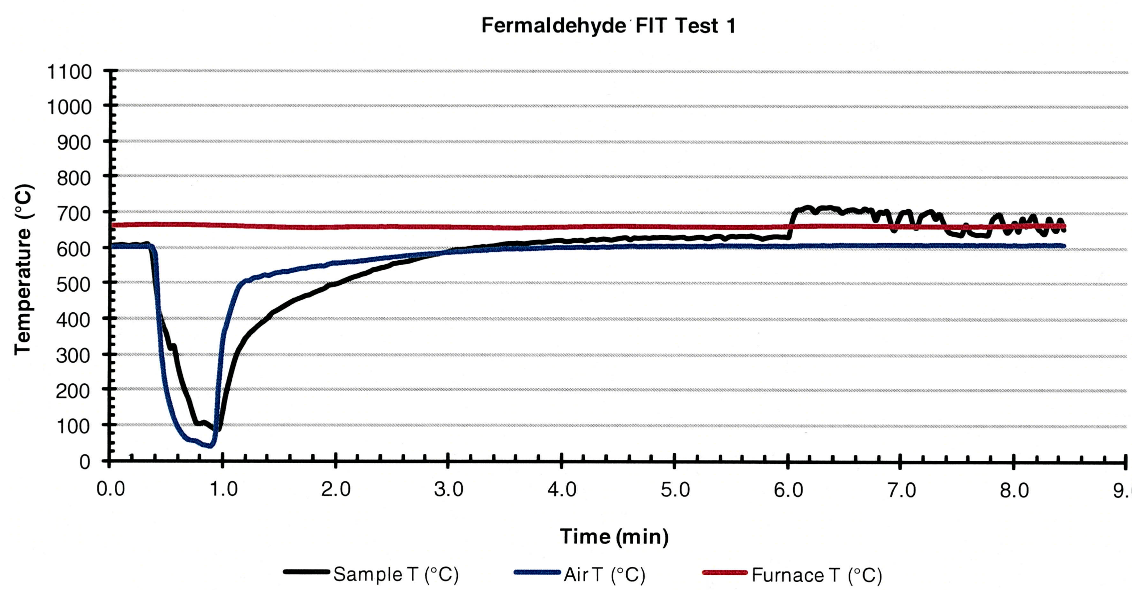
FLASH IGNITION TEMPERATURE (°C) : 420

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN00B4F	3.00	1.53	1.47	591	590	649	628	598	648	No
11-335PNN00B1F	3.14	0.75	2.39	425	**420**	436	448	408	435	Yes

FLASH IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Type (min:s)	Observed Soot (min:s)	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN00B4F	0:40	NA	NA	None	Yes	None	None	10:40
11-335PNN00B1F	0:59	NA	Flaming at 5:15	None	Yes	None	None	8:26



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 2, 2011	*Date Prepared by SwRI:*	Cut to size on test date
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 610

** Information/instructions provided by the Client*

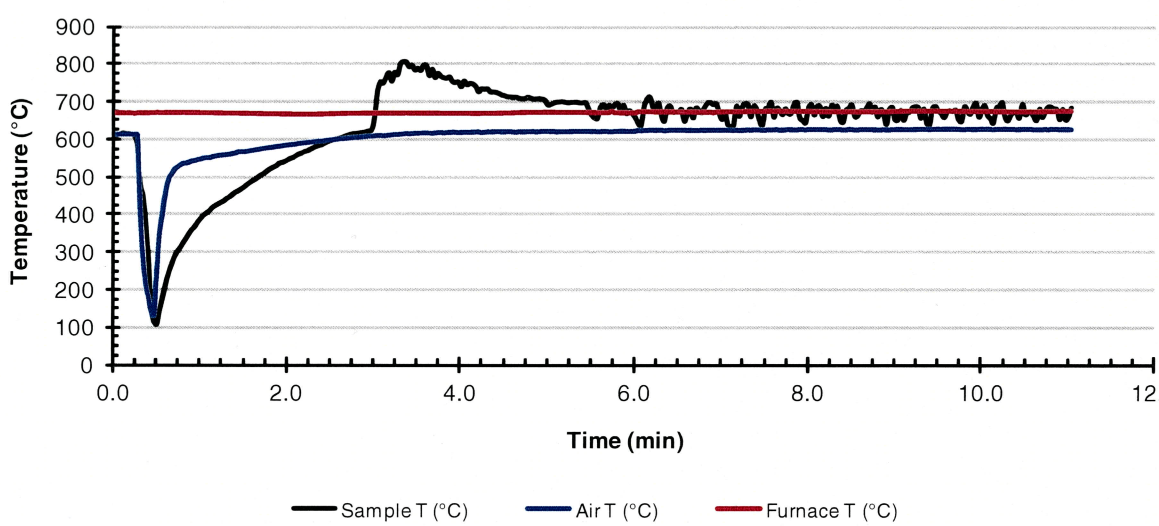
RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN00D15	3.00	1.54	1.46	603	600	658	640	609	658	No
11-336PNN00D25	3.00	1.76	1.24	611	**610**	669	659	619	670	Yes

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN00D15	0:34	NA	NA	None	Yes	None	None	10:34
11-336PNN00D25	0:31	NA	2:34	None	Yes	None	None	6:00

Fermaldehyde SIT Test 2 (Duplicate)



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 2, 2011	*Date Prepared by SwRI:*	Tested as received
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

** Information/instructions provided by the Client*

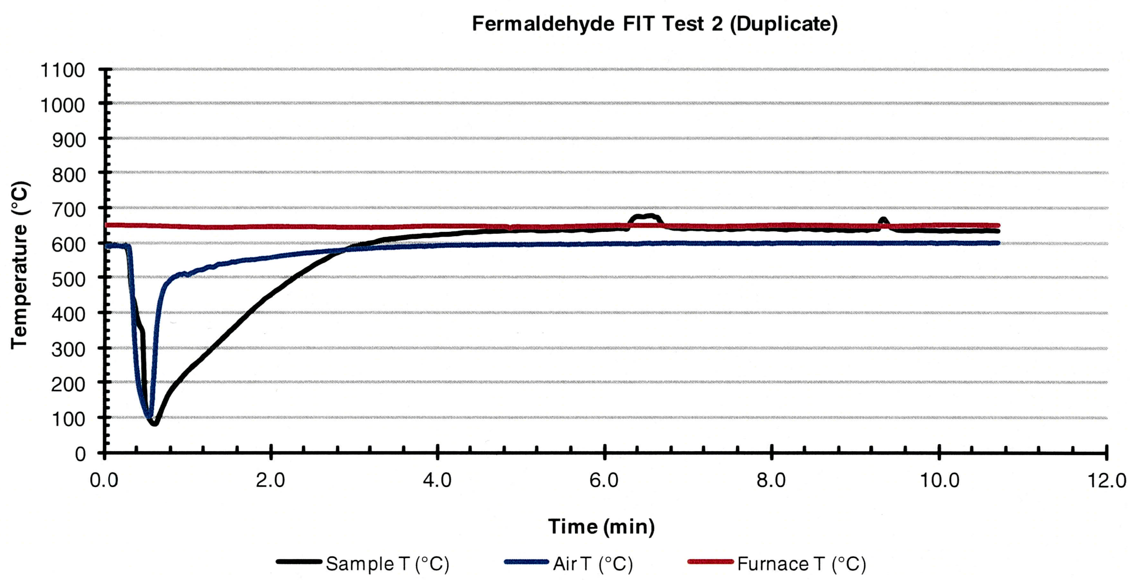
FLASH IGNITION TEMPERATURE (°C) : 590

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN00C3F	3.00	1.57	1.43	599	580	639	622	590	641	Yes
11-335PNN00C2F	3.00	1.45	1.55	589	590	649	633	600	649	Yes
11-335PNN00C1F	3.00	1.40	1.60	576	600	659	639	611	660	No

FLASH IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Type (min:s)	Observed Soot (min:s)	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN00C3F	0:40	NA	NA	None	Yes	None	None	10:40
11-335PNN00C2F	0:41	NA	Flaming at 5:39	None	Yes	None	None	10:42
11-335PNN00C1F	0:41	NA	Flaming at 5:15	None	Yes	None	None	10:42



SOUTHWEST RESEARCH INSTITUTE

CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008c
(ASTM E 1354 Testing)

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CRITICAL HEAT FLUX DETERMINATION USING ASTM E 1354-10, STANDARD TEST METHOD FOR HEAT AND VISIBLE SMOKE RELEASE RATES FOR MATERIALS AND PRODUCTS USING AN OXYGEN CONSUMPTION CALORIMETER

MATERIAL ID: ***RESORCINOL-FORMALDEHYDE POLYMER***

FINAL REPORT
Consisting of 22 Pages

SwRI® Project No. 01.13295.12.008c
Test Dates: November 12, 2011 and December 1 and 3, 2011
Report Date: December 16, 2011

Prepared for:

Battelle Memorial Institute
902 Battelle Blvd, K6-79
P.O. Box 999
Richland, WA 99352

Submitted by:

Eugene F. Horton
Senior Engineering Technologist
Material Flammability Section

Approved by:

for: Matthew S. Blais, Ph.D.
Director
Fire Technology Department

1.0 INTRODUCTION

The objective of this test program was to conduct a fire performance evaluation on a material identified as *Resorcinol-Formaldehyde Polymer,* for Battelle Memorial Institute, located in Richland, Washington. The material was tested in general accordance with ASTM E 1354-10, *Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter.* Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative and duplicate tests were run, in addition to the one standard test. The test apparatus described in ASTM E 1354 was used to determine the critical heat flux of the material. Testing was conducted at a heat flux of 20, 40, 50, 60, 80, 100 kW/m^2. Testing was conducted on November 12, 2011, and December 1 and 3, 2011, at Southwest Research Institute's (SwRI) Fire Technology Department, in San Antonio, Texas.

The negative tests were performed on marinate board, which is a non-combustible material. This material is often used as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture. The positive tests were performed on PMMA (Poly methyl methacrylate). This is a know standard used in testing and it was selected as the positive standard.

This test method is intended to measure and describe the properties of materials or products in response to heat and flame under controlled laboratory conditions. This method is not applicable to end-use products that do not have planar, or nearly planar, external surfaces. The results should not be used alone to describe or appraise the fire hazard or the fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a complete fire hazard assessment or a fire risk assessment, which takes into account all the factors that are pertinent to an assessment of the fire hazard or fire risk of a particular end use. The results apply specifically to the specimens tested, in the manner tested, and not to similar materials, nor to the performance when used in combination with other materials.

1.1 Cone Calorimeter Testing

The cone calorimeter tests were performed on November 12, 2011, December 1 and 3, 2011. Detailed data summaries and plots can be referenced in Appendix A. Table 1 provides a summary of some of the HRR data. Tables 2 and 3 provide a summary of the ignition data and several selected fire performance indices, based on the heat release and ignition tests. Figure 1 shows a plot developed in order to determine the thermal response parameter from the ignition data.

Table 1. Cone Calorimeter Summary HRR and Smoke Data*.

Material ID	Peak HRR (kW/m^2)	THR (MJ/m^2)	EHC (MJ/kg)	SEA (m^2/kg)
RCP @ 50 kW/m²	44	116	13.7**	8***
RCP @ 100 kW/m²	51	123	13.3	12.5****

* Data presented in table for testing at 50 kW/m^2 and 100 kW/m^2, are averages of three and six replicate tests, respectively.

** Average EHC for 2 of 3 runs was 12.2 MJ/kg (one run was 16.7 MJ/kg, which skews the average slightly)

*** Average SEA for 2 of 3 runs was 1.5 m^2/kg (one run was 22 m^2/kg, which skews the average slightly)

**** There is a fair amount of scatter in this measured parameter, likely due to exactly how a given batch of samples was dried prior to testing.

The peak HRR is the maximum single point HRR, as measured by oxygen consumption calorimetry. The total heat released (THR) is the integration of the HRR over the test duration. The effective heat of combustion (EHC) is calculated by dividing the THR by the total mass loss. The specific extinction area (SEA) is a measure of material smoke production. It is calculated as the total smoke produced divided by the total mass loss.

Table 2. Cone Calorimeter Summary of Ignition Fire Indices.

Material ID	Surface Temperature at Ignition (K)	CHF for Ignition - Extrapolated (kW/m^2)	CHF for Ignition – Bracketed (kW/m^2)	TRP ($kW\text{-}s^{1/2}/m^2$)
RCP	873	40.3	42	465

Table 3. Cone Calorimeter Summary of Fire Indices.

Material ID	Peak HRR at 50 kW/m^2 (kW)	FPI at 50 kW/m^2 ($m^{5/3}/kW^{2/3}\text{-}s^{1/2}$)	Peak HRR at 100 kW/m^2 (kW)	FPI at 100 kW/m^2 ($m^{5/3}/kW^{2/3}\text{-}s^{1/2}$)
RCP	44	2.64	51	2.77

The surface temperature at ignition is the directly measured surface temperature at ignition during the selected ignition tests in the cone calorimeter. The CHF for ignition is calculated from the surface temperature at ignition based on the following equation:

$$CHF = \frac{h_c}{\varepsilon}(T_{ig} - T_\infty) + \sigma(T_{ig}^4 - T_\infty^4)\text{, where}$$

h_c = convection coefficient (0.012 kW/m^2K);

ε = surface emissivity (estimated at 0.9);

T_{ig} = surface temperature at ignition (K);

T_∞ = ambient temperature (293 K); and

σ = Boltzmann constant (5.67×10^{-11} kW/m^2K^4)

It is important to note that since the surface temperature at ignition is used for the CHF calculation, the reported value above assumes that this is the absolute minimum heat flux required over an infinitely long heating period to ignite the material. In practice, the ignition tests are conducted for 20 min and if there is no ignition, the test is concluded and another test is performed at the next heat flux. There is some difference between these calculated critical heat fluxes and the ignition observations both in small-scale and in the standoff distance tests for much shorter heating periods. This explains the difference between the extrapolated and bracketed critical heat fluxes provided in Table 3.

The TRP is a measure of the thermal inertia of a material (product of thermal conductivity, density, and specific heat). It is calculated by plotting the inverse square root of the ignition time versus the external heat flux and fitting a line through the data points. The inverse of the slope of this line is defined as the TRP.

The fire propagation index (FPI) is a measure of full-scale flame spread propensity and is calculated based on ignition data (TRP) and HRR data (peak HRR). Typically, this index is computed with the peak HRR measured at 50 kW/m^2 exposure irradiance to the test specimen. However, since the Client was interested in HRR data at a heat flux of 100 kW/m^2, values of FPI were calculated for both heat fluxes and are provided in Table 4. The FPI is calculated based on the following equation:

$$FPI = \frac{1000(0.042 \cdot Q_{peak})^{1/3}}{TRP}, \text{ where}$$

Q_{peak} = peak heat release rate in cone at 50 and 100 kW/m^2 (kW/m^2);
TRP = thermal response parameter ($kW\text{-}s^{1/2}/m^2$);

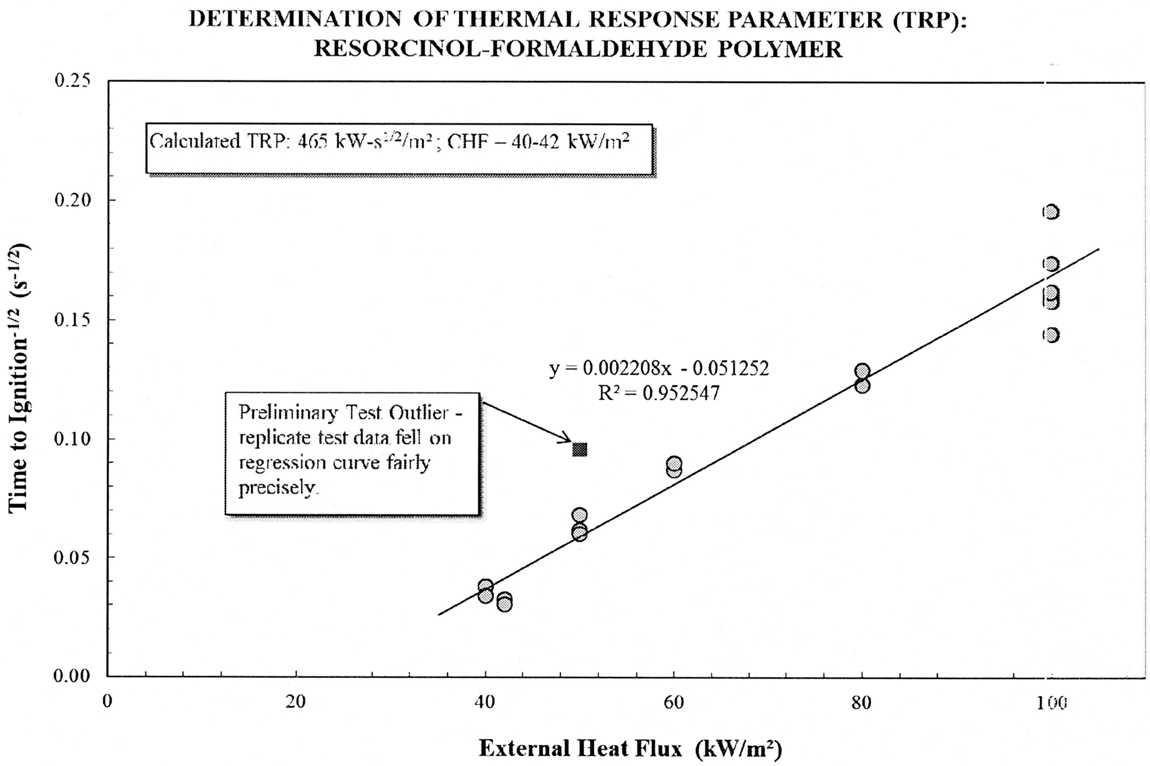


Figure 1. Plot of Ignition Data to Determine the TRP.

2.0 CONE CALORIMETER

For organic solids, liquids, and gases, a nearly constant net amount of heat is released per unit mass of oxygen consumed for complete combustion. An average value for this constant of 13.1 MJ/kg of O_2 can be used for practical applications and is accurate with very few exceptions to within ± 5%. Therefore, measurements of the oxygen consumed in a combustion system can be used to determine the net heat released. This technique, generally referred to as the "oxygen consumption technique", is now the most widely used and accurate method for measuring heat release rate in experimental fires.

The Cone Calorimeter is a sophisticated small-scale test apparatus, which measures the rate of heat release of materials and products under a wide range of conditions using the oxygen consumption technique. A schematic of the instrument is shown in Figure 2. Other useful information obtained from Cone Calorimeter tests includes time to ignition, mass loss rate, smoke production rate, and effective heat of combustion.

In the Cone Calorimeter, a square sample measuring 100 × 100 mm (4 × 4 in.) is exposed to the radiant flux of an electric heater. The heater is in the shape of a truncated cone and is capable of providing heat fluxes to the specimen in the range of 0–100 kW/m². An electric spark igniter is used for piloted ignition of the pyrolysis gases produced by the radiant heater.

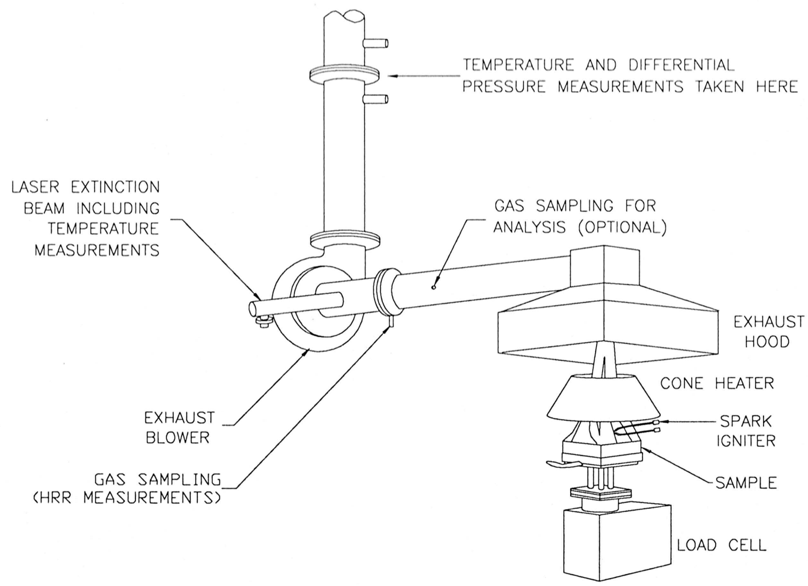


Figure 2. Schematic of the Cone Calorimeter Apparatus.

Test specimens are to be representative of the product's end use and can have a maximum thickness of 50 mm (2 in.). Specimens with a thickness of less than 6 mm are to be tested using a substrate that is representative of end-use conditions. Prior to testing, the test specimen is wrapped in aluminum foil, backed with a layer of low-density refractory fiber blanket, and placed in a standard specimen holder. An optional edge frame can be used to retain the sample within the specimen holder during testing. A load cell is used to measure the mass loss of the specimen throughout the duration of the test.

At the start of a test, the specimen (in the appropriate holder) is placed on the load cell, which is located below the heater. The top edge of the specimen is typically positioned 25 mm below the base plate of the heater. The electric spark igniter is located 13 mm above the center of the specimen. Four seconds after the pyrolysis gases released by the specimen ignite; the electric spark igniter is removed.

The products of combustion and entrained air are collected in a hood and extracted through an exhaust duct by a fan. A gas sample is drawn from the exhaust duct and analyzed for oxygen concentration. The gas temperature and differential pressure across an orifice plate are used for calculating the mass flow rate of the exhaust gases. Smoke production is determined based on the measured light obscuration in the duct using a laser photometer located close to the gas sampling point.

The Cone Calorimeter apparatus, calibration procedure, and test protocol are standardized in the United States as ASTM E 1354-10 and NFPA 271:2009, *Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter* and internationally as ISO 5660-1:2002, *Fire tests—Reaction to fire—Part 1: Rate of heat release from building products—(Cone calorimeter method)*. ASTM E 1354 and NFPA 271 are functionally identical. SwRI's Cone Calorimeter is capable of performing tests in accordance with the ASTM, NFPA, and ISO standards.

Although the test procedure is prescribed in the ASTM, NFPA, and ISO standards, the standards do not specify operating parameters such as heat flux, specimen orientation, use of retainer frame and grid, etc. The choice of a suitable set of parameters is a function of the type and application of the product being tested. These parameters are usually specified by the Client, and are described in Section 4.0 of this report.

3.0 DESCRIPTION OF TEST SPECIMENS

Battelle Memorial Institute provided a material identified as *Resorcinol-Formaldehyde Polymer* for testing in accordance with ASTM E 1354-10. See Table 4 for the test sample description provided by the Client. SwRI received one 10 liter bottle of specimen described in the table below on November 23 and 28, 2011, and December 1 and 3, 2011.

Table 4. Test Sample Identification Description as Provided by Battelle Memorial Institute.

Material ID	Description	Color
Resorcinol-Formaldehyde Polymer	Micro Beads	Red

Specimen preparation was in accordance with ASTM E 1354-10. The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material, and then drying it in an oven. The sample was dried for a minimum of 10 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. After drying, the sample consisted of only the micro bead material; the beads became statically charged in the absence of the water medium and therefore, they were placed in a closed container after drying, in an effort not to lose any beads. Each sample was then conditioned in a controlled environment maintained at 23 °C ± 3 °C and 50% ± 5% relative humidity until just prior to testing.

4.0 TEST PROCEDURE

The critical heat flux of the material was determined by samples exposed to a fixed heat flux for a period of 10 min. If the sample ignited, the heat flux was decreased, and the test repeated. If the sample did not ignite, the heat flux was increased and the test repeated. This process was repeated until the critical heat flux was determined to within ± 0.5 kW/m^2. The Resorcinol-Formaldehyde Polymer, using the ASTM E 1354 standard, had a heat flux of 42 kW/m^2. For the characterization of Resorcinol-Formaldehyde Polymer, using additional thermocouples in the specimens, you may refer to the Appendix.

TEST RESULTS

Testing was conducted at SwRI's Fire Technology Department located in San Antonio, Texas, on November 12, 2011, and December 1 and 3, 2011. Heat release, mass loss, and smoke production data (SPR, SR_1, and TSR) are calculated. The Resorcinol-Formaldehyde Polymer, using the ASTM E 1354 standard, had a heat flux of 42KW/m^2. For the characterization of Resorcinol-Formaldehyde Polymer, using additional thermocouples in the specimens, you may refer to Appendix A.

The following terms and abbreviations are used:

- t_{ig} — time to ignition/sustained flaming (flame over specimen surface for at least 4 s)
- Test Duration — total test duration (time from the start of test until any flaming or other signs of combustion cease, the average mass loss over a 1-min period has dropped below 150 g/m^2, or until 60 min have elapsed)
- C-Factor — calibration constant for oxygen consumption analysis ($m^{1/2}$ -$kg^{1/2}$ -$K^{1/2}$)
- HRR_{peak} — maximum value of the heat release rate per unit area (kW/m^2)
- THR — total amount of heat released per square meter (MJ/m^2)
- HRR_{60s} — average heat release rate over the first 60 s (1 min) after ignition
- HRR_{180s} — average heat release rate over the first 180 s (3 min) after ignition
- HRR_{300s} — average heat release rate over the first 300 s (5 min) after ignition
- $HRR_{30s,\ max}$ — the maximum 30-s sliding average of the heat release rate per unit area (kW/m^2)
- Initial Mass — the initial mass of the test specimen, prior to testing (g)
- Mass at Ignition — the mass of the test specimen at the time of sustained ignition (g)
- Final Mass — the mass of the test specimen at the end of the test (g)
- Mass Loss — total specimen mass loss over the test (g/m^2)
- MLR — average specimen mass loss rate per unit area (g/m^2·s) computed over the test duration
- 10-90 MLR — average specimen mass loss rate per unit area (g/m^2·s) computed over the period starting when 10% of the specimen mass loss occurred and ending when 90% of the specimen mass loss occurred
- EHC — effective heat of combustion (the ratio of heat release rate to mass loss rate–MJ/kg) averaged over the test duration or the entire test if ignition does not occur
- $S_{A,1}$ — smoke production per unit area of exposed specimen (m^2/m^2) prior to ignition
- $S_{A,2}$ — smoke production per unit area of exposed specimen (m^2/m^2) from ignition until flameout or the end of the test; equal to zero if ignition does not occur
- S_A — total smoke production per unit area of exposed specimen during the test duration ($S_{A,1} + S_{A,2}$)
- SEA — specific smoke extinction area (the ratio of smoke production to specimen mass loss–m^2/kg) averaged over the test duration.

APPENDIX A
CONE CALORIMETER DATA
(Consisting of 13 Pages)

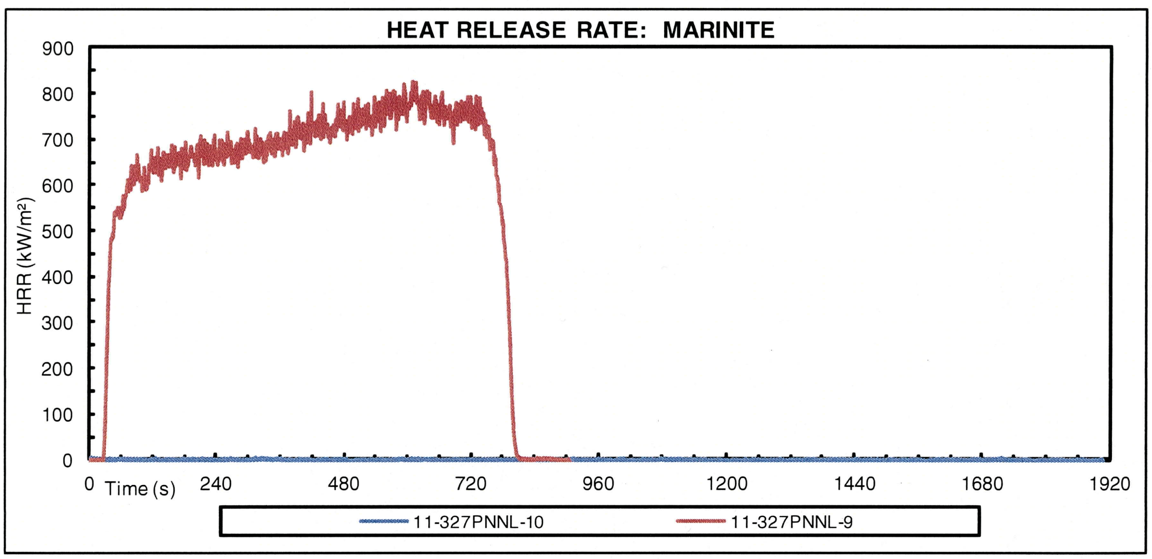
CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	See Notes
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	50 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.00884 m²
Frame:	Yes	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-327PNNL-10	11/23/11	NI	1800	0.0444	7	0.5	1	0	0	1
11-327PNNL-9	11/23/11	24	624	0.0444	826	405.7	455	581	618	787
Average		24	1212	---	416	203.1	228	290	309	394

NI = No Ignition

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
88.4	NI	84.7	417	0.2	0.5	1.3	63	N/A	63	150
213.6	213.3	48.0	18735	30.0	32.2	21.7	1	1289	1290	69
151.0	213.3	66.3	9576	15.1	16.4	11.5	32	1289	676	109



CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	See Notes
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	50 kW/m²

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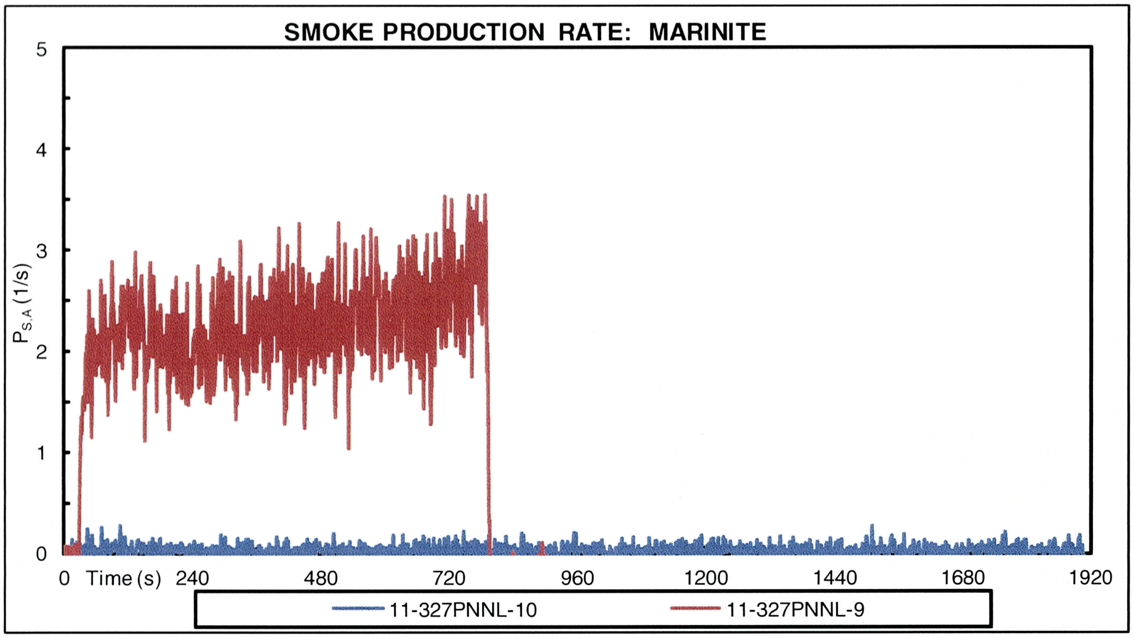
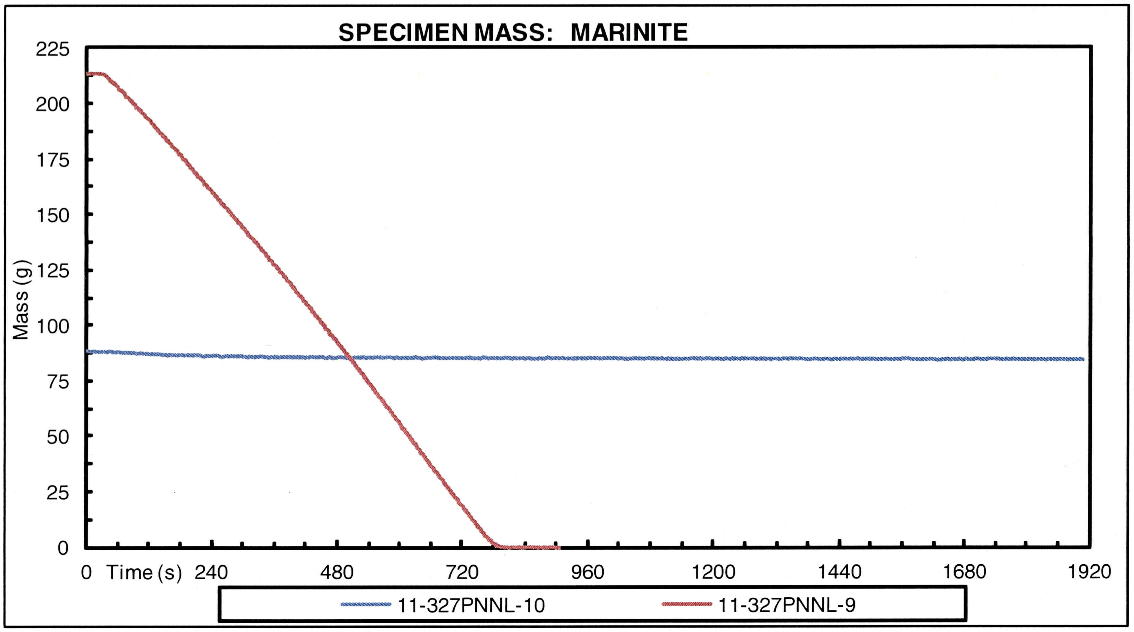




Notes & Observations: 11-327PNNL-9 PMMA Standard
11-327PNNL-10 Marinite Standard

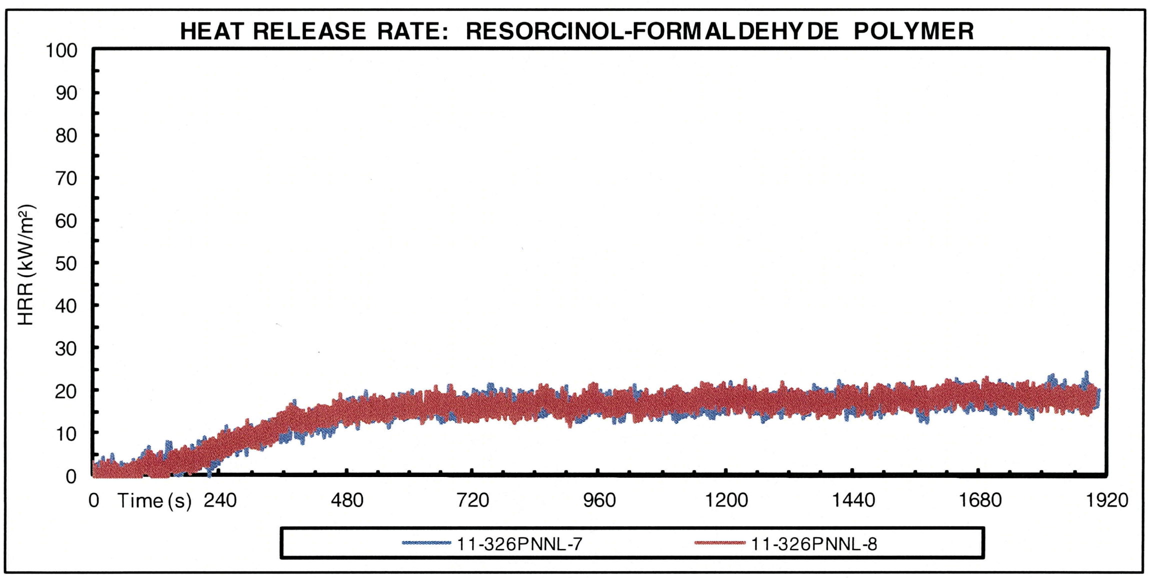
CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	Resorcinol-Formaldehyde polymer
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	20 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-326PNNL-7	11/22/11	NI	1800	0.0439	23	24.6	0	2	3	19
11-326PNNL-8	11/22/11	NI	1800	0.0439	23	25.4	0	1	3	19
Average		NI	NI	---	23	25.0	0	1	3	19

NI = No Ignition

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
114.6	NI	83.3	3127	1.7	1.9	7.9	4	N/A	4	1
114.5	NI	84.0	3046	1.7	1.9	8.3	28	N/A	28	9
114.6	NI	83.7	3086	1.7	1.9	8.1	16	N/A	16	5



CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institute		
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	20 kW/m²

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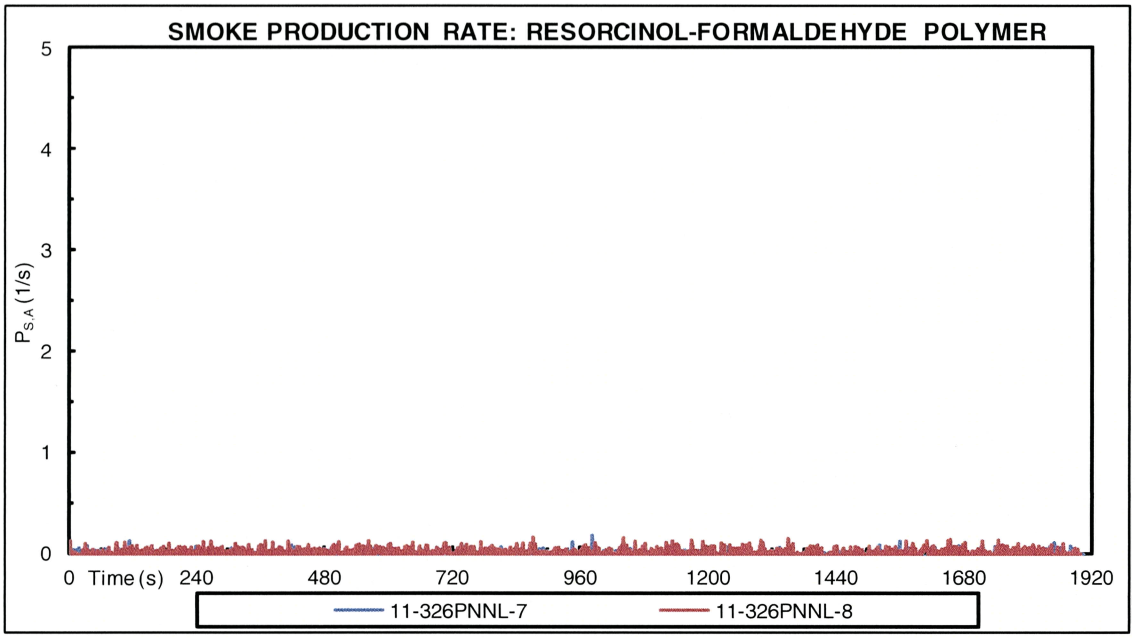
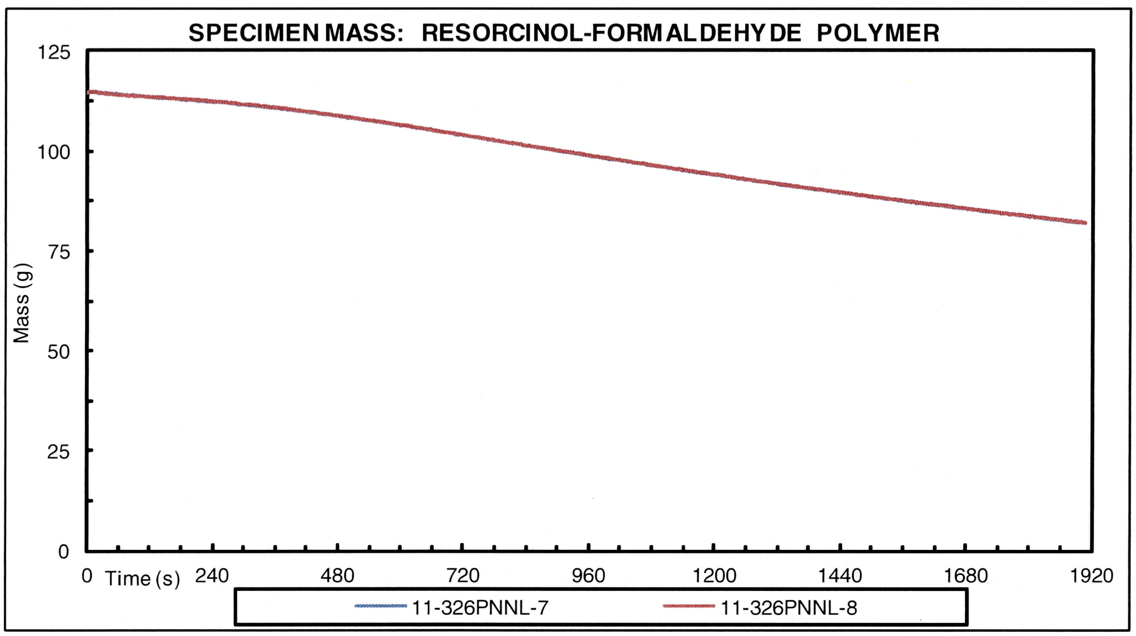




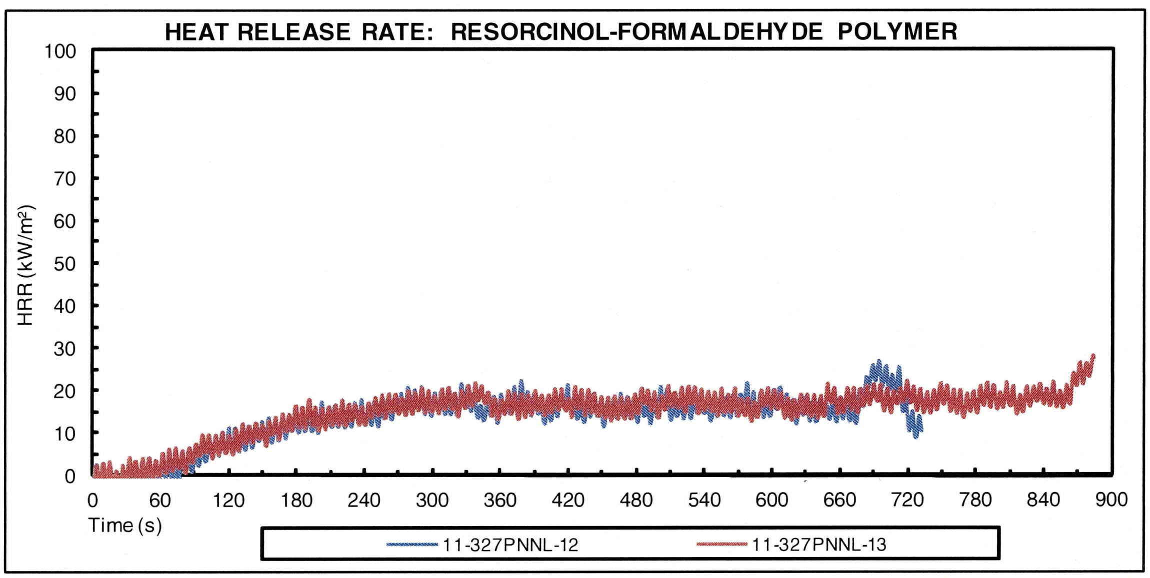
Notes & Observations: 11-326PNNL-7 Regular Run
11-326PNNL-8 Duplicate Run

CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	Resorcinol-Formaldehyde polymer
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	40 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-327PNNL-12	11/23/11	682	733	0.0444	27	9.8				23
11-327PNNL-13	11/23/11	861	892	0.0444	28	13.1				24
Average		772	813	---	28	11.5	-	-	-	23

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
128.3	101.9	100.1	2816	3.9	3.8	3.5	2	0	2	0
127.2	93.6	92.7	3446	3.9	3.9	3.8	23	1	24	0
127.8	97.8	96.4	3131	3.9	3.8	3.6	12	0	13	0



CONE CALORIMETER TEST REPORT

Client: Battelle Memorial Institute
SwRI Project No: 01.13295.12.008c
Material ID: Resorcinol-Formaldehyde polymer
Heat Flux: 40 kW/m²

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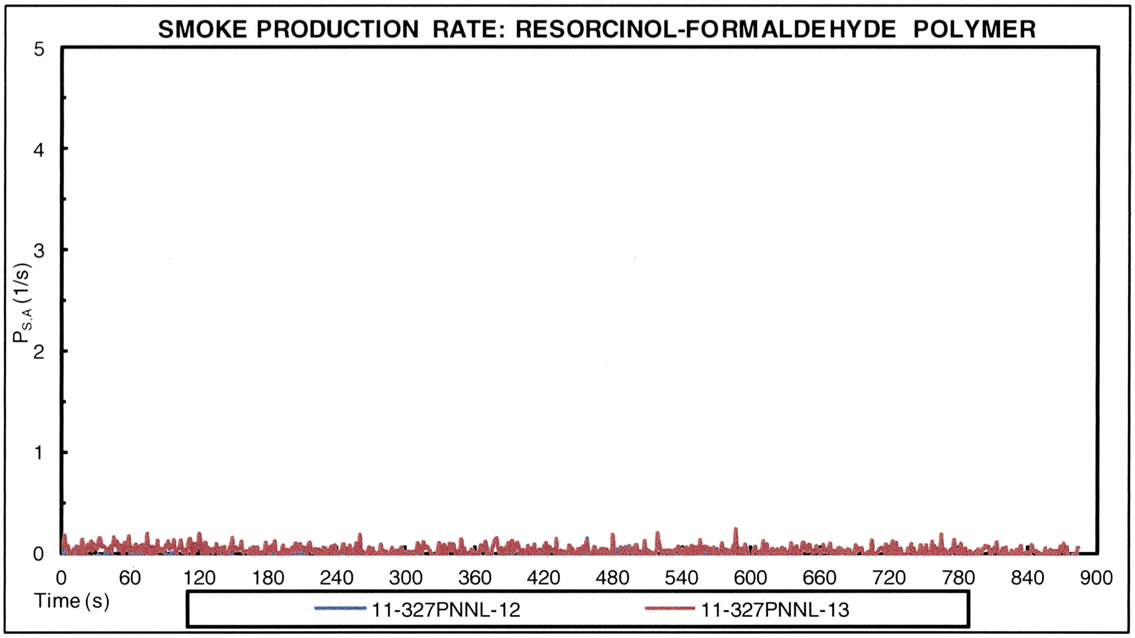
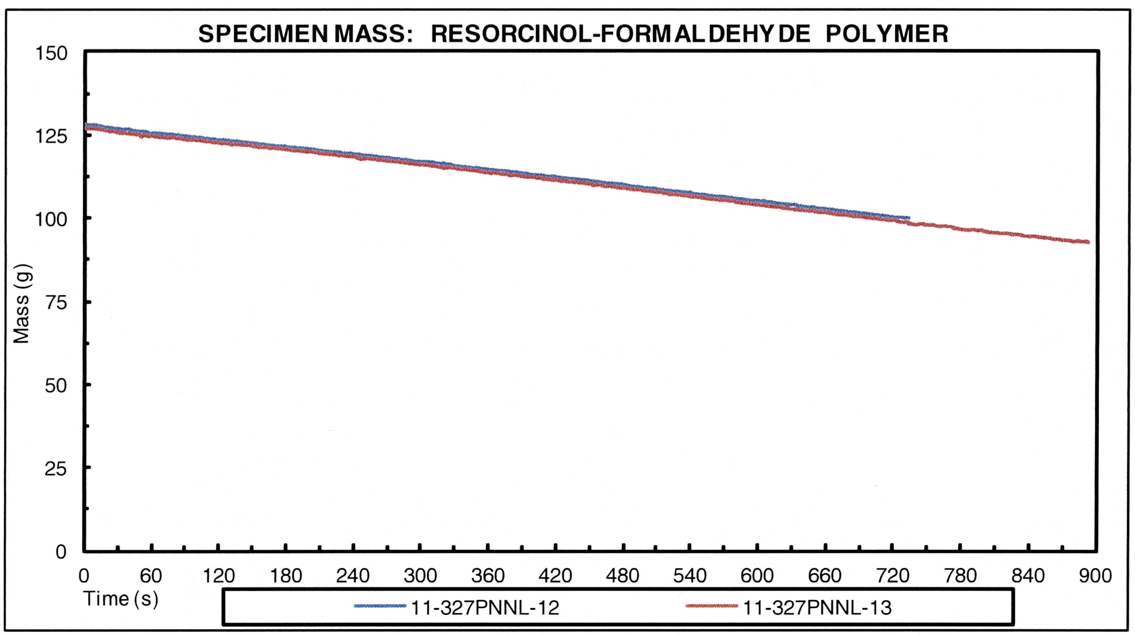




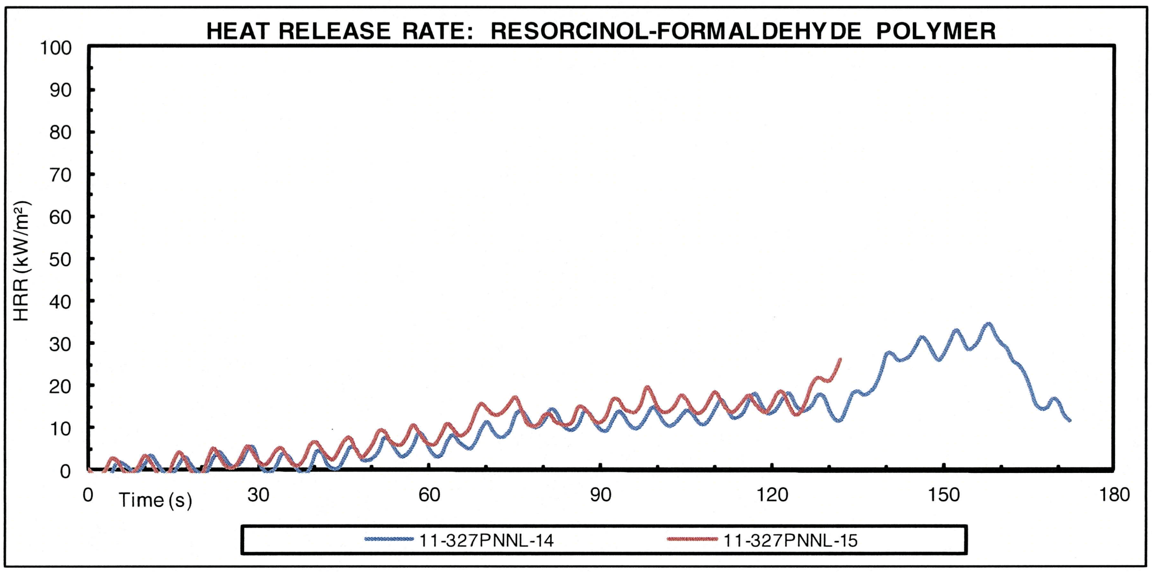
Notes & Observations: 11-327PNNL-12 Regular Run
11-327PNNL-13 Duplicate Run

CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institutec		
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	60 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-327PNNL-14	11/23/11	132	172	0.0444	35	1.9				28
11-327PNNL-15	11/23/11	123	132	0.0444	26	1.2				19
Average		128	152	---	30	1.6	-	-	-	23

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
129.4	121.8	120.0	937	5.5	5.4	2.1	1	0	1	0
126.0	118.1	117.8	819	6.2	6.1	1.5	1	0	2	0
127.7	120.0	118.9	878	5.9	5.7	1.8	1	0	1	0



CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institute		
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	60 kW/m²

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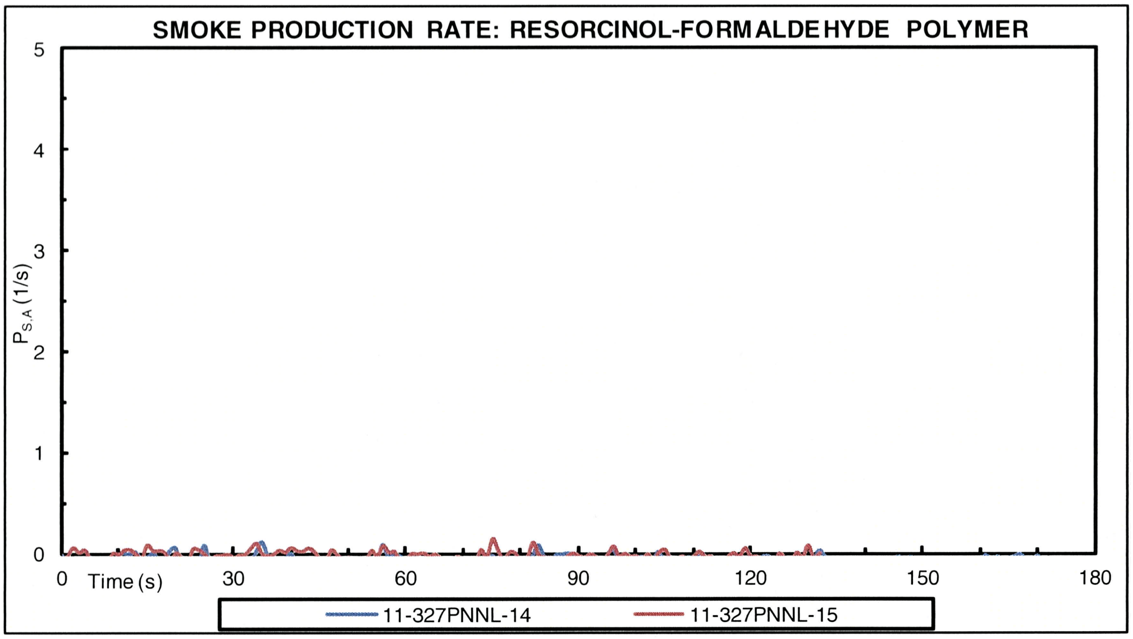
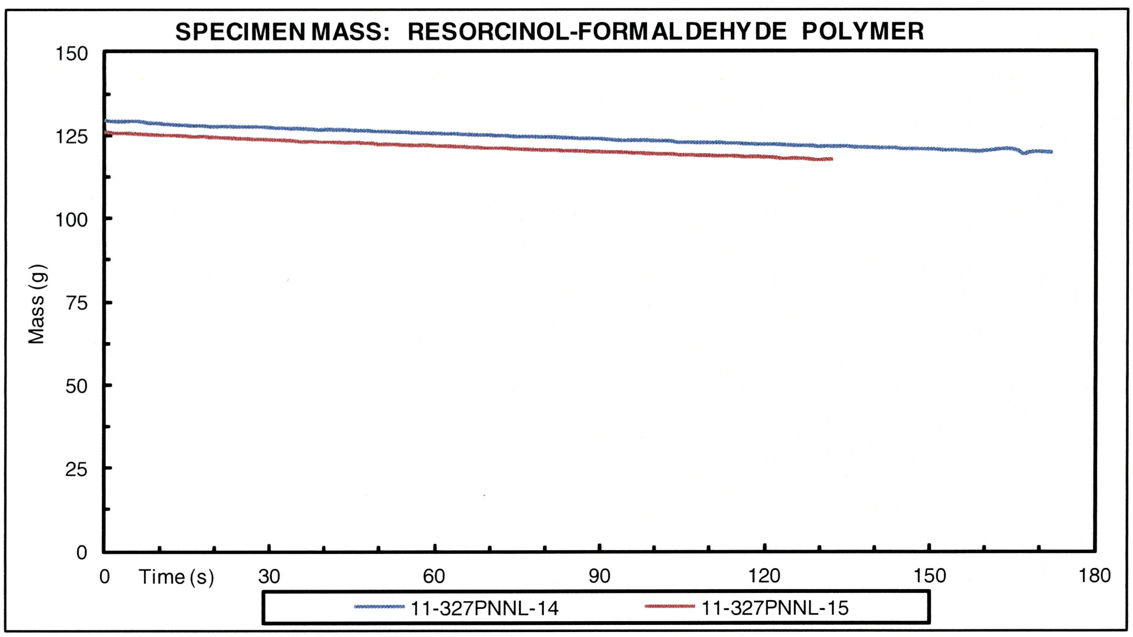




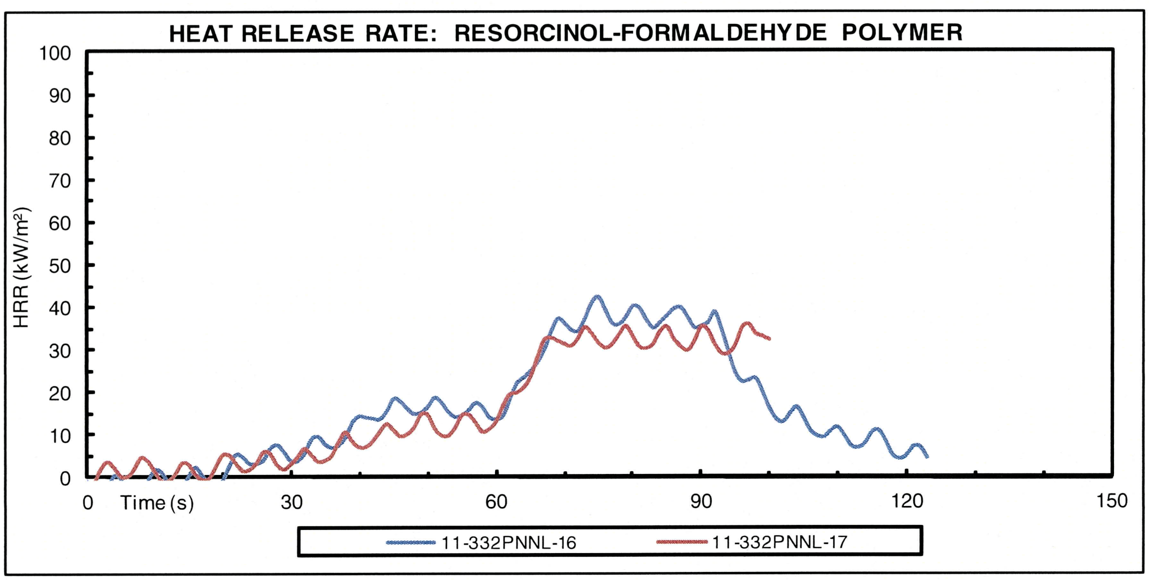
Notes & Observations:
11-327PNNL-14 Regular Run
11-327PNNL-15 Duplicate Run

CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	Resorcinol-Formaldehyde polymer
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	80 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s,\ max}$ (kW/m²)
11-332PNNL-16	11/28/11	66	123	0.0432	42	2.0	-			36
11-332PNNL-17	11/28/11	60	100	0.0432	36	1.5	-			33
Average		63	112	---	39	1.8	-	-	-	34

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
125.2	120.4	118.0	719	5.9	7.1	2.7	2	2	4	3
124.8	120.2	117.5	732	7.6	7.5	2.1	2	1	3	2
125.0	120.3	117.7	725	6.8	7.3	2.4	2	2	4	2



CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institute		
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	80 kW/m²

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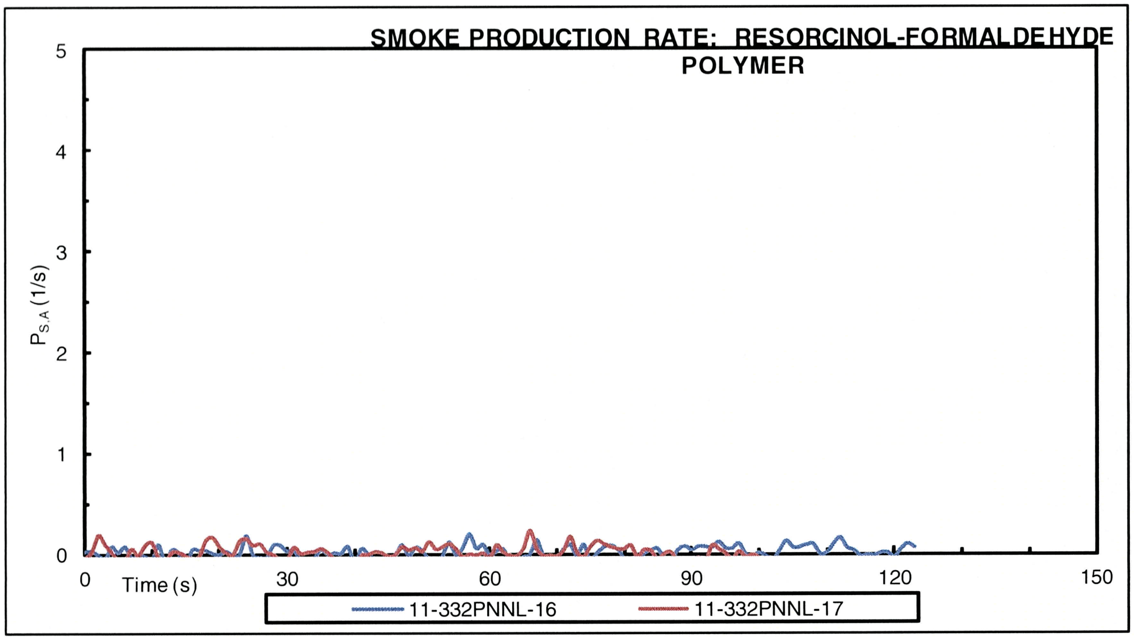
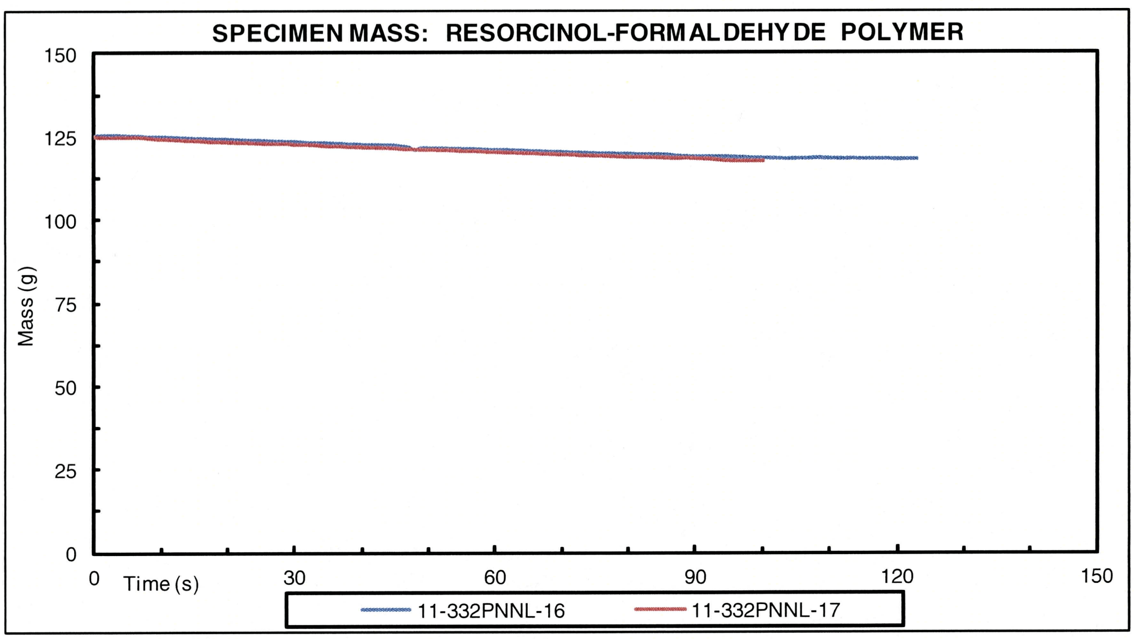




Notes & Observations:

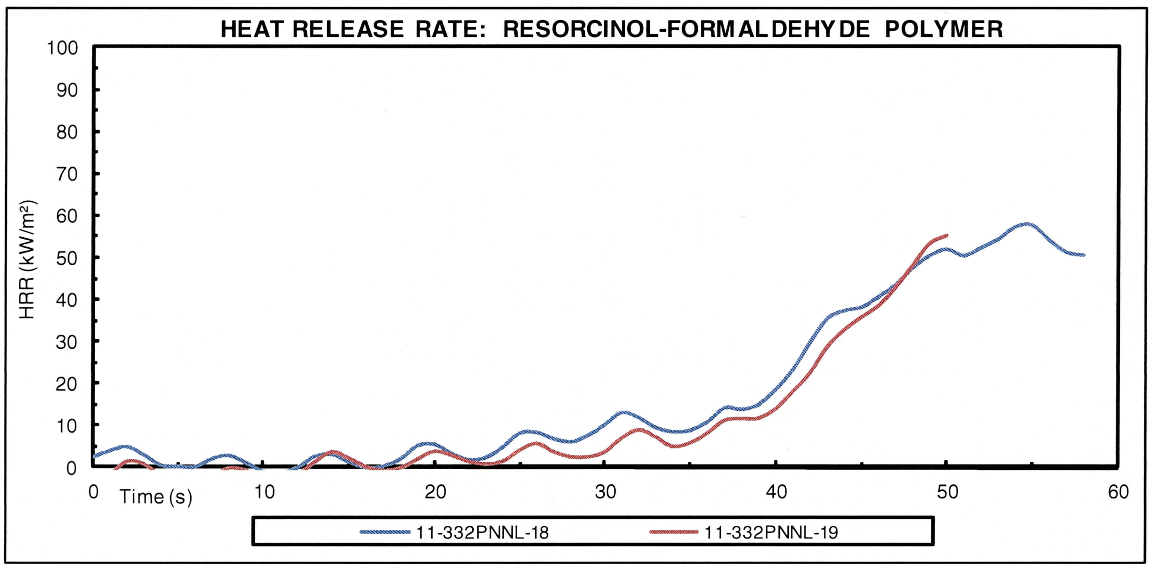
11-332PNNL-16 Regular Run
11-332PNNL-17 Duplicate Run

CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	Resorcinol-Formaldehyde polymer
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	100 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-332PNNL-18	11/28/11	40	58	0.0432	58	1.0				44
11-332PNNL-19	11/28/11	38	58	0.0432	55	0.9				39
Average		39	58	---	56	0.9	#DIV/0!	#DIV/0!	#DIV/0!	42

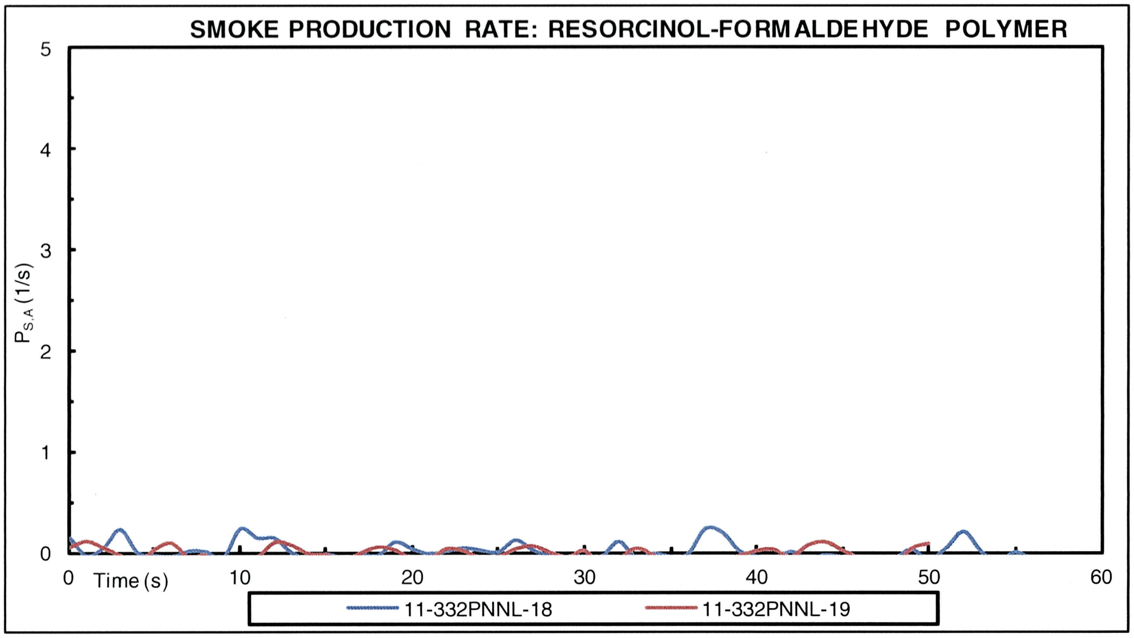
Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
129.3	125.4	123.7	559	8.7	10.4	1.8	2	0	2	1
130.8	127.0	125.0	579	9.8	10.3	1.5	1	1	1	1
130.1	126.2	124.4	569	9.3	10.3	1.7	1	0	2	1

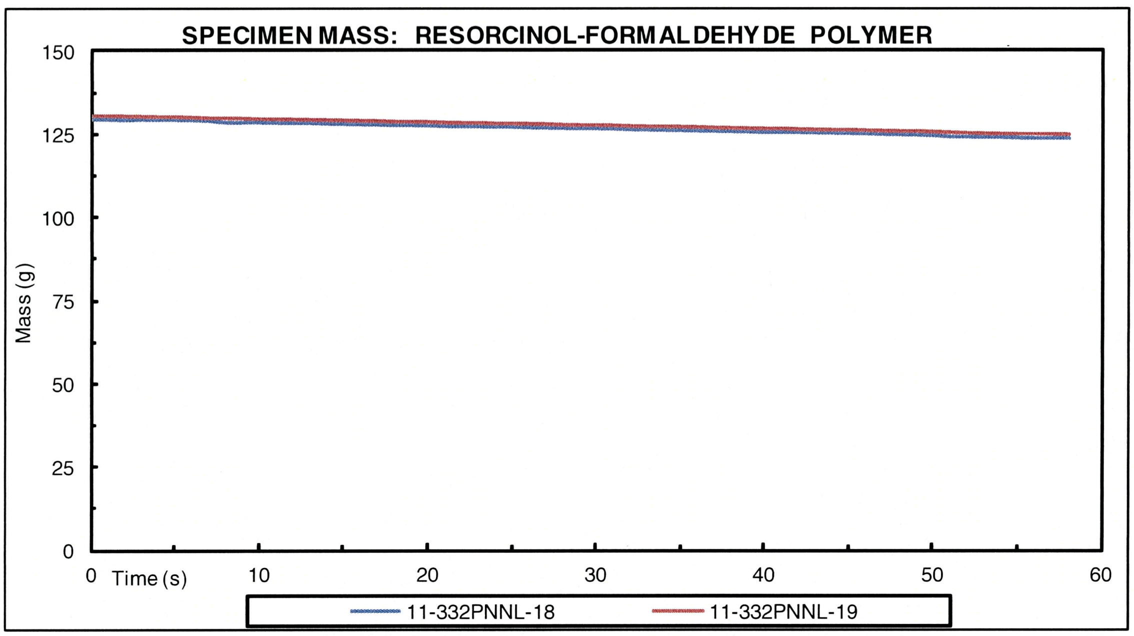


CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institute		
SwRI Project No:	01.13295.12.008c	*Heat Flux:*	100 kW/m²

(Page 2)





Notes & Observations: 11-332PNNL-18 Regular Run
11-332PNNL-19 Duplicate Run

Material	CHF (kW/m²) Bracket	CHF (kW/m²) Extrap.	TRP (kW-s$^{1/2}$/m²)	HRR_{peak} (kW/m²) (@ 50 kW/m²)	HRR_{peak} (kW/m²) (@ 100 kW/m²)	FPI, 50 kW/m² (m$^{5/3}$/kW$^{2/3}$-s$^{1/2}$)	FPI, 100 kW/m² (m$^{5/3}$/kW$^{2/3}$-s$^{1/2}$)
RFP	42.0	40.3	465	44	51	2.64	2.77

Resorcinol-Formaldehyde Polymer (RFP)		
Heat Flux	t_{ig}	$t_{ig}^{-1/2}$
40	682	0.0383
40	861	0.0341
42	938	0.0327
42	1075	0.0305
50	215	0.0682
50	263	0.0617
50	274	0.0604
50	109	0.0958
60	132	0.0870
60	123	0.090166963
80	66	0.123091491
80	60	0.129099445
100	39	0.160128154
100	48	0.144337567
100	40	0.158113883
100	33	0.174077656
100	26	0.196116135
100	26	0.196116135
100	40	0.158113883
100	38	0.162221421

Material ID	T_{ig} (K)	CHF (kW/m²)
RFP-40	733	21.8
RFP-40	811	31.0
RFP-42	862	38.5
RFP-42	949	54.4
RFP-50	705	19.1
RFP-50	832	33.9
RFP-50	884	42.1
RFP-50	430.05	3.3
RFP-60	364.95	1.5
RFP-60	613.95	11.9
RFP-80	488.85	5.4
RFP-80	447.65	3.9
RFP-100	646.65	14.2
RFP-100	364.35	1.5
RFP-Average	873	40.3

Peak Temp (°C)
660.1
612.4
655.2
664.1

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CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008b
(ASTM E 1354 Testing)

SOUTHWEST RESEARCH INSTITUTE®

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CHEMISTRY AND CHEMICAL ENGINEERING DIVISION
FIRE TECHNOLOGY DEPARTMENT
WWW.FIRE.SWRI.ORG
FAX (210) 522-3377

Registered to ISO 9001:2008

FIRE PERFORMANCE EVALUATION IN GENERAL ACCORDANCE WITH ASTM E 1354-10, *STANDARD TEST METHOD FOR HEAT AND VISIBLE SMOKE RELEASE RATES FOR MATERIALS AND PRODUCTS USING AN OXYGEN CONSUMPTION CALORIMETER*

MATERIAL ID: ***RESORCINOL-FORMALDEHYDE POLYMER***

FINAL REPORT
Consisting of 13 Pages

SwRI® Project No. 01.13295.12.008b
Test Dates: November 17, 21, and 22, 2011
Report Date: December 15, 2011

Prepared for:

Battelle Memorial Institute
902 Battelle Blvd, K6-79
P.O. Box 999
Richland, WA 99352

Submitted by:

Eugene F. Horton
Senior Engineering Technologist
Material Flammability Section

Approved by:

FOR: Matthew S. Blais, Ph.D.
Director
Fire Technology Department

1.0 INTRODUCTION

The objective of this test program was to conduct a fire performance evaluation on a material identified as *Resorcinol-Formaldehyde Polymer,* for Battelle Memorial Institute, located in Richland, Washington. The material was tested in general accordance with ASTM E 1354-10, *Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter.* Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative, and a duplicate test (consisting of three runs) were run in addition to the one standard test (consisting of three runs). Testing was conducted at a heat flux of 50 kW/m^2 on the positive and negative standards and 100 kW/m^2 on the Resorcinol-Formaldehyde Polymer. Testing was conducted on November 17, 21, and 22, 2011, at Southwest Research Institute's (SwRI) Fire Technology Department, in San Antonio, Texas.

The negative tests were performed on marinate board; measuring 100 × 100 × 18 mm which is a non-combustible material. This material is often used as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture. The positive tests were performed on PMMA (Poly methyl methacrylate) measuring approximately 100 × 100 × 10 mm. This is a know standard used in testing and it was selected as the positive standard.

This test method is intended to measure and describe the properties of materials or products in response to heat and flame under controlled laboratory conditions. This method is not applicable to end-use products that do not have planar, or nearly planar, external surfaces. The results should not be used alone to describe or appraise the fire hazard or the fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a complete fire hazard assessment or a fire risk assessment, which takes into account all the factors that are pertinent to an assessment of the fire hazard or fire risk of a particular end use. The results apply specifically to the specimens tested, in the manner tested, and not to similar materials, nor to the performance when used in combination with other materials.

2.0 CONE CALORIMETER

For organic solids, liquids, and gases, a nearly constant net amount of heat is released per unit mass of oxygen consumed for complete combustion. An average value for this constant of 13.1 MJ/kg of O_2 can be used for practical applications and is accurate with very few exceptions to within ± 5%. Therefore, measurements of the oxygen consumed in a combustion system can be used to determine the net heat released. This technique, generally referred to as the "oxygen consumption technique", is now the most widely used and accurate method for measuring heat release rate in experimental fires.

The Cone Calorimeter is a sophisticated small-scale test apparatus, which measures the rate of heat release of materials and products under a wide range of conditions using the oxygen consumption technique. A schematic of the instrument is shown in Figure 1. Other useful information obtained from Cone Calorimeter tests includes time to ignition, mass loss rate, smoke production rate, and effective heat of combustion.

In the Cone Calorimeter, a square sample measuring 100 × 100 mm (4 × 4 in.) is exposed to the radiant flux of an electric heater. The heater is in the shape of a truncated cone and is capable of providing heat fluxes to the specimen in the range of 0–100 kW/m². An electric spark igniter is used for piloted ignition of the pyrolysis gases produced by the radiant heater.

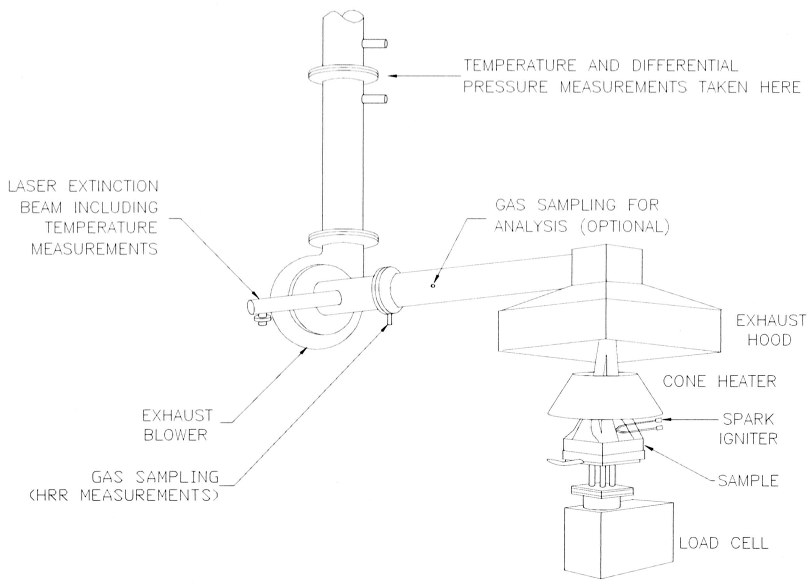


Figure 1. Schematic of the Cone Calorimeter Apparatus.

Test specimens are to be representative of the product's end use and can have a maximum thickness of 50 mm (2 in.). Specimens with a thickness of less than 6 mm are to be tested using a substrate that is representative of end-use conditions. Prior to testing, the test specimen is wrapped in aluminum foil, backed with a layer of low-density refractory fiber blanket, and placed in a standard specimen holder. An optional edge frame can be used to retain the sample within the specimen holder during testing. A load cell is used to measure the mass loss of the specimen throughout the duration of the test.

At the start of a test, the specimen (in the appropriate holder) is placed on the load cell, which is located below the heater. The top edge of the specimen is typically positioned 25 mm below the base plate of the heater. The electric spark igniter is located 13 mm above the center of the specimen. Four seconds after the pyrolysis gases released by the specimen ignite; the electric spark igniter is removed.

The products of combustion and entrained air are collected in a hood and extracted through an exhaust duct by a fan. A gas sample is drawn from the exhaust duct and analyzed for oxygen concentration. The gas temperature and differential pressure across an orifice plate are used for calculating the mass flow rate of the exhaust gases. Smoke production is determined based on the measured light obscuration in the duct using a laser photometer located close to the gas sampling point.

The Cone Calorimeter apparatus, calibration procedure, and test protocol are standardized in the United States as ASTM E 1354-10 and NFPA 271:2009, *Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter* and internationally as ISO 5660-1:2002, *Fire tests—Reaction to fire—Part 1: Rate of heat release from building products—(Cone calorimeter method)*. ASTM E 1354 and NFPA 271 are functionally identical. SwRI's Cone Calorimeter is capable of performing tests in accordance with the ASTM, NFPA, and ISO standards.

Although the test procedure is prescribed in the ASTM, NFPA, and ISO standards, the standards do not specify operating parameters such as heat flux, specimen orientation, use of retainer frame and grid, etc. The choice of a suitable set of parameters is a function of the type and application of the product being tested. These parameters are usually specified by the Client, and are described in Section 4.0 of this report.

3.0 DESCRIPTION OF TEST SPECIMENS

Battelle Memorial Institute, provided a material identified as *Resorcinol-Formaldehyde Polymer* for testing in accordance with ASTM E 1354-10. See Table 1 for the test sample description provided by the Client. SwRI received one 20 liter bottle of specimen described in the table below on November 11, 2011.

Table 1. Test Sample Identification Description as Provided by Battelle Memorial Institute.

Material ID	Description	Color
Resorcinol-Formaldehyde Polymer	Micro Beads	Red

* Measured by SwRI personnel.

Specimen preparation was in accordance with ASTM E 1354-10. The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material, and then drying it in an oven. The sample was dried for a minimum of 10 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. After drying, the sample consisted of only the micro bead material; the beads became statically charged in the absence of the water medium and therefore, they were placed in a closed container after drying, in an effort not to lose any beads. Each sample was then conditioned in a controlled environment maintained at 23 °C ± 3 °C and 50% ± 5% relative humidity until just prior to testing.

4.0 TEST PROCEDURE

Two triplicate tests were performed in general accordance with ASTM E 1354-10 at a heat flux of 100 kW/m². Single runs were conducted on PMMA & Marinite standards at a heat flux of 50 kW/m². Tests were conducted in the horizontal orientation. The data collection sampling interval was set at 1 s. Tests were concluded 2 min after flameout.

5.0 TEST RESULTS

Testing was conducted at SwRI's Fire Technology Department located in San Antonio, Texas, on November 17, 21, and 22, 2011. Cone Calorimeter test results are summarized in tabular and graphical form in the standard SwRI ASTM E 1354 Cone Calorimeter data sheets provided in Appendix A. Heat release, mass loss, and smoke production data (SPR, SR_1, and TSR) are calculated. The following terms and abbreviations are used:

- t_{ig} — time to ignition/sustained flaming (flame over specimen surface for at least 4 s)
- Test Duration — total test duration (time from the start of test until any flaming or other signs of combustion cease, the average mass loss over a 1-min period has dropped below 150 g/m², or until 60 min have elapsed)
- C-Factor — calibration constant for oxygen consumption analysis ($m^{1/2}$ -$kg^{1/2}$ -$K^{1/2}$)
- HRR_{peak} — maximum value of the heat release rate per unit area (kW/m²)
- THR — total amount of heat released per square meter (MJ/m²)
- HRR_{60s} — average heat release rate over the first 60 s (1 min) after ignition
- HRR_{180s} — average heat release rate over the first 180 s (3 min) after ignition
- HRR_{300s} — average heat release rate over the first 300 s (5 min) after ignition
- $HRR_{30s,\ max}$ — the maximum 30-s sliding average of the heat release rate per unit area (kW/m²)
- Initial Mass — the initial mass of the test specimen, prior to testing (g)
- Mass at Ignition — the mass of the test specimen at the time of sustained ignition (g)
- Final Mass — the mass of the test specimen at the end of the test (g)
- Mass Loss — total specimen mass loss over the test (g/m^2)
- MLR — average specimen mass loss rate per unit area (g/m²·s) computed over the test duration
- 10-90 MLR — average specimen mass loss rate per unit area (g/m²·s) computed over the period starting when 10% of the specimen mass loss occurred and ending when 90% of the specimen mass loss occurred
- EHC — effective heat of combustion (the ratio of heat release rate to mass loss rate– MJ/kg) averaged over the test duration or the entire test if ignition does not occur
- $S_{A,1}$ — smoke production per unit area of exposed specimen (m²/m²) prior to ignition
- $S_{A,2}$ — smoke production per unit area of exposed specimen (m²/m²) from ignition until flameout or the end of the test; equal to zero if ignition does not occur
- S_A — total smoke production per unit area of exposed specimen during the test duration ($S_{A,1} + S_{A,2}$)

- SEA specific smoke extinction area (the ratio of smoke production to specimen mass loss–m^2/kg) averaged over the test duration.

Appendix A

CONE CALORIMETER DATA

(Consisting of 6 Pages)

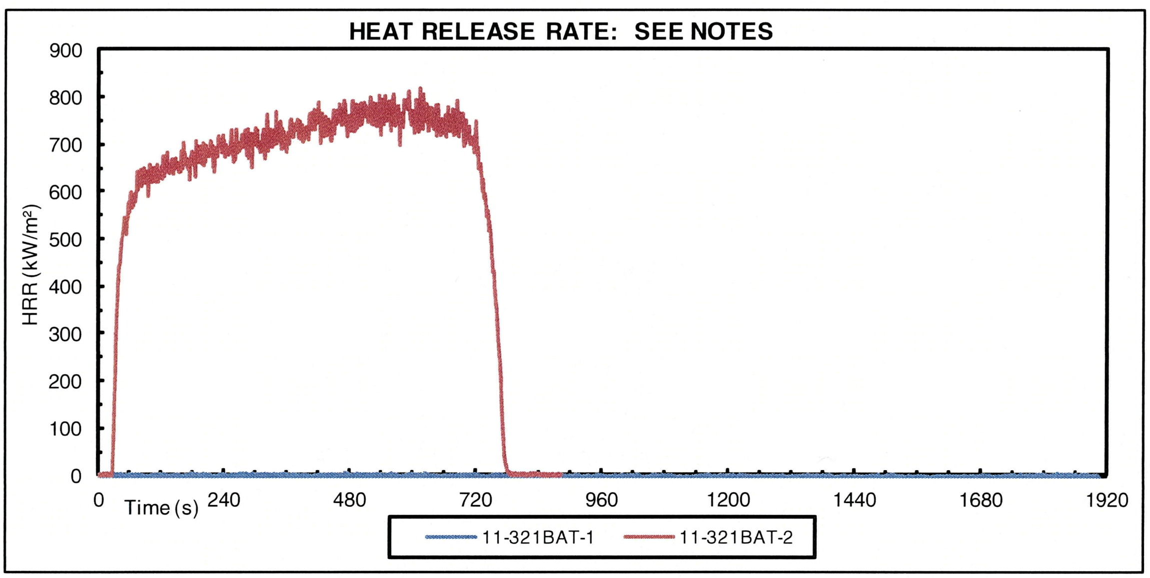
CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	See Notes
SwRI Project No:	01.13295.12.008b	*Heat Flux:*	50 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.00884 m²
Frame:	Yes	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-321BAT-1	11/17/11	NI	1800	0.0437	5	0.2	-1	-1	-1	0
11-321BAT-2	11/17/11	23	623	0.0437	819	414.1	455	587	631	771
Average		23	1212	---	412	207.1	227	293	315	386

NI = No Ignition

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
87.4	NI	83.3	465	0.2	0.3	0.3	42	N/A	42	91
208.1	207.9	38.9	19139	30.7	32.9	21.6	1	1377	1377	72
147.8	207.9	61.1	9802	15.5	16.6	11.0	22	1377	710	81



CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	See Notes
SwRI Project No:	01.13295.12.008b	*Heat Flux:*	50 kW/m²

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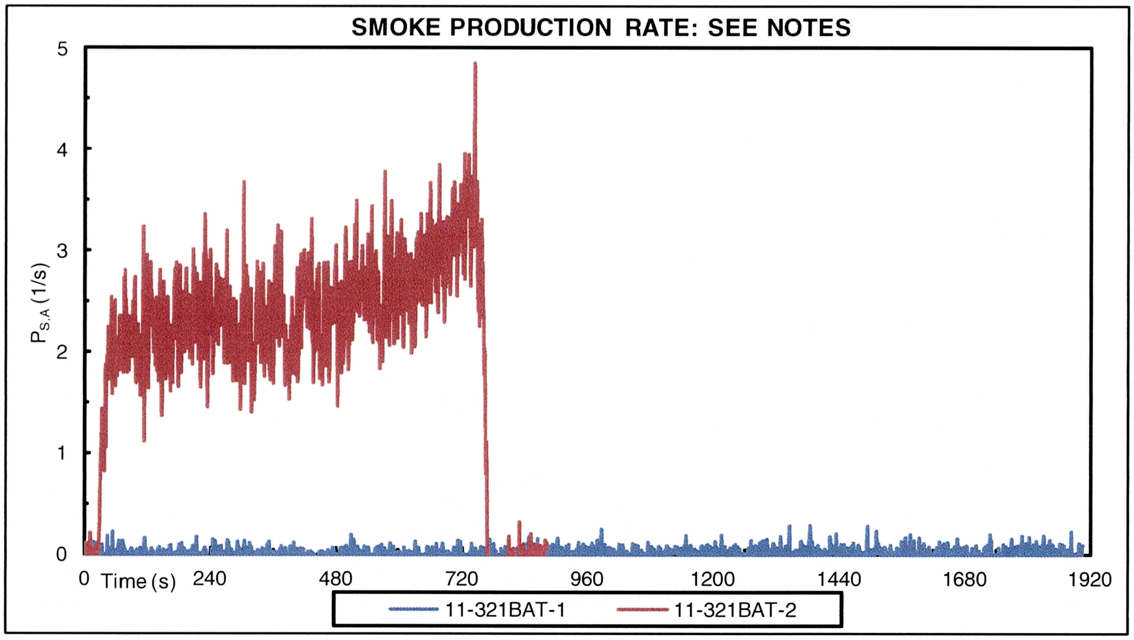
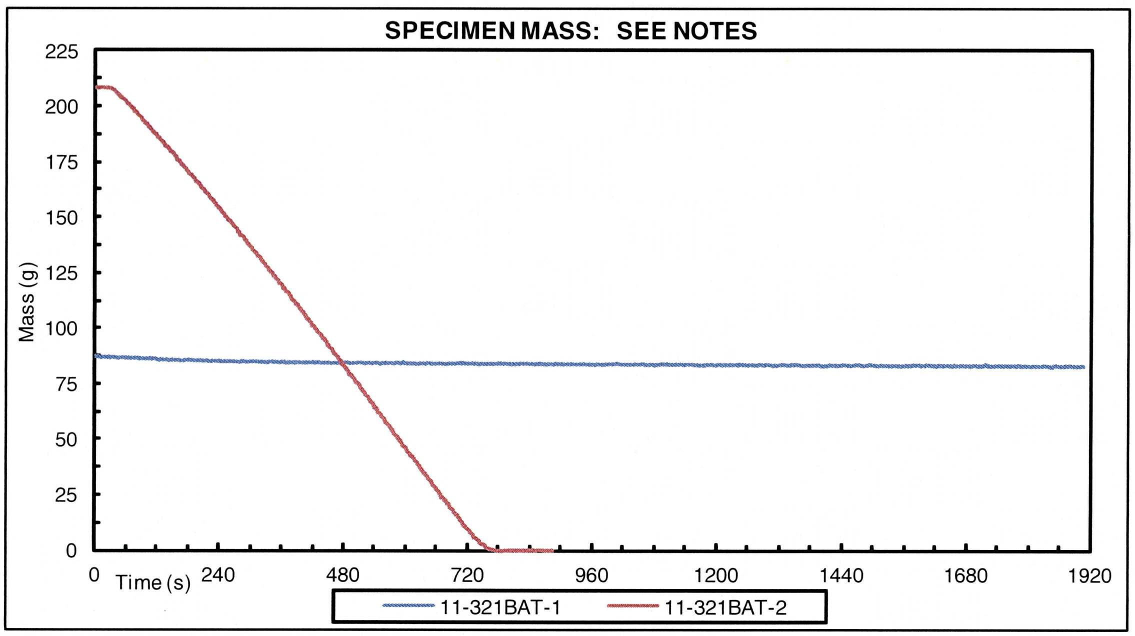




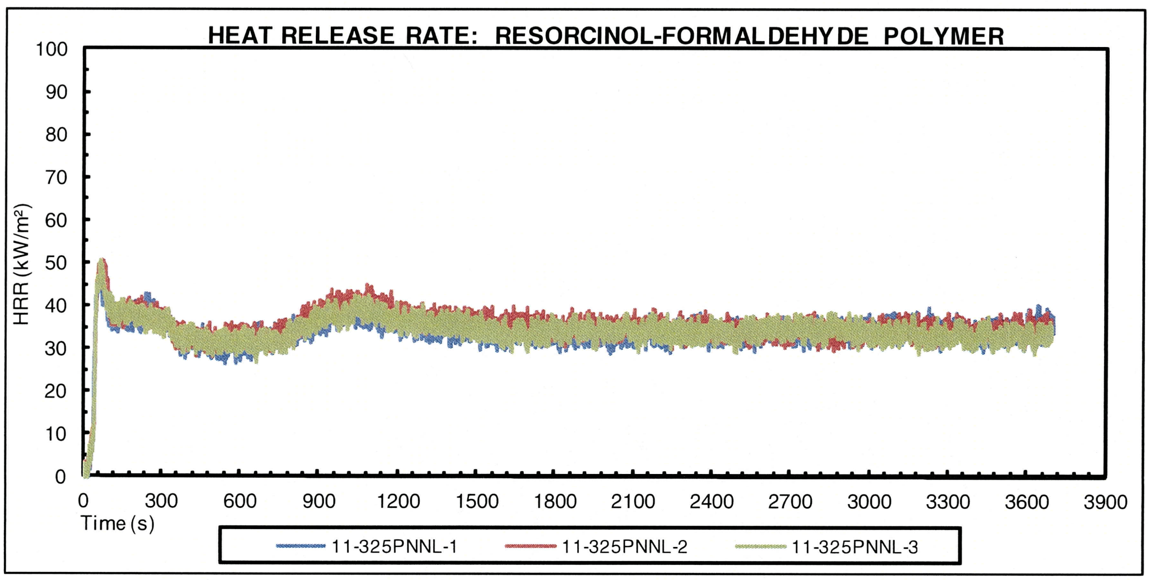
Notes & Observations: 11-321BAT-1 Marinate Standard
11-321BAT-2 PMMA Standard

CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	Resorcinol-Formaldehyde polymer
SwRI Project No:	01.13295.12.008b	*Heat Flux:*	100 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-325PNNL-1	11/21/11	39	3600	0.0436	48	120.6	41	38	38	44
11-325PNNL-2	11/21/11	48	3600	0.0436	51	125.3	44	40	39	47
11-325PNNL-3	11/21/11	40	3600	0.0436	50	121.9	42	39	38	46
Average		42	3600	---	50	122.6	42	39	38	46

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
125.2	121.6	27.6	9756	2.7	3.0	12.4	0	13	14	1
128.8	123.9	30.2	9859	2.7	3.0	12.7	1	83	84	8
128.7	124.6	30.5	9817	2.7	3.0	12.4	1	129	129	13
127.6	123.4	29.5	9810	2.7	3.0	12.5	1	75	76	8



CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institute		
SwRI Project No:	01.13295.12.008b	*Heat Flux:*	100 kW/m²

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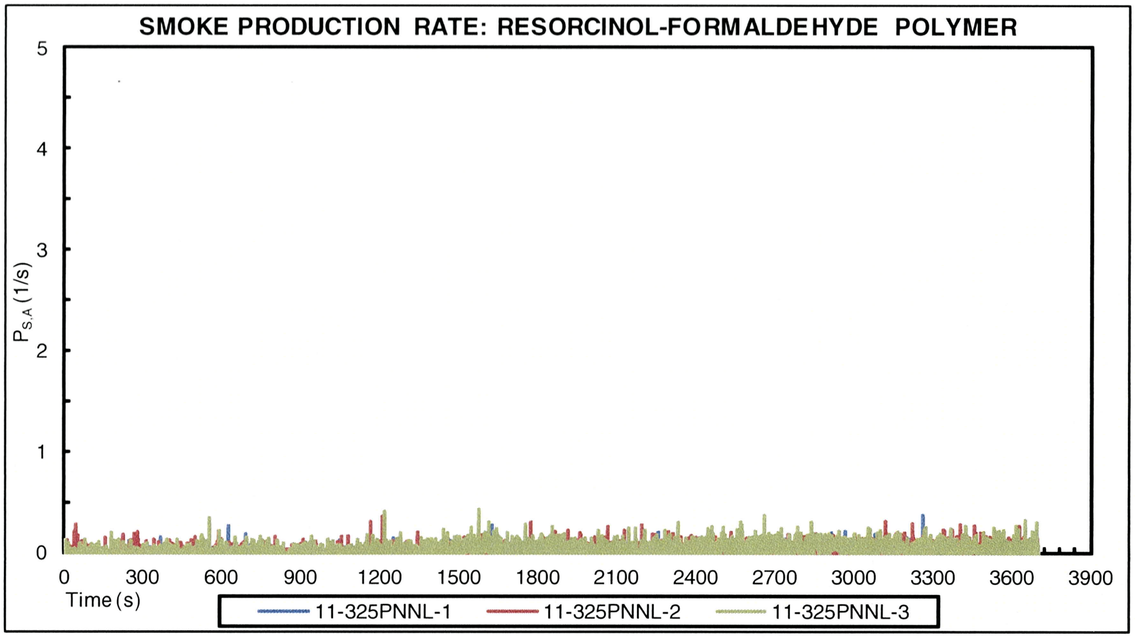
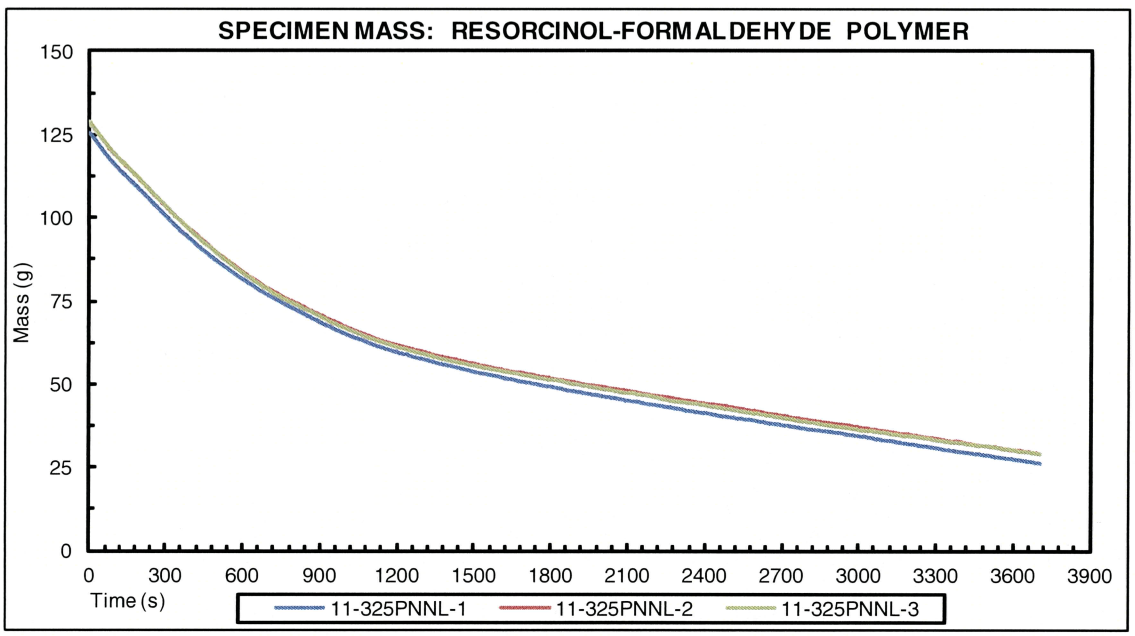




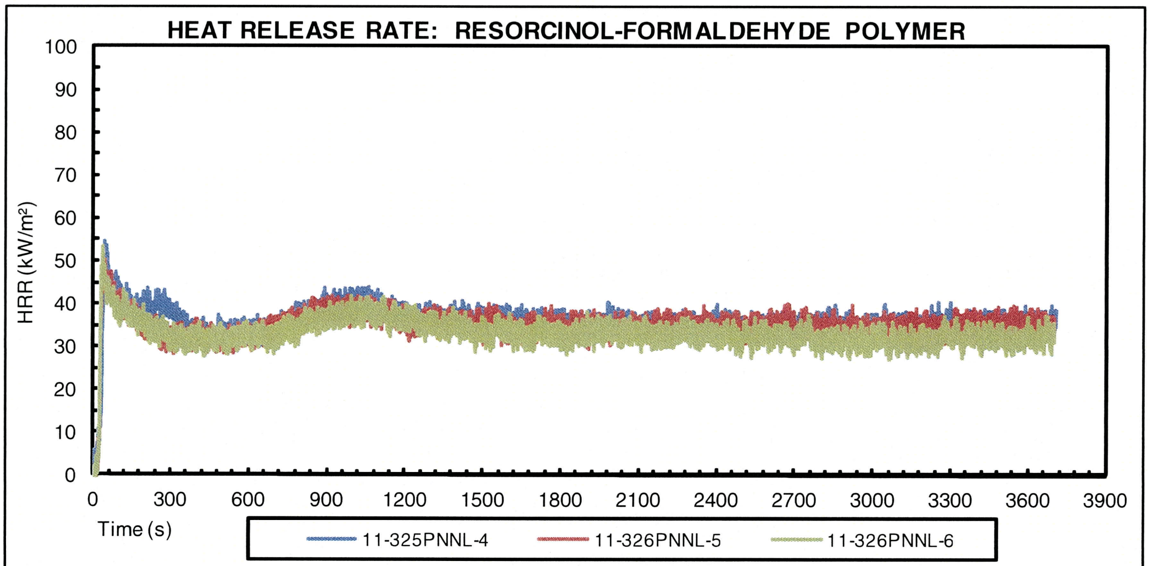
Notes & Observations:

CONE CALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*Material ID:*	Resorcinol-Formaldehyde polymer
SwRI Project No:	01.13295.12.008b	*Heat Flux:*	100 kW/m²
		Duct Flow:	24 l/s
Orientation:	Horizontal	*Sample Area:*	0.01000 m²
Frame:	No	*Distance:*	25 mm
Spark Igniter:	Yes	*Operator:*	Alan K. Lowry

Test ID	Test Date	t_{ig} (s)	Test Duration (s)	C-Factor (SI Units)	HRR_{peak} (kW/m²)	THR (MJ/m²)	HRR_{60s} (kW/m²)	HRR_{180s} (kW/m²)	HRR_{300s} (kW/m²)	$HRR_{30s, max}$ (kW/m²)
11-325PNNL-4	11/21/11	33	3600	0.0436	55	128.1	45	42	41	49
11-326PNNL-5	11/22/11	26	3600	0.0439	50	125.0	43	39	37	46
11-326PNNL-6	11/22/11	26	3600	0.0439	53	119.2	43	39	37	46
Average		28	3600	---	53	124.1	44	40	38	47

Initial Mass (g)	Mass at Ignition (g)	Final Mass (g)	Mass Loss (g/m²)	MLR (g/m²-s)	10-90 MLR (g/m²-s)	EHC (MJ/kg)	$S_{A,1}$ (m²/m²)	$S_{A,2}$ (m²/m²)	S_A (m²/m²)	SEA (m²/kg)
124.1	121.1	32.2	9186	2.6	2.8	13.9	2	197	199	21
119.8	117.4	31.3	8847	2.5	2.6	14.1	1	244	245	28
119.7	117.9	36.6	8315	2.3	2.4	14.3	0	34	34	4
121.2	118.8	33.4	8782	2.5	2.6	14.1	1	158	159	18



CONE CALORIMETER TEST REPORT

		Material ID:	Resorcinol-Formaldehyde polymer
Client:	Battelle Memorial Institute		
SwRI Project No:	01.13295.12.008b	*Heat Flux:*	100 kW/m^2

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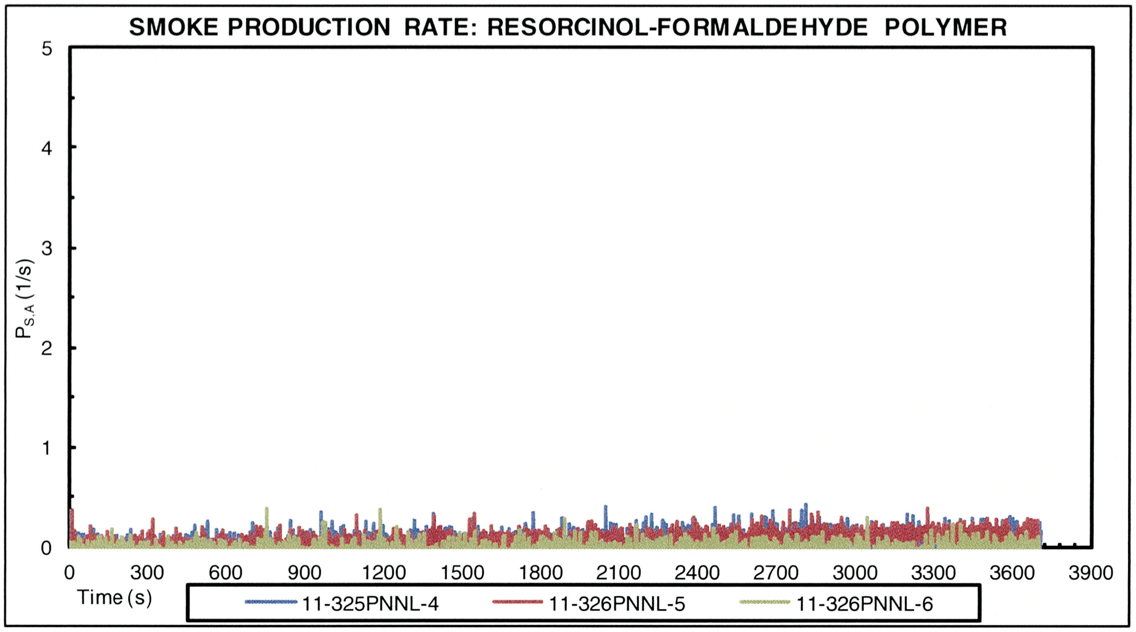

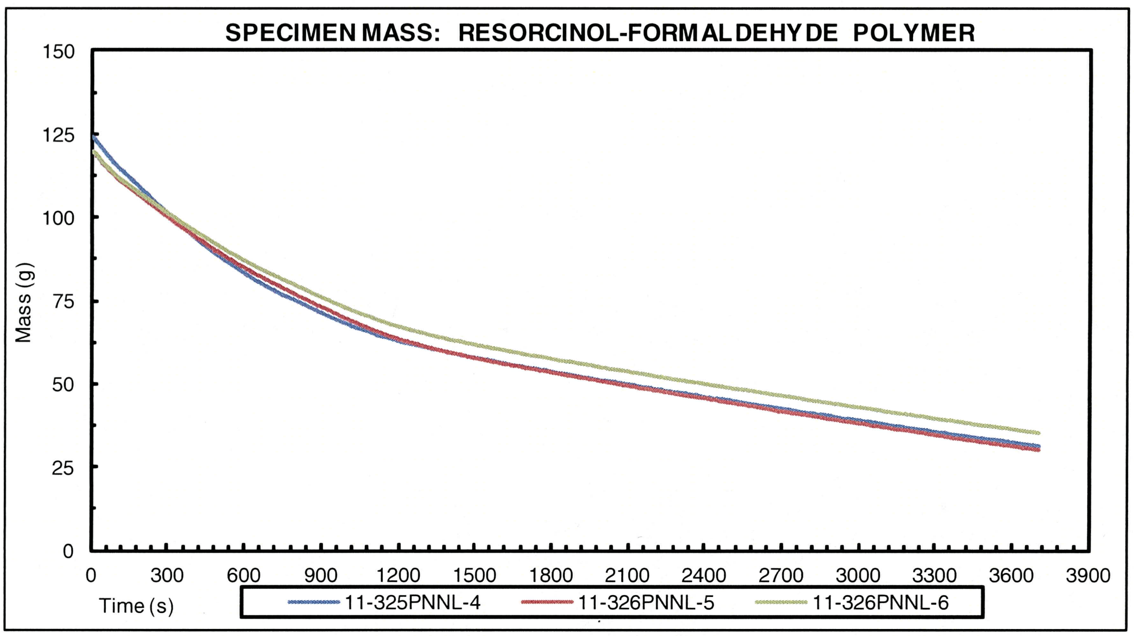

Notes & Observations: This triplicate run is a duplicate test as per HASQARD requirements.

SOUTHWEST RESEARCH INSTITUTE

CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008d
(ASTM D 7309 Testing)

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CHEMISTRY AND CHEMICAL ENGINEERING DIVISION
FIRE TECHNOLOGY DEPARTMENT
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FIRE PERFORMANCE EVALUATION OF *RESORCINOL-FORMALDEHYDE* POLYMER TESTED IN ACCORDANCE WITH ASTM D 7309-07a, *STANDARD TEST METHOD FOR DETERMINING FLAMMABILITY CHARACTERISTICS OF PLASTICS AND OTHER SOLID MATERIALS USING MICROSCALE COMBUSTION CALORIMETERY*

FINAL REPORT
Consisting of 15 Pages

SwRI® Project No. 01.13295.12.008d
Test Dates: November 16–18, 2011
Report Date: December 16, 2011

Prepared for:

Battelle Memorial Institute
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Director
Fire Technology Department

HOUSTON, TEXAS (713) 977-1377 • WASHINGTON, DC (301) 881-0226

1.0 INTRODUCTION

This report describes a small-scale fire test conducted on a material identified as *Resorcinol-Formaldehyde Polymer* in accordance with ASTM D 7309-07a, *Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry*, (Test Method A), for Battelle Memorial Institute, located in Richland, Washington. Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative, and duplicate tests were run, in addition to the one standard test. Testing was conducted on November 16–18, 2011, at the Fire Technology Department of Southwest Research Institute (SwRI), located in San Antonio, Texas.

The positive tests were performed on PMMA (Poly methyl methacrylate). This is a know standard used in testing and it was selected as the positive standard. The negative tests were performed on marinate board, which is a non-combustible material. This material often is used as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture.

The test method described in ASTM D 7309-07a is intended to measure and describe the properties of materials or products in response to heat and flame under controlled laboratory conditions. The results should not be used alone to describe or appraise the fire hazard or the fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a complete fire hazard for fire risk assessment, which takes into account all the factors that are pertinent to an assessment of the fire hazard or risk of a particular end-use.

The results presented in this report apply specifically to the specimens tested, in the manner tested, and not to the entire production of these or similar materials, nor to the performance when used in combination with other materials.

2.0 DESCRIPTION OF TEST APPARATUS AND PROCEDURE

The Microscale Combustion Calorimeter is a sophisticated small-scale test apparatus, which measures the rate of heat release of materials and products using the oxygen consumption technique.

For organic solids, liquids, and gases a nearly constant net amount of heat is released per unit mass of oxygen consumed for complete combustion. An average value for this constant of 13.1 MJ/kg of O_2 can be used for practical applications and is accurate with very few exceptions to within ± 5%. Therefore, measurements of the oxygen consumed in a combustion system can be used to determine the net heat released. This technique, generally referred to as the "oxygen consumption technique", is now the most widely used and accurate method for measuring heat release rate in experimental fires.

The ASTM D 7309-07a apparatus (see Figure 1) consists primarily of two heating chambers and a specimen holder (a part of the specimen thermocouple post). The heating chambers are two sections of a vertical tube; the sample sits in the pyrolysing chamber, and the pyrolysis gases move up towards the combustor chamber.

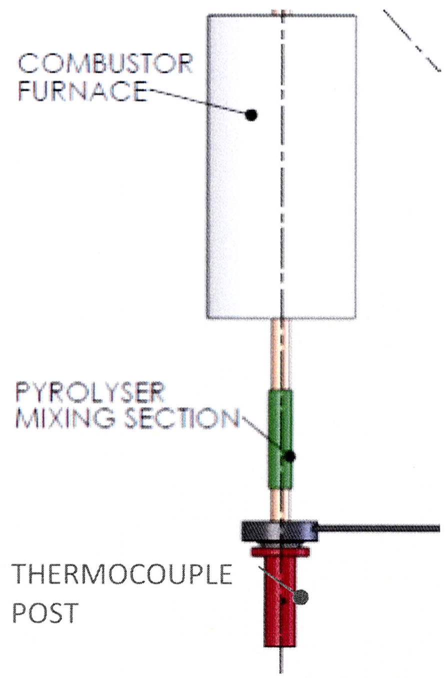


Figure 1. Schematic of Microcalorimeter.

The combustor chamber is typically set at 900 °C. The pyrolysis chamber is set at 75 °C and is ramped up to 850 °C at a specified heating rate β, which is typically set to 1 K/s. The ASTM D 7309-07a specifies two different test methods: Method A is controlled thermal decomposition, and Method B is controlled thermal oxidative decomposition. The primary difference between the two methods is where the oxygen is introduced. In Method A, the purge gas nitrogen flows through the pyrolyser section then mixes with oxygen and the nitrogen/oxygen mixture along with any pyrolysis gases from the sample travel through the combustor furnace. In this method, the heat of combustion of the volatile components (gases) is measured, but not the heat of combustion of any solid residue. In Method B, the nitrogen/oxygen mixture is introduced into the pyrolyser section. In this method, the net calorific value of the specimen gases and solid residue are measured. Because Method A can be used to obtain the heat release capacity η_c (J/g-K), which is a flammability parameter unique to this test method, Method A is typically used.

The heat release rate is calculated based on oxygen consumption, and the specimen mass loss is determined based on weight measurements before and after testing.

3.0 DESCRIPTION OF TEST SPECIMENS

Battelle Memorial Institute provided one 10 liter bottle of specimen, identified as *Resorcinol-Formaldehyde Polymer*, and it was received by SwRI on November 11, 2011. A description of the material provided by the Client can be found in Table 1.

Table 1. Test Sample Description Provided by the Client.

Material ID	Sample Description	Color
Resorcinol-Formaldehyde Polymer	Micro Beads	Red

Prior to testing, the specimen was prepared in accordance with the standard to an approximate weight of 3 g. The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material and then drying it in an oven. After drying, the sample consisted of only the micro bead material; the beads became statically charged in the absence of the water medium and therefore, they were placed in a closed container after drying, in an effort not to lose any beads. Each sample was then conditioned in a controlled environment maintained at 23 °C ± 3 °C and 50% ± 5% relative humidity until just prior to testing.

4.0 TEST RESULTS

Triplicate testing was conducted on November 16–18, 2011, by Nathaniel Ramos and Alan Lowry. Tabular test data and graphs of the measured heat release plotted with respect to temperatures and time are presented in Appendix A.

APPENDIX A

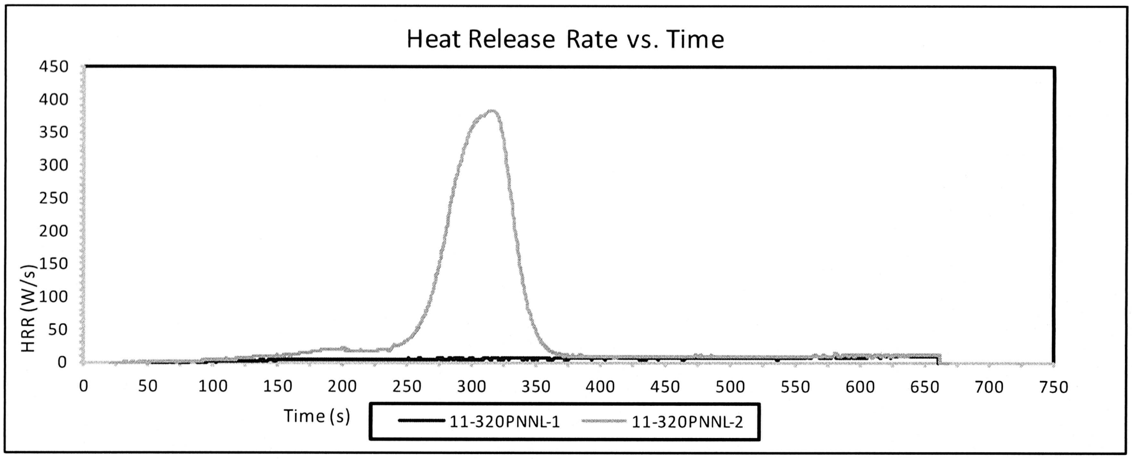
MICROCALORIMETER DATA SHEETS

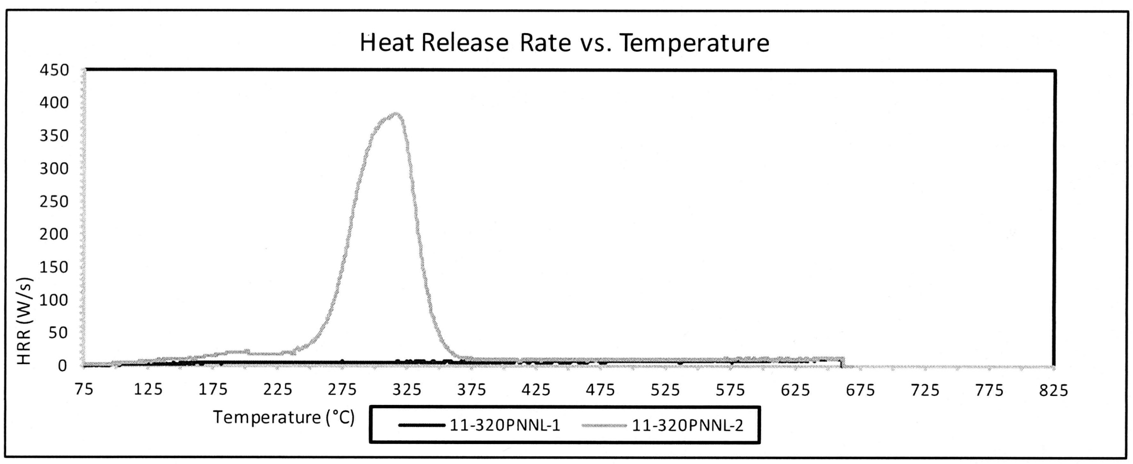
(CONSISTING OF 10 PAGES)

MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	See Notes	*Test Method:*	ASTM D7309/A
Preparation:	November 16, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-320PNNL-1	November 17, 2011	2.65	2.65	9.668	1009.775	9.668	3350.30425	1	-
11-320PNNL-2	November 17, 2011	2.46	0	383.783	673.684	383.783	27652.6835	0	27652.68
Average		2.555	1.325	196.726	841.7295	196.7255	15501.4939	0.5	27652.68



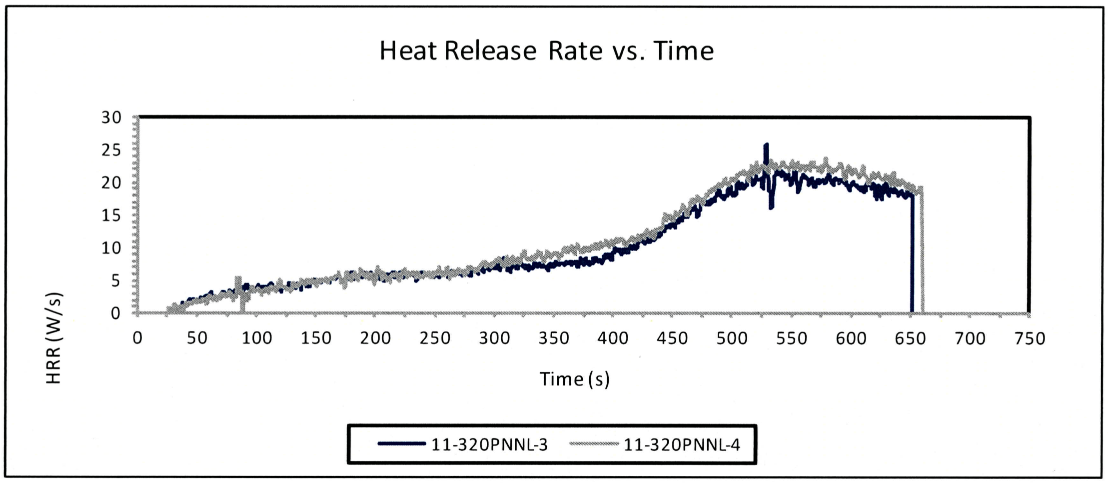


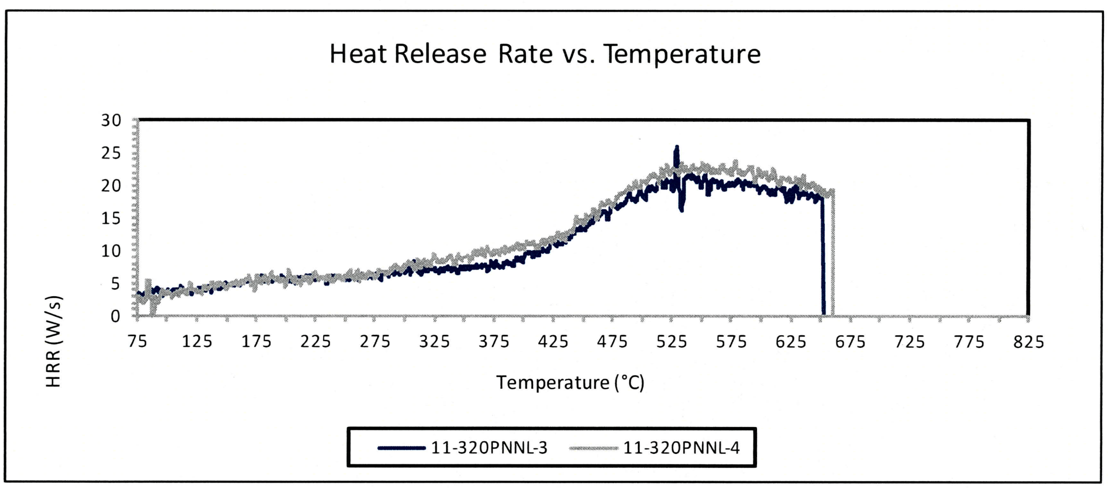
NOTES:
Silver: 11-320PNNL-1
PMMA: 11-320PNNL-2

MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer	*Test Method:*	ASTM D7309/A
Preparation:	November 17, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-320PNNL-3	November 16, 2011	2.55	1.45	25.944	898.249	25.944	6373.7685	0.568627	14775.55
11-320PNNL-4	November 16, 2011	2.43	1.44	23.734	939.012	23.734	7079.18325	0.592593	17376.18
Average		2.49	1.445	24.839	918.6305	24.839	6726.47588	0.58061	16075.87

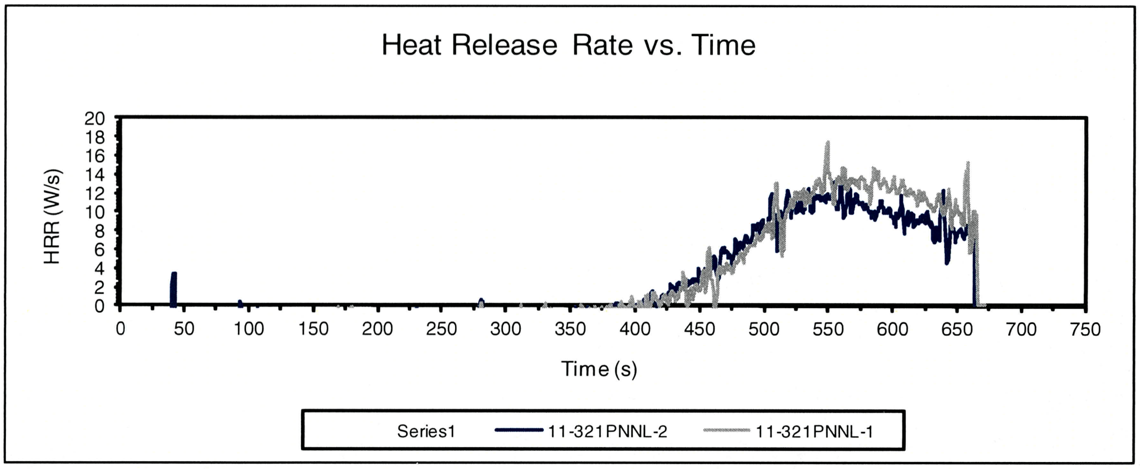


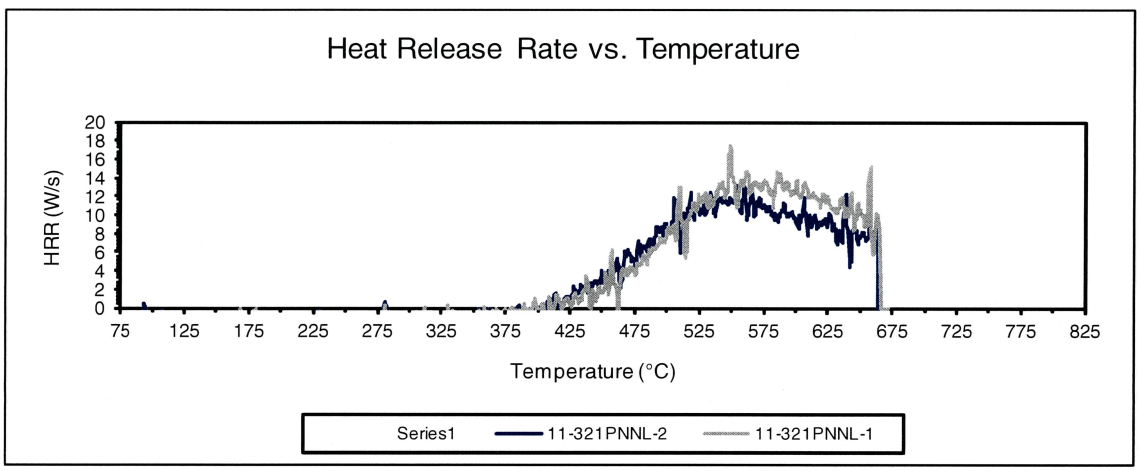


MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer	*Test Method:*	ASTM D7309/A
Preparation:	November 17, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-321PNNL-1	November 17, 2011	2.46	1.39	17.509	904.262	17.509	2060.3068	0.565041	4736.78
11-321PNNL-2	November 17, 2011	2.33	1.4	13.228	917.423	13.228	1173.086	0.600858	2939.02
Average		2.395	1.395	15.3685	910.843	15.3685	1616.6964	0.58295	3837.9



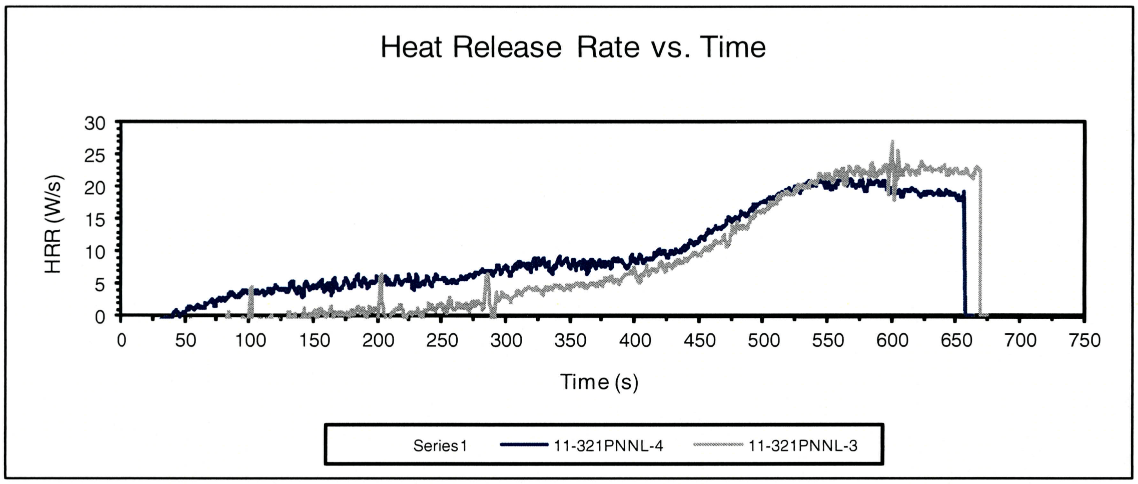


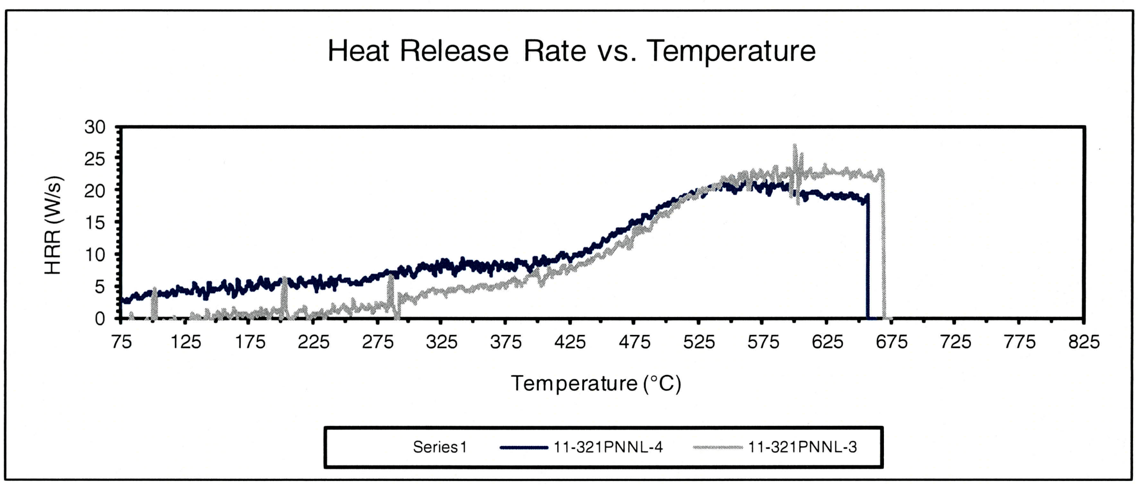
Note: Duplicate Run

MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer	*Test Method:*	ASTM D7309/A
Preparation:	November 17, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-321PNNL-4	November 17, 2011	2.58	1.54	22.172	914.779	22.172	6292.5048	0.596899	15610.3
11-321PNNL-3	November 17, 2011	2.58	1.44	27.092	952.411	27.092	5244.4058	0.55814	11868.9
Average		2.58	1.49	24.632	933.595	24.632	5768.4553	0.577519	13739.6

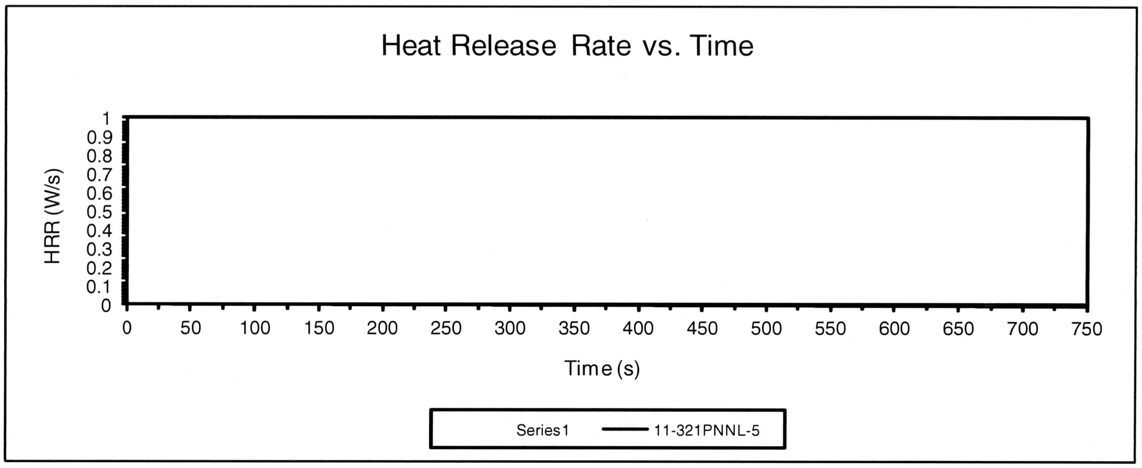


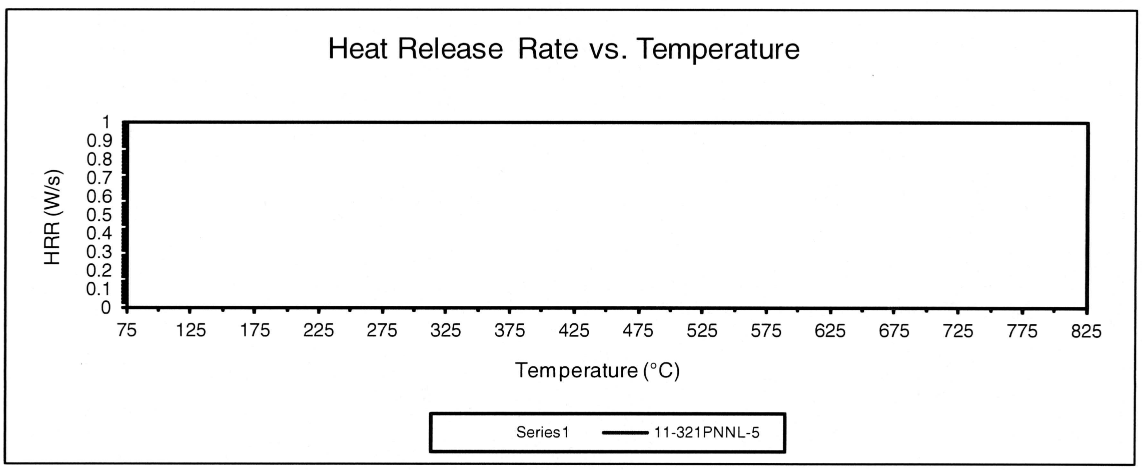


MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Silver, Negative Standard	*Test Method:*	ASTM D7309/B
Preparation:	November 17, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-321PNNL-5	November 17, 2011	2.65	2.65	0	1021.63	0	-27.55975	1	–
Average		2.65	2.65	0	1021.63	0	-27.55975	1	–

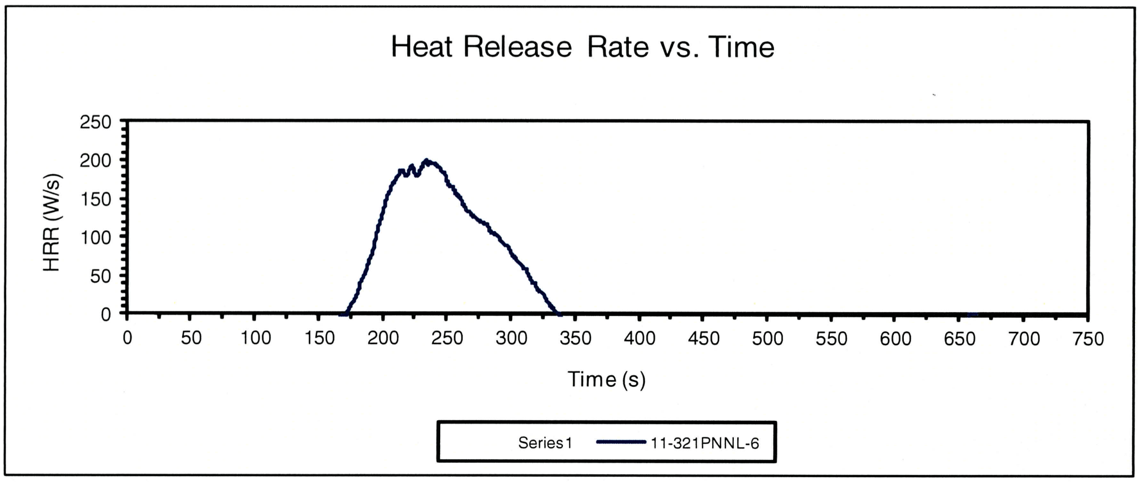


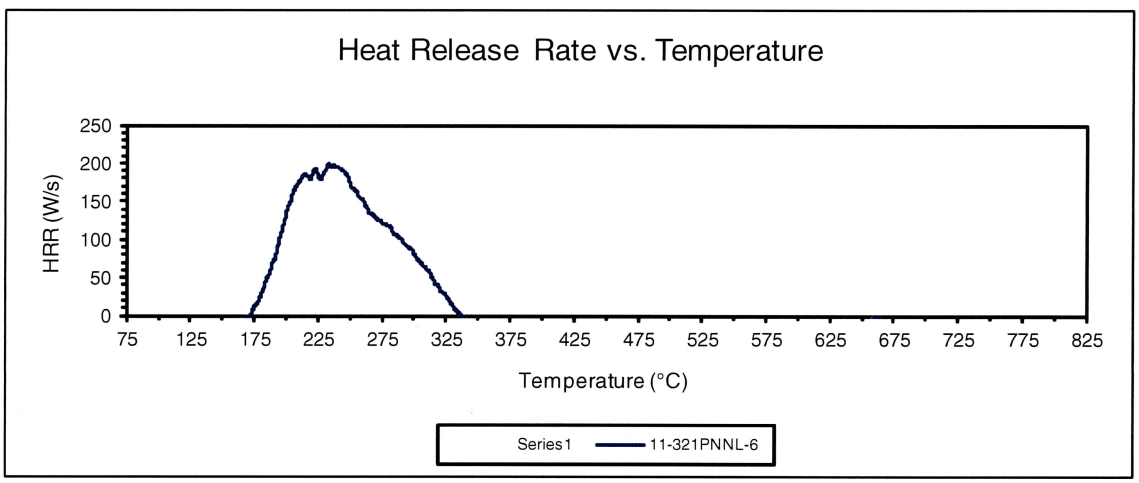


MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	PMMA, Positive Standard	*Test Method:*	ASTM D7309/B
Preparation:	November 17, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-321PNNL-6	November 17, 2011	2.4	0	199.459	595.79	199.459	18347.795	0	18347.8
Average		2.4	0	199.459	595.79	199.459	18347.795	0	18347.8

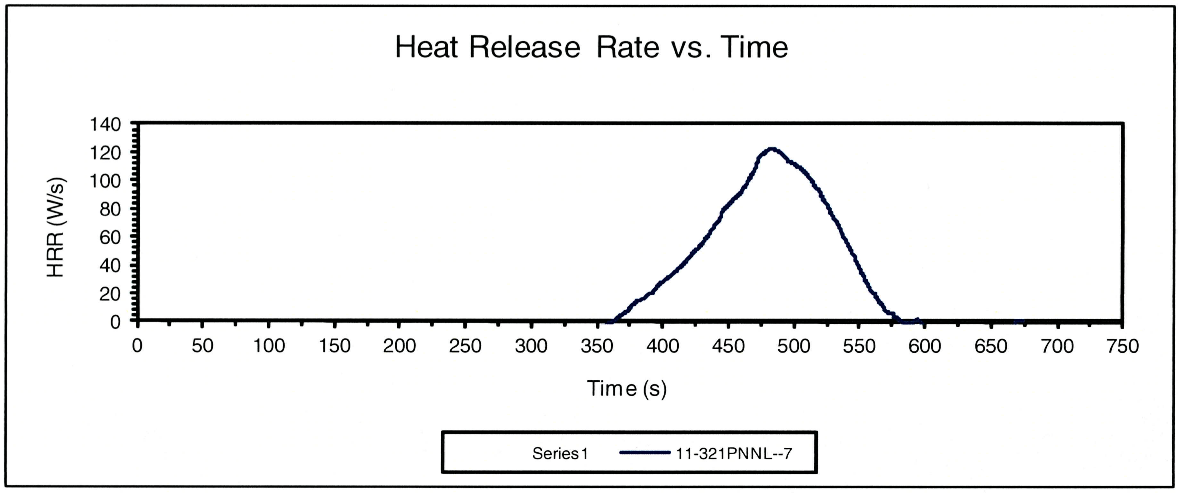


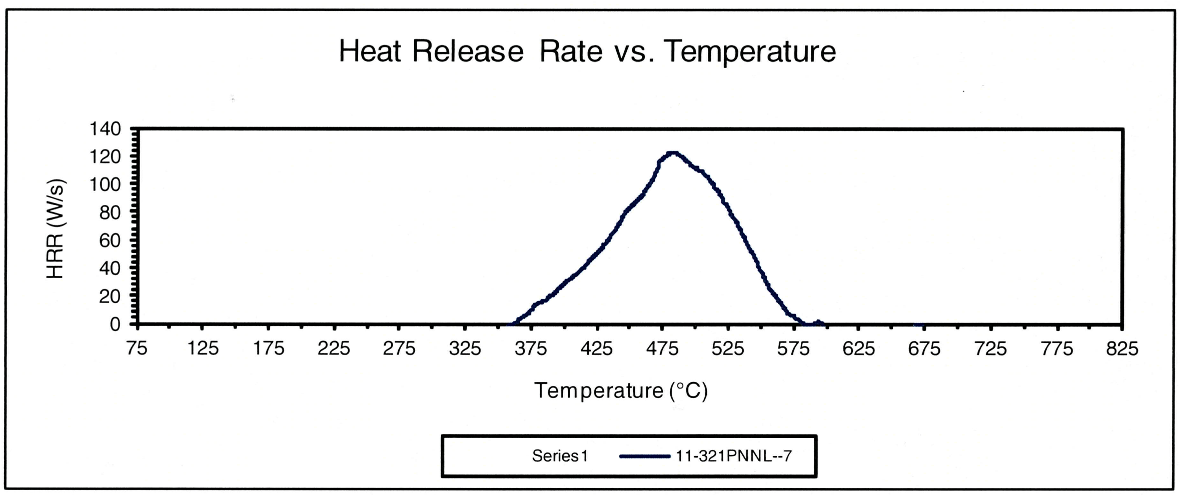


MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer	*Test Method:*	ASTM D7309/B
Preparation:	November 17, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-321PNNL--7	November 17, 2011	2.34	0	122.904	838.435	122.904	13034.482	0	13034.5
Average		2.34	0	122.904	838.435	122.904	13034.482	0	13034.5

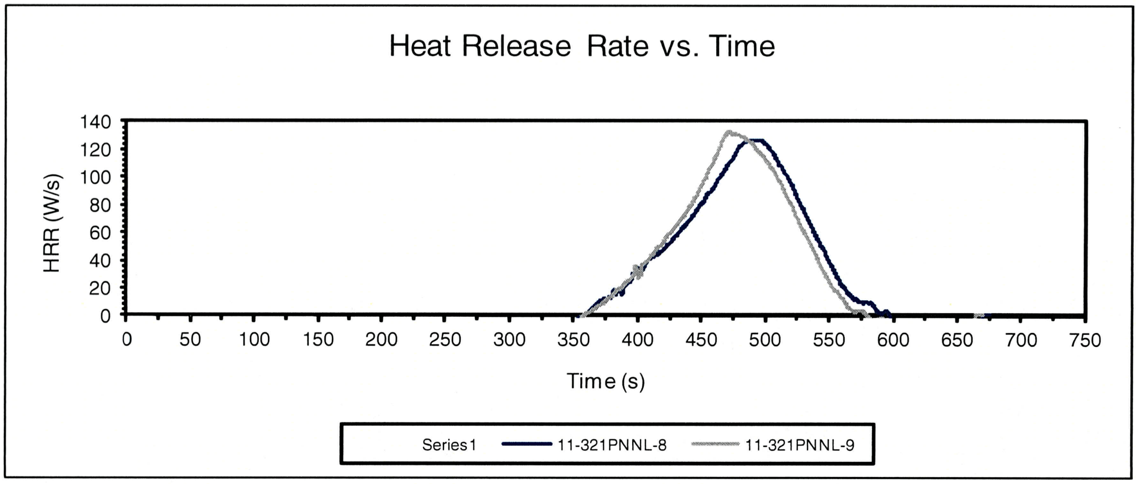


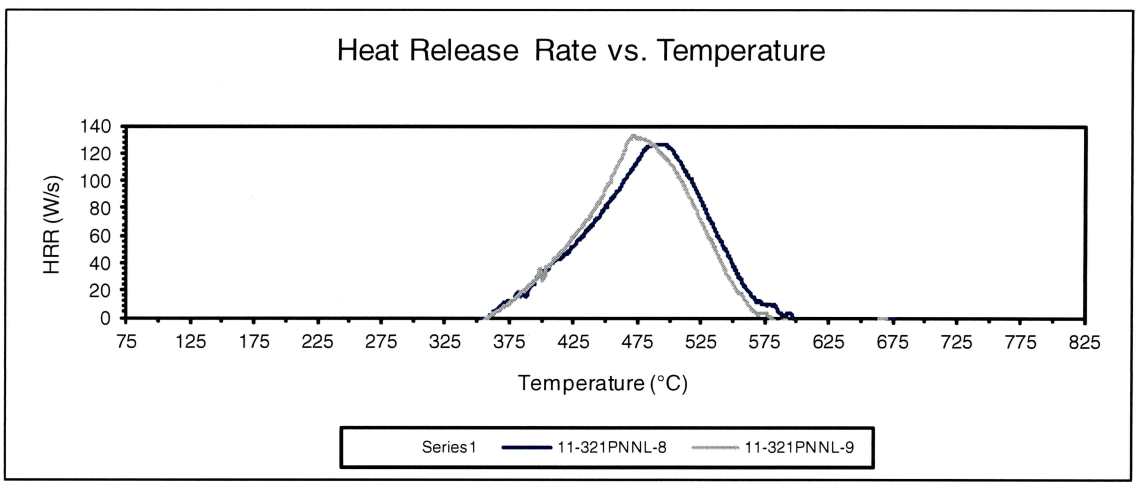


MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer	*Test Method:*	ASTM D7309/B
Preparation:	November 18, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Alan Lowry

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-321PNNL-8	November 17, 2011	2.46	0	126.902	847.799	126.902	13696.4295	0	13696.4
11-321PNNL-9	November 17, 2011	2.42	0	133.069	831.371	133.069	13392.585	0	13392.6
Average		2.44	0	129.986	839.585	129.9855	13544.5073	0	13544.5

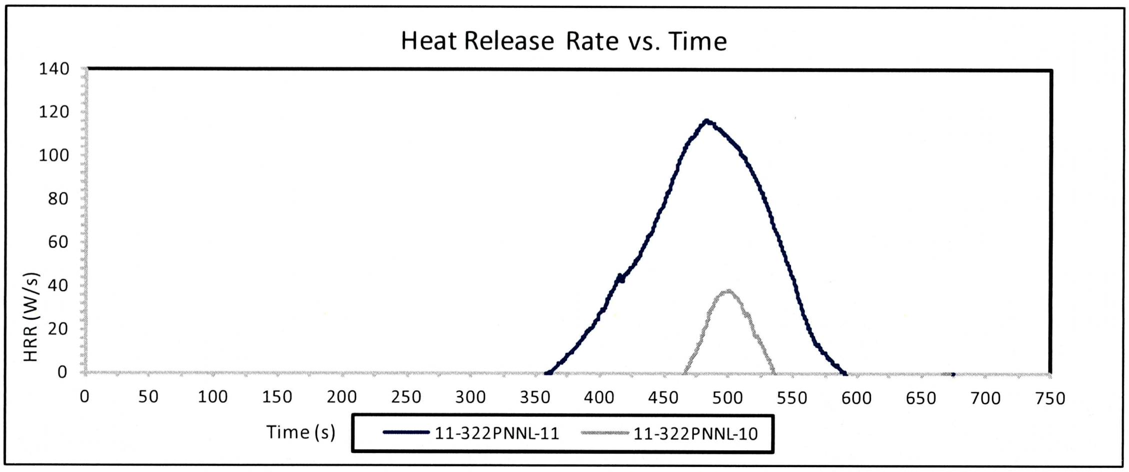


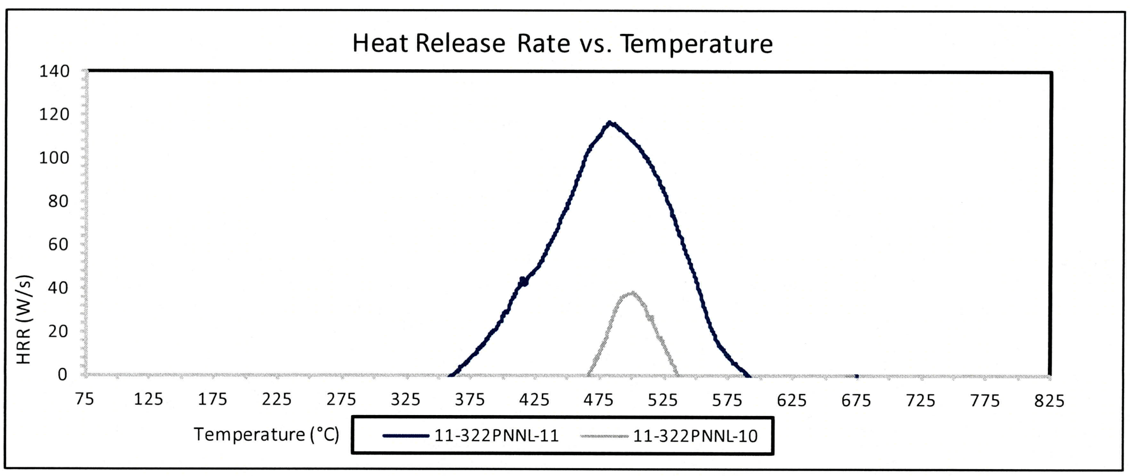


MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute	*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer	*Test Method:*	ASTM D7309/B
Preparation:	November 18, 2011	*Heating Rate:*	1K/S
		Temperature Range:	75 °C-750 °C
		Operator:	Alan Lowry

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-322PNNL-11	11/18/18	2.63	0	116.627	836.752	116.627	12980.497	0	12980.5
11-322PNNL-10	**11/18/18**	**2.5**	**0**	**37.975**	**856.401**	**37.975**	**1509.7223**	**0**	**1509.722**
Average		2.565	0	77.301	846.5765	77.301	7245.1095	0	7245.11



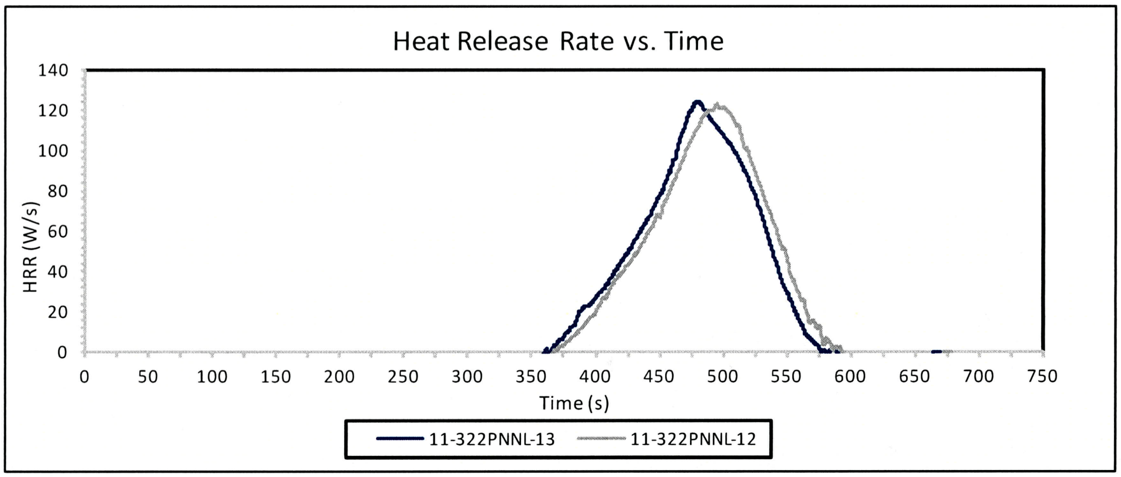


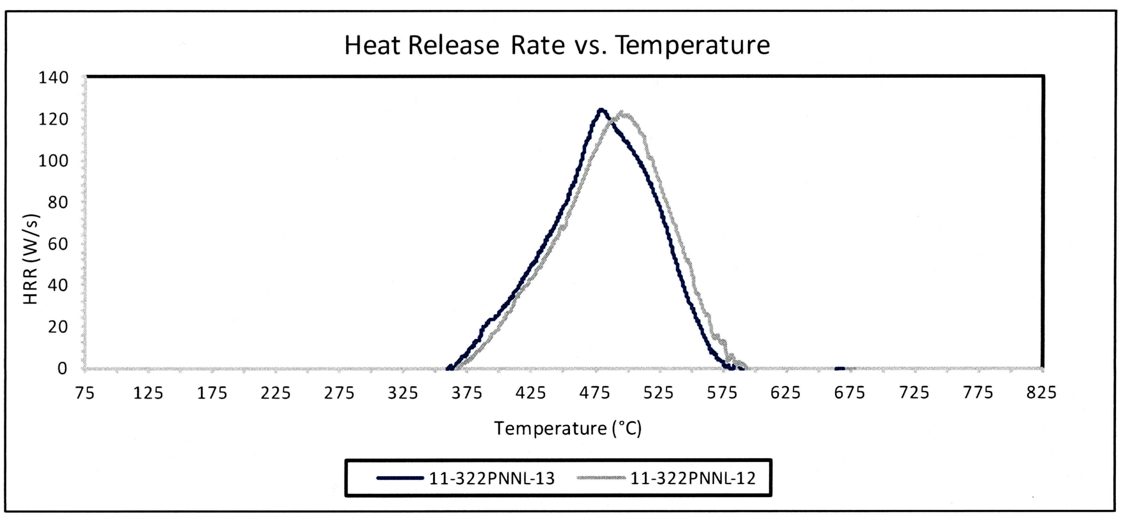
NOTE : 11-322PNNL-10 retested due to malfunction
QA Required Duplicate Run

MICROCALORIMETER TEST REPORT

Client:	Battelle Memorial Institute		*SwRI Project No:*	01.13295.12.008d
Material ID:	Resorcinol-Formaldehyde Polymer		*Test Method:*	ASTM D7309/B
Preparation:	November 18, 2011		*Heating Rate:*	1K/S
			Temperature Range:	75 °C-750 °C
			Operator:	Nathaniel Ramos

Test ID	Test Date	Initial Mass (g)	Final Mass (g)	Qmax (W/g)	Tmax (K)	η (J/g-K)	hc (J/g)	Yp (g/g)	hc, gas (kW/m²)
11-322PNNL-13	November 18, 2011	2.5	0	124.447	838.97	124.447	12437.639	0	12437.64
11-322PNNL-12	November 18, 2011	2.51	0	123.355	845.69	123.355	12689.77925	0	12689.78
Average		2.505	0	123.901	842.33	123.901	12563.70913	0	12563.71





QA Required Duplicate Run

SOUTHWEST RESEARCH INSTITUTE

CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008e
(ASTM E 662 and ASTM E 800 Testing)

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LASER GENERATED PARTICLE SIZE AND COUNT EVALUATION IN GENERAL ACCORDANCE WITH ASTM E 662-09, *STANDARD TEST METHOD FOR SPECIFIC OPTICAL DENSITY OF SMOKE GENERATED BY SOLID MATERIALS*, AND SUPPLEMENTED WITH TOXICITY EVALUATION TESTED IN ACCORDANCE WITH ASTM E 800-07, *STANDARD GUIDE FOR MEASUREMENT OF GASES PRESENT OR GENERATED DURING FIRES*

MATERIAL ID: *RESORCINOL-FORMALDEHYDE POLYMER*

FINAL REPORT
Consisting of 7 Pages

SwRI® Project No. 01.13295.12.008e
Test Dates: November 17–29, 2011
Report Date: December 16, 2011

Prepared for:

Battelle Memorial Institute
902 Battelle Blvd, K6-79
P.O. Box 999
Richland, WA 99352

Submitted by:

Eugene F. Horton
Senior Engineering Technologist
Material Flammability Section

Approved by:

For: Matthew S. Blais, Ph.D.
Director
Fire Technology Department

1.0 INTRODUCTION

This report presents the results of a particle count and size evaluation conducted at Southwest Research Institute's (SwRI) Fire Technology Department, located in San Antonio, Texas.

Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative, and a duplicate test were run, in addition to the one standard test.

The positive tests were performed in the flaming mode on a Schneller Standard Core Panel specimen measuring 75 × 75 × 10mm. The negative test was performed on calcium silicate specimen measuring 75 × 75 × 10 mm, which is a non-combustible material. This material is often used as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture, and; therefore, for particle size and count, it was used as a negative material test.

A radiant heat furnace was used to provide a constant irradiance of 25 kW/m^2 in a flaming exposures. The furnace used was not the same as prescribed in the ASTM E 662, *Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials,* standard. Because the material could not sit in the sample holder in a vertical orientation, the ISO 5659 furnace, a radiator cone, was used. The ISO 5659 is a similar smoke generation test which is typically run in the horizontal orientation. A pilot flame was used during flaming exposure and was centered in front of and parallel to the specimen holder. The pilot flame was also different from that described in the ASTM E 662, which is a six-flamelet pilot burner. Instead the ISO 5659, single pilot flame burner was used.

The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material, and then drying it in an oven. The sample was predried for a minimum of 10 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. After drying, the sample consisted of only the microbead material; the beads became statically charged in the absence of the water medium and therefore, they were placed in a closed container after drying, in an effort not to lose any beads. This pre-conditioning process is a deviation from the standard which requires samples to be predried for 24 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. Additionally specimens should placed in a conditioned environment maintained at 23 °C ± 3 °C (73 °F ± 5 °F) and a relative humidity of 50% ± 5% from the time they are received until drying, sample preparation, and testing.

Test specimens measuring 3 × 3 in., +0, -0.03 in. (76.2 × 76.2 mm, +0, -0.8 mm) were covered across the back, and along the edges with a single sheet of aluminum foil with the dull side in contact with the specimen.

ASTM E 662 requires the test to be conducted for a period of 3 min after a minimum light transmittance value is reached, or for an exposure of 20 min, whichever occurs first. SwRI conducted the tests for 20 minutes since the main directive for this test was particle size and distribution.

2.0 ACCEPTANCE CRITERIA

No acceptance criteria for the particle size and distribution are published in ASTM E 662 800, nor were any provided by the Client.

3.0 MATERIAL TESTED

Battelle Memorial Institute provided one 10-L jug of a material identified as *Resorcinol-Formaldehyde Polymer,* which was received by SwRI on November 11, 2011. Table 1 contains a description of the material on the safety data sheet provided by the Client. A description of the materials tested as observed by SwRI personnel is provided in Table 2.

Table 1. *Resorcinol-Formaldehyde Polymer* Sample Description.

Substance	***Resorcinol-Formaldehyde Polymer***
Trade Names/Synonyms	1,3-benzenediol-formaldehyde resin; formaldehyde-resorcinal polymer; resorcin-formaldehyde polymer
Chemical Family	Polymers
Physical State	Solid Particles Dispersed in Water
Color	Orange to Dark Red

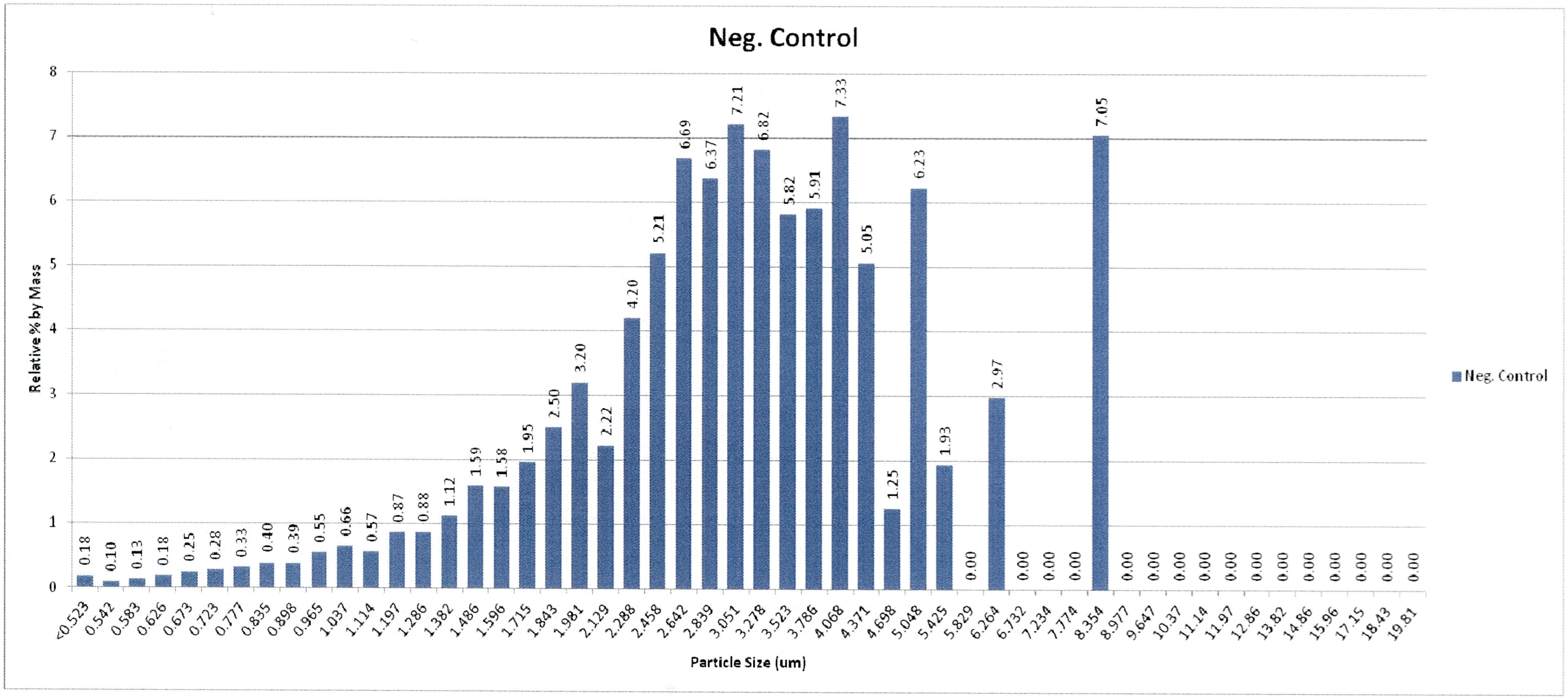
4.0 TEST RESULTS

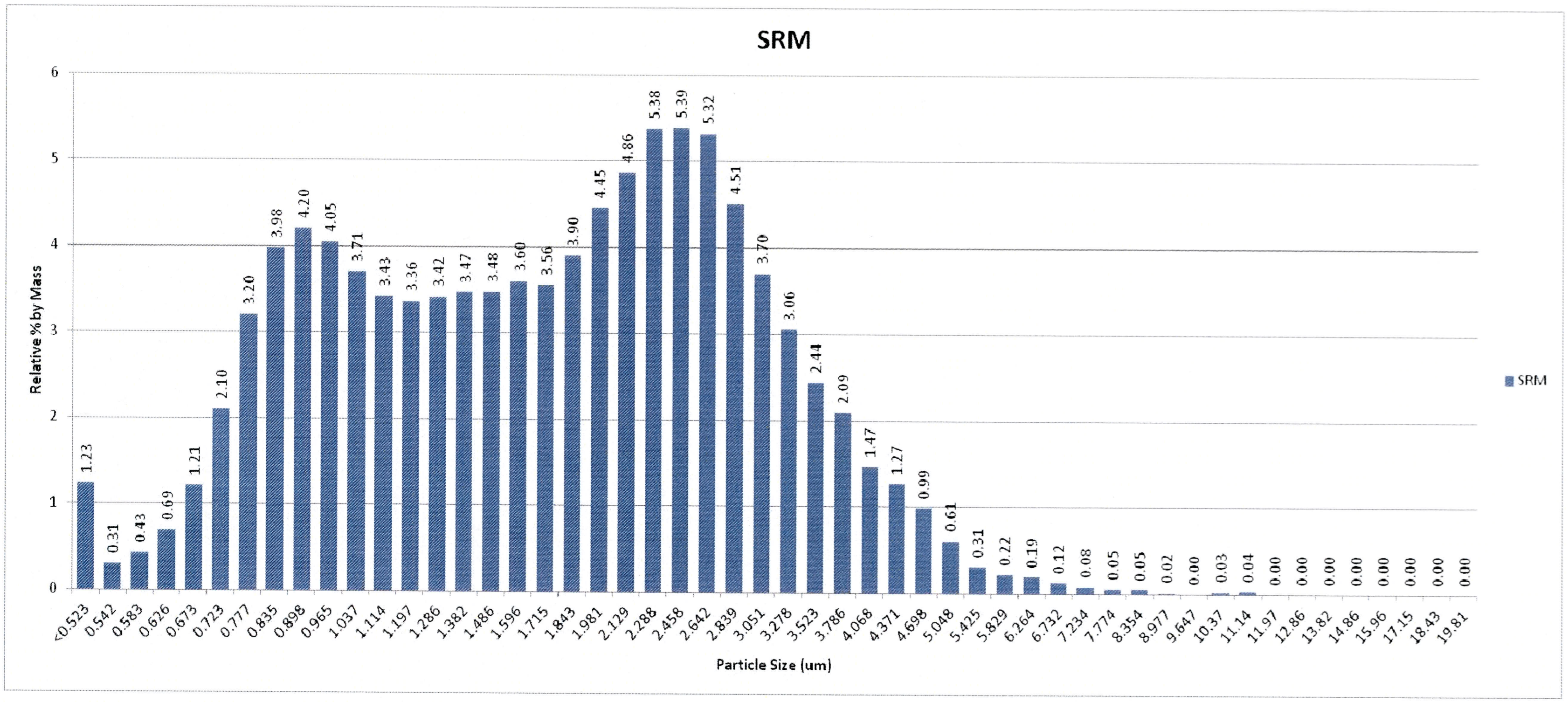
The tests were conducted on November 17–29, 2011, in general accordance with ASTM E 662. Test runs were carried out with a chamber wall temperature of 35 °C ± 2 °C (95 °F ± 4 °F). Test results for particle count and size are summarized in graphic form in Appendix A.

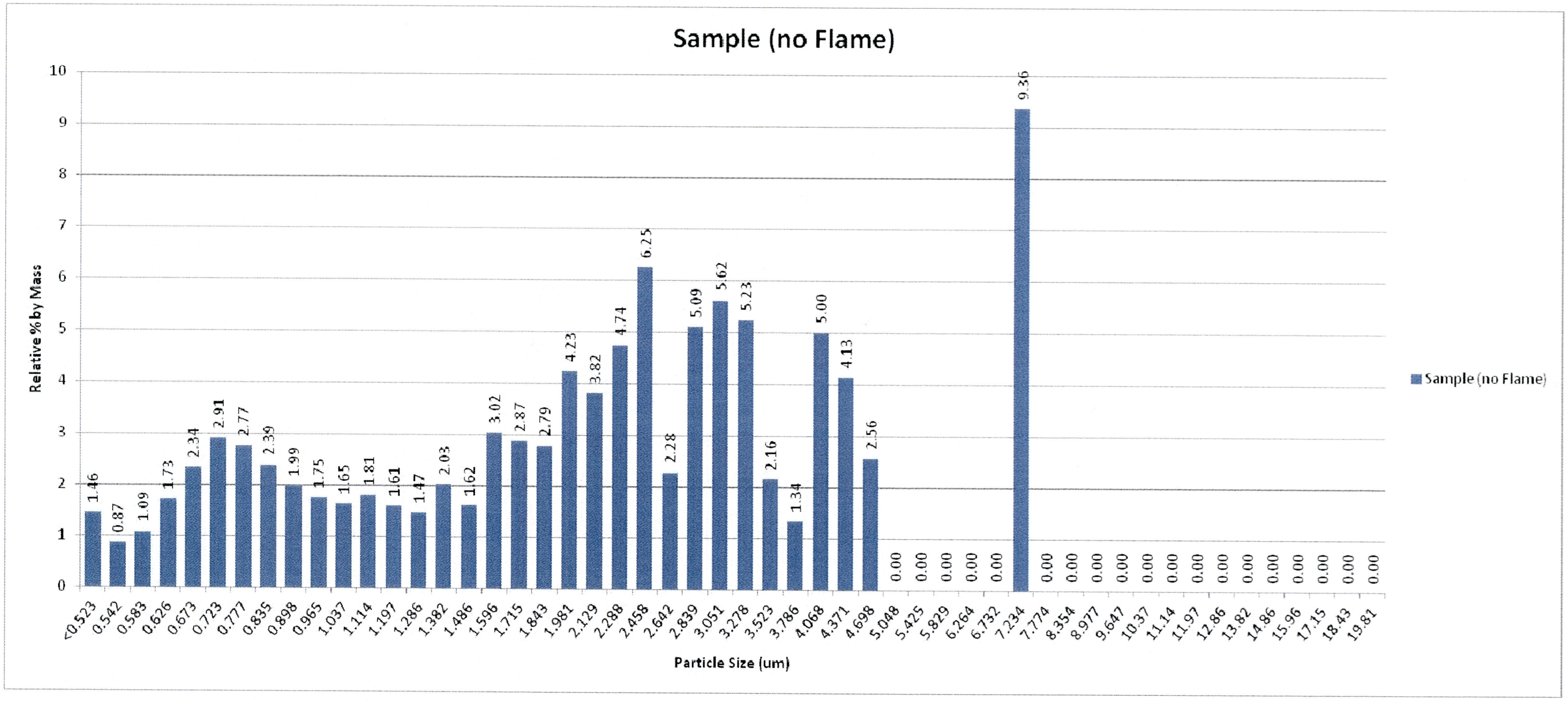
APPENDIX A

TEST RESULTS

(CONSISTING OF 3 PAGES)


Neg. Control
Relative % by Mass
Particle Size (um)
Neg. Control
0.18 0.10 0.13 0.18 0.25 0.28 0.33 0.40 0.39 0.55 0.66 0.57 0.87 0.88 1.12 1.59 1.58 1.95 2.50 3.20 2.22 4.20 5.21 6.69 6.37 7.21 6.82 5.82 5.91 7.33 5.05 1.25 6.23 1.93 0.00 2.97 0.00 0.00 0.00 7.05 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
<0.523 0.542 0.583 0.626 0.673 0.723 0.777 0.835 0.898 0.965 1.037 1.114 1.197 1.286 1.382 1.486 1.596 1.715 1.843 1.981 2.129 2.288 2.458 2.642 2.839 3.051 3.278 3.523 3.786 4.068 4.371 4.698 5.048 5.425 5.829 6.264 6.732 7.234 7.774 8.354 8.977 9.647 10.37 11.14 11.97 12.86 13.82 14.86 15.96 17.15 18.43 19.81
0 1 2 3 4 5 6 7 8


SRM
Relative % by Mass
Particle Size (um)
SRM



Sample (no Flame)
Relative % by Mass
Particle Size (um)
Sample (no Flame)
10
9
8
7
6
5
4
3
2
1
0
1.46
0.87
1.09
1.73
2.34
2.91
2.77
2.39
1.99
1.75
1.65
1.81
1.61
1.47
2.03
1.62
3.02
2.87
2.79
4.23
3.82
4.74
6.25
2.28
5.09
5.62
5.23
2.16
1.34
5.00
4.13
2.56
0.00
0.00
0.00
0.00
0.00
9.36
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
0.00
<0.523
0.542
0.583
0.626
0.673
0.723
0.777
0.835
0.898
0.965
1.037
1.114
1.197
1.286
1.382
1.486
1.596
1.715
1.843
1.981
2.129
2.288
2.458
2.642
2.839
3.051
3.278
3.523
3.786
4.068
4.371
4.698
5.048
5.425
5.829
6.264
6.732
7.234
7.774
8.354
8.977
9.647
10.37
11.14
11.97
12.86
13.82
14.86
15.96
17.15
18.43
19.81

SOUTHWEST RESEARCH INSTITUTE

CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008f
(ASTM E 662 and ASTM E 800 Testing)

SOUTHWEST RESEARCH INSTITUTE®

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FIRE TECHNOLOGY DEPARTMENT
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Registered to ISO 9001:2008

OPTICAL SMOKE DENSITY EVALUATION IN GENERAL ACCORDANCE WITH ASTM E 662-09, *STANDARD TEST METHOD FOR SPECIFIC OPTICAL DENSITY OF SMOKE GENERATED BY SOLID MATERIALS*, AND SUPPLEMENTED WITH TOXICITY EVALUATION TESTED IN ACCORDANCE WITH ASTM E 800-07, *STANDARD GUIDE FOR MEASUREMENT OF GASES PRESENT OR GENERATED DURING FIRES*

MATERIAL ID: ***RESORCINOL-FORMALDEHYDE POLYMER***

FINAL REPORT
Consisting of 15 Pages

SwRI® Project No. 01.13295.12.008f
Test Date: November 17-29, 2011
Report Date: December 15, 2011

Prepared for:

Battelle Memorial Institute
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P.O. Box 999
Richland, WA 99352

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Submitted by:

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Senior Engineering Technologist
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Matthew S. Blais, Ph.D.
Director
Fire Technology Department

ABSTRACT

Battelle Memorial Institute located in Richland, Washington, submitted a material identified as *Resorcinol-Formaldehyde Polymer* for testing in general accordance with ASTM E 662 (2009), *Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials,* supplemented with toxicity analysis using ASTM E 800 (2007), *Standard Guide for Measurement of Gases Present or Generated during Fires,* as a guide. Four sets of tests were performed, one on a negative/blank specimen, one on a positive/standard specimen, one on the provided material (Resorcinol-Formaldehyde Polymer) and a duplicate on the provided material. The maximum concentrations detected using Fourier transform infrared (FTIR) spectroscopy are reported below along with summarized smoke density results. Maximum smoke density values are presented as an average of the corrected optical density values.

RESORCINOL-FORMALDEHYDE POLYMER **PERFORMANCE SUMMARY.**

Specific Optical Density (D_S) Results			
25 kW/m^2	**Average D_S at 1.5 min**	**Average D_S at 4 min**	**Average Max Corrected D_S**
Blank - Flaming	0.38	0.43	0.50
Blank - Nonflaming	0.24	0.22	0.00
Standard - Flaming	105	90	92
Standard - Nonflaming	18	148	150
Material - Flaming	0.26	0.28	0.56
Material - Nonflaming	0.21	0.22	0.30
Material Duplicate - Nonflaming	0.36	0.44	0.41
Material Duplicate – Flaming	0.15	0.12	0.21

Maximum Concentrations (ppm)								
Test	**CO**	**CO_2**	**HBr**	**HCl**	**HCN**	**HF**	**NO_X**	**SO_2**
Blank - Nonflaming	—**	116 ± 21	—	—	—	—	—	—
Blank - Flaming	—	1113 ± 38	—	—	10 ± 2	—	—	—
Standard - Nonflaming	332 ± 2	820 ± 37	—	—	—	—	—	3 ± 1
Standard - Flaming	20 ± 1	501 ± 7	—	—	—	—	—	—
Material - Nonflaming	803 ± 6	834 ± 41	—	—	—	—	—	3 ± 1
Material - Flaming	1681 ± 18	1628 ± 63	—	—	—	—	—	7 ± 2
Duplicate - Nonflaming	88 ± 1	3421 ± 123	—	—	—	—	—	2.3 ± 1.5
Duplicate - Flaming	762 ± 55	1057 ± 43	—	—	—	—	—	7 ± 3

* ± Represents 90% confidence interval.
** Represents gas not detected.

1.0 INTRODUCTION

This report presents the results of a smoke density and toxicity evaluation conducted at Southwest Research Institute's (SwRI) Fire Technology Department, located in San Antonio, Texas. The smoke density evaluation was conducted in accordance with ASTM E 662 (2006), *Standard Test Method for Specific Optical Density of Smoke Generated by Solid Materials*. The toxicity evaluation was performed using Fourier transform infrared (FTIR) spectroscopy using ASTM E 800 (2007), *Standard Guide for Measurement of Gases Present or Generated during Fires*. The sample for FTIR analysis is drawn from smoke runs of the E 662 test. The deviations from the standard include the pre-conditioning procedure, the heater and orientation, and the additional tests.

Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative and a duplicate test were run, in addition to the one standard test.

The negative tests were performed on calcium silicate, which is a non-combustible material. This material is used as often as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture, and therefore for smoke and toxicity, it was used as a negative material test. The positive tests were performed in the nonflaming mode on an α-cellulose material from Fire Testing Technologies, and in the flaming mode on Schneller Standard Core Panel.

A radiant heat furnace was used to provide a constant irradiance of 25 kW/m^2 in two exposures: flaming and nonflaming. The furnace used was not the same as prescribed in the ASTM E 662 standard. Because the material could not sit in the sample holder in a vertical orientation, the ISO 5659 furnace, a radiator cone, was used. The ISO 5659 is a similar smoke generation test which is typically run in the horizontal orientation. A pilot flame was used during flaming exposure and was centered in front of and parallel to the specimen holder. The pilot flame was also different from that described in the ASTM E662, which is a six-flamelet pilot burner. Instead the ISO 5659, single pilot flame burner was used.

Smoke density measurement is made based on the attenuation of a vertically oriented light beam through smoke accumulating within the chamber. Results are expressed in terms of the dimensionless specific optical density, D_s. A clear beam value is recorded upon evacuation of smoke from the box to determine a correction factor. The maximum corrected smoke density value from each of the runs at a single exposure condition are averaged and reported. Per the standard, smoke density values are rounded to two significant figures.

The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material, and then drying it in an oven. The sample was predried for a minimum of 10 h at 60

60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. After drying, the sample consisted of only the microbead material; the beads became statically charged in the absence of the water medium and therefore, they were placed in a closed container after drying, in an effort not to lose any beads. This pre-conditioning process is a deviation from the standard which requires samples to be predried for 24 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. Additionally specimens should placed in a conditioned environment maintained at 23 °C ± 3 °C (73 °F ± 5 °F) and a relative humidity of 50% ± 5% from the time they are received until drying, sample preparation, and testing.

Test specimens measuring 3 × 3 in., +0, -0.03 in. (76.2 × 76.2 mm, +0, -0.8 mm) were covered across the back, and along the edges with a single sheet of aluminum foil with the dull side in contact with the specimen.

ASTM E 662 requires the test to be conducted for a period of 3 min after a minimum light transmittance value is reached, or for an exposure of 20 min, whichever occurs first. SwRI conducts tests for 10 min unless a minimum light transmittance value has not yet occurred, then the test is continued for an additional 10 min. Notes are made relevant to any burning characteristics such as delamination, intumescence, shrinkage, and ignition.

Gas analysis is performed during the third run in each exposure mode and analyzed using an FTIR spectrometer. The sample is drawn through a preheated line to prevent condensation and adsorption, and filtered into a preheated gas cell. Analysis is conducted for carbon monoxide (CO), carbon dioxide (CO_2), hydrogen chloride (HCl), hydrogen cyanide (HCN), hydrogen fluoride (HF), hydrogen bromide (HBr), sulfur dioxide (SO_2), and oxides of nitrogen (NO_X).

This test method is intended to measure and describe the properties of products in response to heat and flame under controlled laboratory conditions. The results should not be used alone to describe or appraise the fire hazard or the fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a complete fire hazard assessment or a fire risk assessment, which takes into account all the factors that are pertinent to an assessment of the fire hazard or fire risk of a particular end use. The results apply specifically to the specimens tested, in the manner tested, and not to similar materials, nor to the performance when used in combination with other materials.

2.0 ACCEPTANCE CRITERIA

No acceptance criteria for smoke density or toxicity are published in ASTM E 662 or ASTM E 800, nor were any provided by the Client.

3.0 MATERIAL TESTED

Battelle Memorial Institute, located in Richland, Washington, provided one 10-L jug of a material identified as *Resorcinol-Formaldehyde Polymer,* which was received by SwRI on November 11, 2011. Table 1 contains a description of the material on the safety data sheet provided by the Client. A description of the materials tested as observed by SwRI personnel is provided in Table 2.

Table 1. *RESORCINOL-FORMALDEHYDE POLYMER* Sample Description.

Substance	Rescorcinol-formaldehyde polymer
Trade Names/Synonyms	1,3-benzenediol-formaldehyde resin; formaldehyde-resorcinal polymer; resorcin-formaldehyde polymer
Chemical Family	Polymers
Physical State	Solid particles dispersed in water
Color	Orange to dark red

Table 2. Test Sample Descriptions.

Material ID	Nominal Tested Thickness*	Nominal Tested Mass*	Color*
Resorcinol-Formaldehyde Polymer (beads)	25 mm	82 g	Rust Red
Calcium silicate (blank)	25 mm	92 g	White
α-cellulose material (Standard nonflaming)	1.5 mm	3 g	White
Schneller Core Panel (Standard flaming)	3.8 mm	8 g	Amber

* Measured by SwRI personnel.

4.0 TEST RESULTS

The tests were conducted on November 17-29, 2011, in accordance with ASTM E 662 and ASTM E 800. Test runs were carried out with a chamber wall temperature of 35 °C ± 2 °C (95 °F ± 4 °F). Test results for smoke density are summarized in Tables 2 – 7 with toxicity results summarized in Table 8. Figures 1 – 8, the standard ASTM E 662 test data sheets, provide more detailed information including mass loss, time to maximum smoke density, and visual observations for each test run.

Table 2. Flaming Exposure Specific Optical Density Results for Blank and Standard.

Material	Test ID	D_S at 1.5 min	D_S at4.0 min	Maximum D_S	Maximum D_S (corrected)	Time to Max D_S (s)
Blank	11-321P3	0.38	0.43	0.50	0.50	239
Standard	11-332P1	110	90	110	90	86

Table 3. Nonflaming Exposure Specific Optical Density Results for Blank and Standard.

Material	Test ID	D_S at 1.5 min	D_S at4.0 min	Maximum D_S	Maximum D_S (corrected)	Time to Max D_S (s)
Blank	11-321P2	0.24	0.22	0.47	0.00	1082
Standard	11-321P1	18	150	180	180	150

Table 4. Flaming Exposure Specific Optical Density Results for Resorcinol-Formaldehyde Polymer.

Test ID	D_S at 1.5 min	D_S at4.0 min	Maximum D_S	Maximum D_S (corrected)	Time to Maximum D_S (s)
11-332P2	0.02	0.00	0.61	0.61	968
11-332P3	0.33	0.39	0.47	0.47	278
11-332P4	0.42	0.44	0.70	0.59	686
Average:	0.26	0.28	0.60	0.56	644
Range:	0.02 – 0.42	0.00 – 0.44	0.47 – 0.70	0.47 – 0.61	

Table 5. Nonflaming Exposure Specific Optical Density Results for Resorcinol-Formaldehyde Polymer.

Test ID	D_S at 1.5 min	D_S at 4.0 min	Maximum D_S	Maximum D_S (corrected)	Time to Maximum D_S (s)
11-322P1	0.20	0.28	0.40	0.40	475
11-322P2	0.23	0.18	0.36	0.36	416
11-322P3	0.19	0.22	0.57	0.13	1165
Average:	0.21	0.22	0.44	0.30	685
Range:	0.19 – 0.23	0.18 – 0.28	0.36 – 0.57	0.13 – 0.40	

Table 6. Duplicate Flaming Exposure Specific Optical Density Results for Resorcinol-Formaldehyde Polymer.

Test ID	D_S at 1.5 min	D_S at4.0 min	Maximum D_S	Maximum D_S (corrected)	Time to Maximum D_S (s)
11-333P1	0.05	0.04	0.15	0.15	747
11-333P2	0.61	0.74	1.14	0.45	836
11-333P3	0.41	0.54	0.83	0.62	1198
Average:	0.36	0.44	0.70	0.41	927
Range:	0.05 – 0.61	0.04 – 0.74	0.15 – 1.14	0.15 – 0.62	

Table 7. Duplicate Nonflaming Exposure Specific Optical Density Results for Resorcinol-Formaldehyde Polymer.

Test ID	D_S at 1.5 min	D_S at 4.0 min	Maximum D_S	Maximum D_S (corrected)	Time to Maximum D_S (s)
11-322P4	0.08	0.03	0.24	0.24	587
11-322P5	0.18	0.10	0.28	0.28	37
11-323P1	0.18	0.23	0.27	0.11	239
Average:	0.15	0.12	0.26	0.21	288
Range:	0.08 – 0.18	0.03 – 0.23	0.24 – 0.28	0.11 – 0.28	

Table 8. Maximum Concentrations Detected in Parts Per Million (ppm).

Material	Exposure Mode	CO	CO_2	HBr	HCl	HCN	HF	NO_X	SO_2
Blank	Nonflaming	—**	116 ± 21*	—	—	—	—	—	—
	Flaming	—	1113 ± 38	—	—	10 ± 2	—	—	—
Standard	Nonflaming	332 ± 2	820 ± 37	—	—	—	—	—	3 ± 1
	Flaming	20 ± 1	501 ± 7	—	—	—	—	—	—
Resorcinol-Formaldehyde Polymer	Nonflaming	803 ± 6	834 ± 41	—	—	—	—	—	3 ± 1
	Flaming	1681 ± 18	1628 ± 63	—	—	—	—	—	7 ± 2
Resorcinol-Formaldehyde Polymer	Nonflaming	88 ± 1	3421 ± 123	—	—	—	—	—	2.3 ± 1.5
	Flaming	762 ± 55	1057 ± 43	—	—	—	—	—	7 ± 3

* ± Represents the 90% confidence level in the concentration measurement.

**Represents gas not detected

SOUTHWEST RESEARCH INSTITUTE

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	Piloted (Flaming)
Test Date(s):	November 17, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Blank**	*Color:*	White
		Thickness:	25 mm
Description:*	Non-combustible calcium silicate board	*Receipt Date:*	Not Applicable
		Date Prepared by SwRI:	Cut to size on test date
		Oven Cure:	None
		Note:	

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-321P3	95.76	93.96	1.80	0.38	0.43	0.50	239	0.00	0.50
Average	95.76	93.96	1.80	0.38	0.43	0.50	239	0.00	**0.50**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-321P3	-	-	-	-	-	-	-	-
Average	-	-	-	-	-	-	-	-

OPTICAL DENSITY CHART

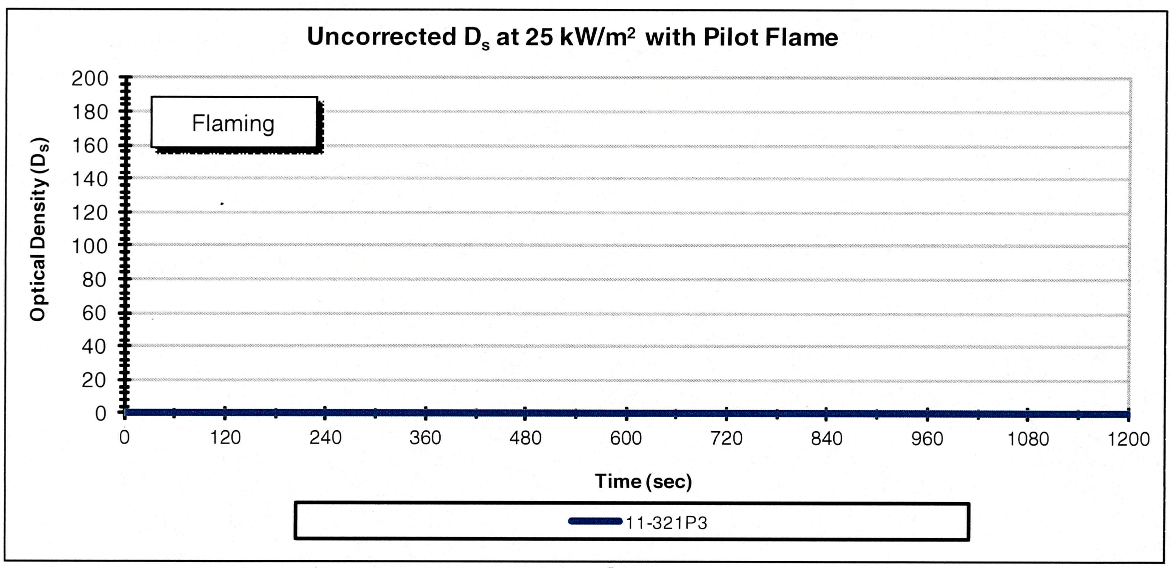

Figure 1. Smoke Density Results during Flaming Exposure - Blank.

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	No pilot (nonflaming)
Test Date(s):	November 18 and 19, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Blank**	*Color:*	White
		Thickness:	25 mm
Description:*	Non-combustible calcium silicate board	*Receipt Date:*	Not Applicable
		Date Prepared by SwRI:	Cut to size on test date
		Oven Cure Date:	None
		Note:	

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-321P2	90.62	88.62	2.00	0.24	0.22	0.47	1082	0.47	0.00
Average	90.62	88.62	2.00	0.24	0.22	0.47	1082	0.47	**0.00**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-321P2	-	-	-	-	-	-	-	-
Average	-	-	-	-	-	-	-	-

OPTICAL DENSITY CHART

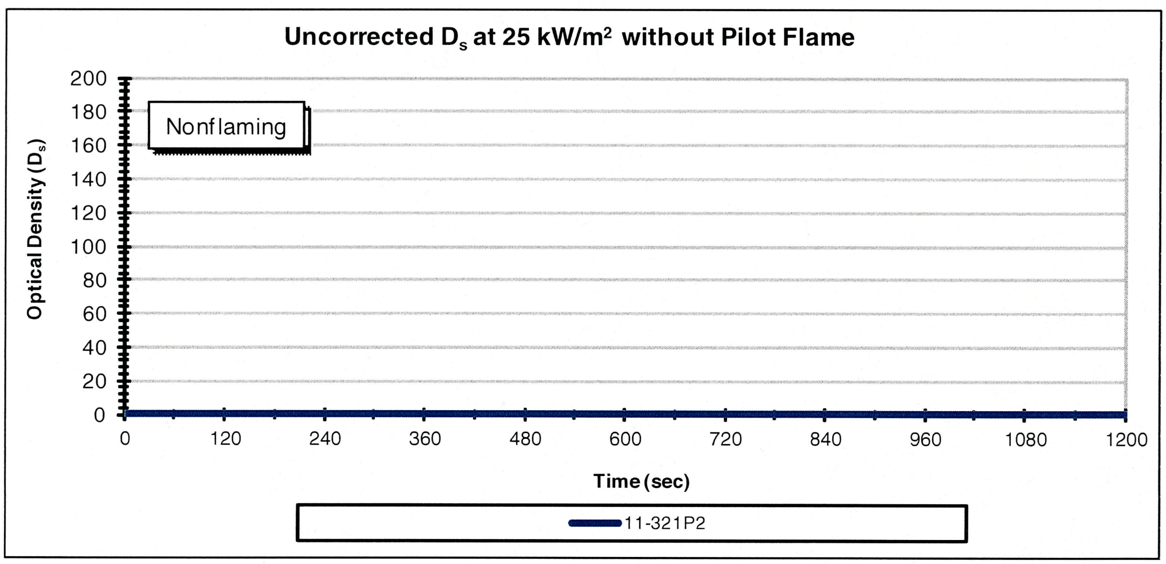

Figure 2. Smoke Density Results during Nonflaming Exposure - Blank.

SOUTHWEST RESEARCH INSTITUTE

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	Piloted (Flaming)
Test Date(s):	November 28, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Standard Reference Material**	*Color:*	Amber
		Thickness:	3.82 mm
Description:*	Reference material from Schneller - Schneller Standard Core Panel with Ha211 applied	*Receipt Date:*	November 18, 2011
		Date Prepared by SwRI:	Cut to size on test date
		Oven Cure:	None
		Note:	

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-332P1	7.84	4.58	3.26	105.03	90.02	107.65	86	15.67	91.98
Average	7.84	4.58	3.26	105.03	90.02	107.65	86	15.67	**91.98**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-332P1	2	1	-	1	-	58	-	-
Average	2	1	-	1	-	58	-	-

OPTICAL DENSITY CHART

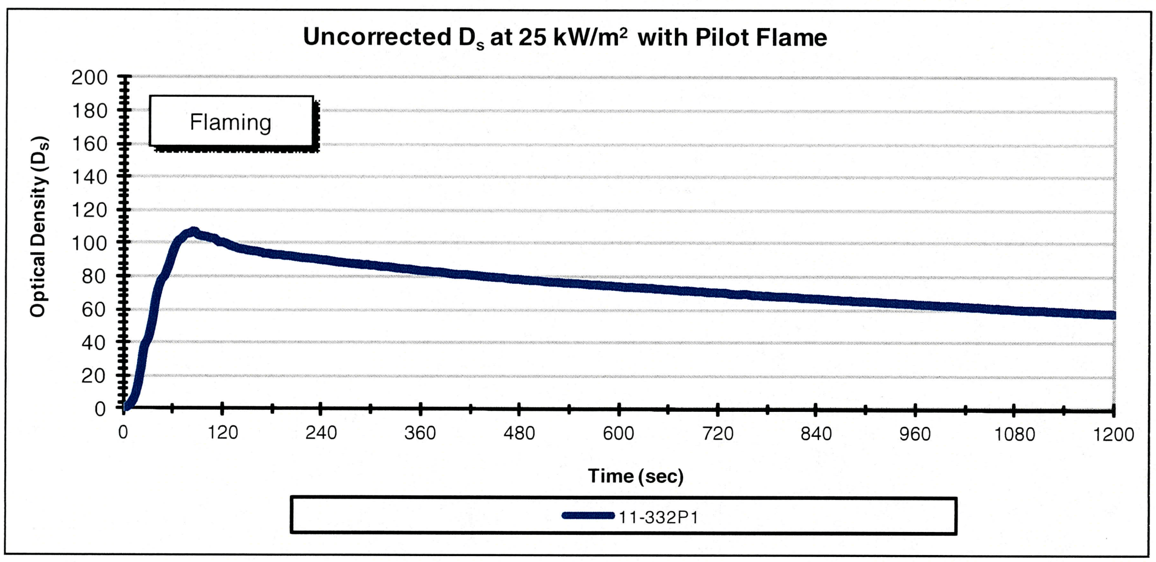

Figure 3. Smoke Density Results during Flaming Exposure - Standard.

S O U T H W E S T   R E S E A R C H   I N S T I T U T E

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	No pilot (nonflaming)
Test Date(s):	November 17, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Standard Reference Material**	*Color:*	White
		Thickness:	1.52 mm
Description:*	Reference Material from Fire Testing Technologies - FTT reference material non-flaming (1006d equivalent) (K159 Batch 2010)	*Receipt Date:*	November 16, 2011
		Date Prepared by SwRI:	Received ready-to-test
		Oven Cure Date:	None
		Note:	

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-321P1	3.21	0.01	3.20	17.99	148.07	178.69	485	28.55	150.14
Average	3.21	0.01	3.20	17.99	148.07	178.69	485	28.55	**150.14**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-321P1	47	52	-	-	-	-	-	-
Average	47	52	-	-	-	-	-	-

OPTICAL DENSITY CHART

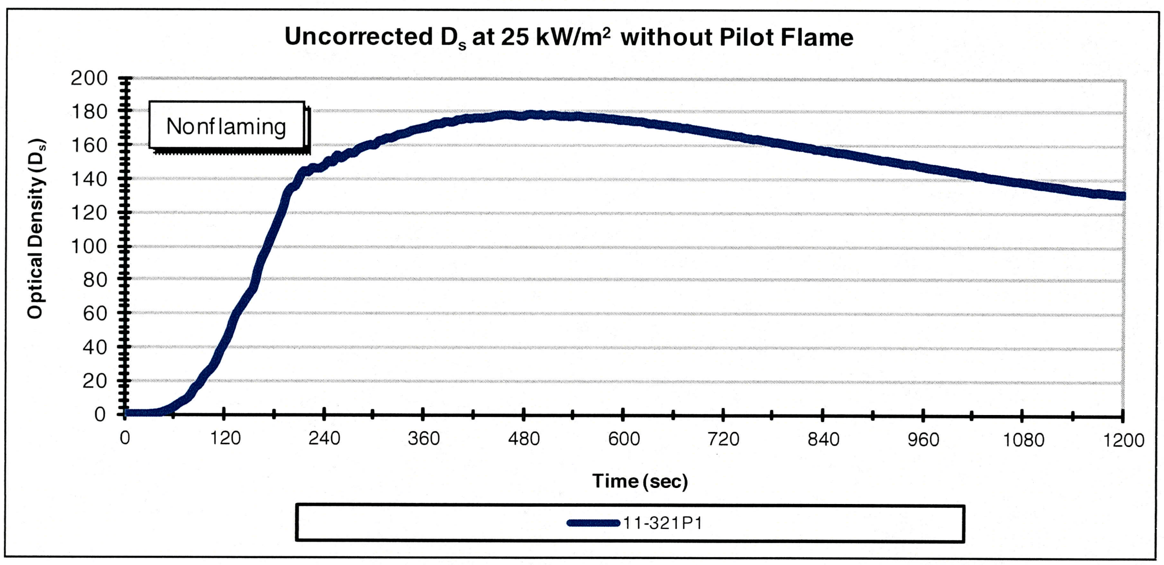

Figure 4. Smoke Density Results during Nonflaming Exposure - Standard.

SOUTHWEST RESEARCH INSTITUTE

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	Piloted (Flaming)
Test Date(s):	November 28, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Resorcinol-Formaldehyde Polymer**	*Color:*	Orange to dark red
		Thickness:	25 mm
Description:*	Resorcinol-formaldehyde polymer	*Receipt Date:*	November 11, 2011
		Date Prepared by SwRI:	Tested as received
		Oven Cure:	Minimum of 24 h
		Note:	*Tested solid component without water*

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-332P2	80.00	62.83	17.17	0.02	0.00	0.61	968	0.00	0.61
11-332P3	80.00	61.66	18.34	0.33	0.39	0.47	278	0.00	0.47
11-332P4	80.00	62.49	17.51	0.42	0.44	0.70	686	0.12	0.59
Average	80.00	62.33	17.67	0.26	0.28	0.60	644	0.04	**0.56**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-332P2	-	42	-	678	-	-	-	-
11-332P3	-	38	-	-	-	-	-	-
11-332P4	-	44	-	641	-	-	-	-
Average	-	41	-	660	-	-	-	-

OPTICAL DENSITY CHART

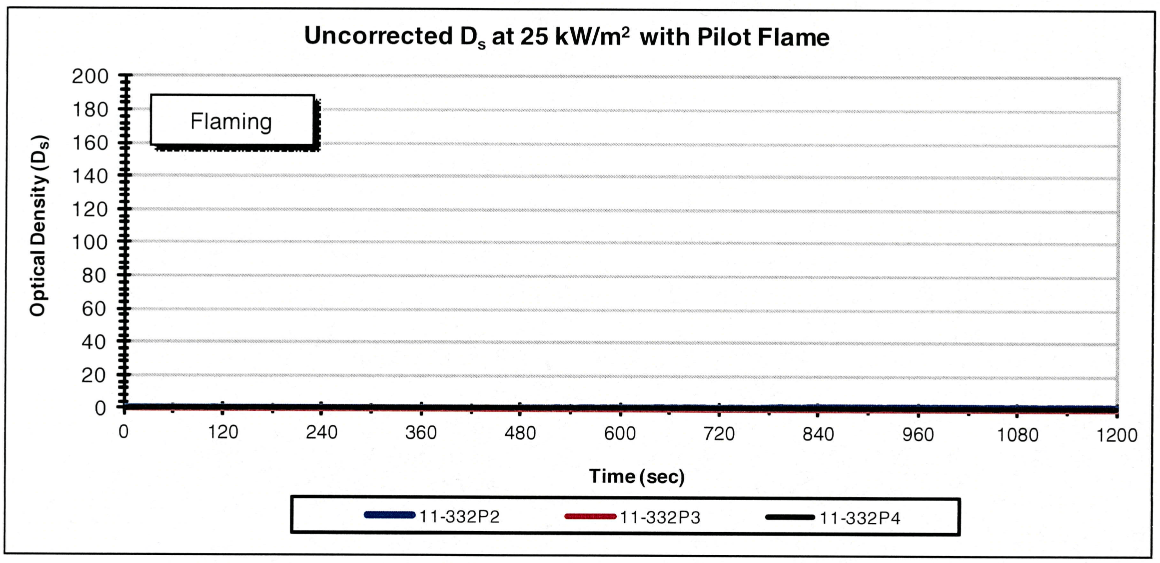

Figure 5. Smoke Density Results during Flaming Exposure - Resorcinol-Formaldehyde Polymer.

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	No pilot (nonflaming)
Test Date(s):	November 18, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Resorcinol-Formaldehyde Polymer**	*Color:*	Orange to dark red
		Thickness:	25 mm
Description:*	Resorcinol-formaldehyde polymer	*Receipt Date:*	November 11, 2011
		Date Prepared by SwRI:	Tested as received
		Oven Cure Date:	Minimum of 24 h
		Note:	*Tested solid component without water*

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-322P1	83.50	65.40	18.10	0.20	0.28	0.40	475	0.00	0.40
11-322P2	83.50	63.52	19.98	0.23	0.18	0.36	416	0.00	0.36
11-322P3	83.50	64.96	18.54	0.19	0.22	0.57	1165	0.44	0.13
Average	83.50	64.63	18.87	0.21	0.22	0.44	685	0.15	**0.30**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-322P1	-	43	-	-	-	-	-	-
11-322P2	-	41	-	-	-	-	-	-
11-322P3	-	39	-	-	-	-	-	-
Average	-	41	-	-	-	-	-	-

OPTICAL DENSITY CHART

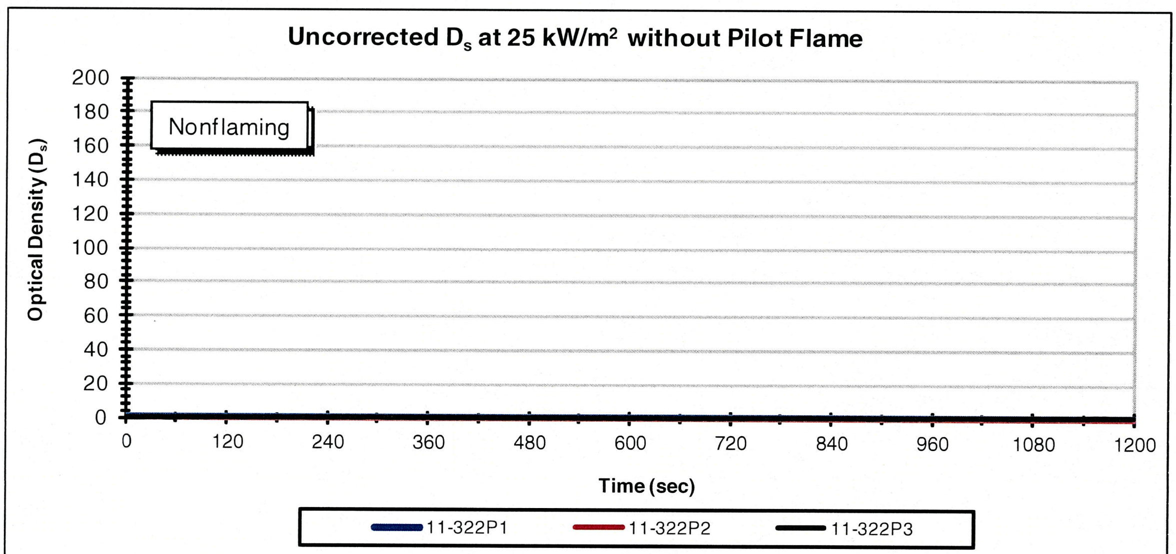

Figure 6. Smoke Density Results during Nonflaming Exposure - Resorcinol-Formaldehyde Polymer.

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	Piloted (Flaming)
Test Date(s):	November 29, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Resorcinol-Formaldehyde Polymer Duplicate Tests**	*Color:*	Orange to dark red
		Thickness:	25 mm
Description:*	Resorcinol-formaldehyde polymer	*Receipt Date:*	November 11, 2011
		Date Prepared by SwRI:	Tested as received
		Oven Cure:	Minimum of 24 h
		Note:	*Tested solid component without water*

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-333P1	80.00	62.45	17.55	0.05	0.04	0.15	747	0.00	0.15
11-333P2	80.00	63.01	16.99	0.61	0.74	1.14	836	0.69	0.45
11-333P3	80.00	62.33	17.67	0.41	0.54	0.83	1198	0.20	0.62
Average	80.00	62.60	17.40	0.36	0.44	0.70	927	0.30	**0.41**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-333P1	-	40	-	606	-	-	-	-
11-333P2	-	36	-	629	-	-	-	-
11-333P3	-	40	-	605	-	-	-	-
Average	-	39	-	613	-	-	-	-

OPTICAL DENSITY CHART

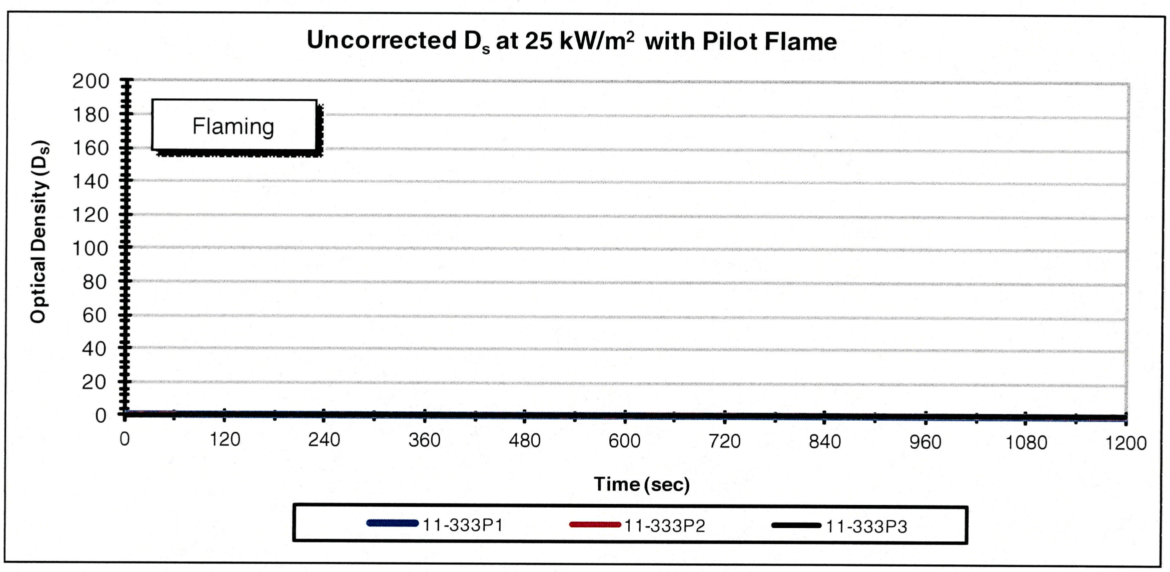

Figure 7. Smoke Density Results during Flaming Exposure - Resorcinol-Formaldehyde Polymer.

SOUTHWEST RESEARCH INSTITUTE

ASTM E 662 SMOKE DENSITY TEST REPORT

Client:	Battelle Memorial Institute	*Heat Flux:*	25 kW/m^2
Operator:	P. Lopez	*Exposure Mode:*	No pilot (nonflaming)
Test Date(s):	November 18 and 19, 2011	*Orientation:*	Horizontal
Project No.	01.13295.12.008f		
Material ID:*	**Resorcinol-Formaldehyde Polymer**	*Color:*	Orange to dark red
	Duplicate Tests	*Thickness:*	25 mm
Description:*	Resorcinol-formaldehyde polymer	*Receipt Date:*	November 11, 2011
		Date Prepared by SwRI:	Tested as received
		Oven Cure Date:	Minimum of 24 h
		Note:	*Tested solid component without water*

** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	1.5 min D_s	4.0 min D_s	Maximum D_s	Time to Max. D_s (sec)	Correction Factor	Corrected Max. D_s
11-322P4	83.50	67.00	16.50	0.08	0.03	0.24	587	0.00	0.24
11-322P5	83.50	67.23	16.27	0.18	0.10	0.28	37	0.00	0.28
11-323P1	83.50	66.52	16.98	0.18	0.23	0.27	239	0.17	0.11
Average	83.50	66.92	16.58	0.15	0.12	0.26	288	0.06	**0.21**

OBSERVATIONS

Test ID	Observed Smoke (sec)	Surface Char (sec)	Observed Blistering (sec)	Time to Ignition (sec)	Flames to Top (sec)	Initial Flame Out (sec)	Second Ignition (sec)	Final Flame Out (sec)
11-322P4	-	44	-	-	-	-	-	-
11-322P5	-	41	-	-	-	-	-	-
11-323P1	-	52	-	-	-	-	-	-
Average	-	46	-	-	-	-	-	-

OPTICAL DENSITY CHART

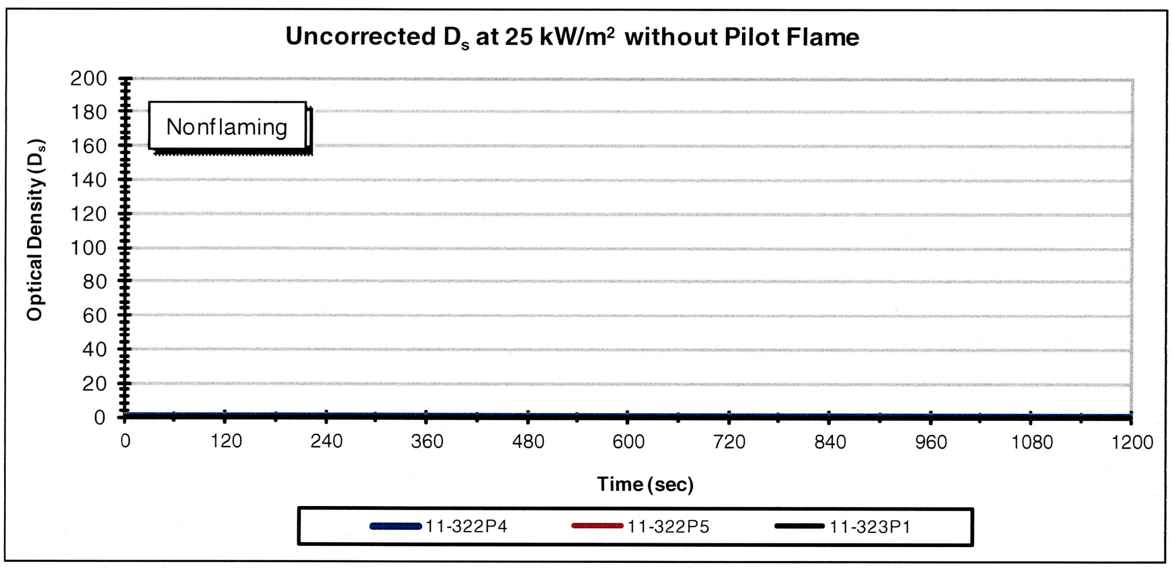

Figure 8. Smoke Density Results during Nonflaming Exposure - Resorcinol-Formaldehyde Polymer.

SOUTHWEST RESEARCH INSTITUTE

CLIENT:	**Battelle Memorial Ins. PNNL**
TASK ORDER#:	**111111-12**
SRR#:	**46069**
SDG#:	**479235**
VTSR:	**11/11/2011**
PROJECT #:	**13295.12.00X**

R13295.12.008
Instrumentation Record Sheets

Southwest Research Institute
Chemistry and Chemical Engineering Division
Department of Fire Technology

INSTRUMENTATION RECORD SHEET

Client: Battelle PNNL

Project No.: **01.12195.12.008**

Test Date: Nov 17, 2011

Test ID: ASTM D 7309

Critical Equipment	Manufacturer	Model Number	Serial Number	Next Cal Due
FLOW CONTROLER	MKS	1179A12CS1BVBZ	080363323	Oct 7, 2012
FLOW CONTROLER	MKS	179A22CS3BMBZ	080363326	Oct 7, 2012
FLOW CONTROLER	MKS	1179A51CS1BVCG	080363325	Oct 7, 2012
BALANCE	DENVER	PI-225D	23202158	Dec 23, 2011
CLASS F WEIGHT	UNKNOWN	N/A	12451	May 16, 2012
DATA ACQUISITION/CONTROL UNIT	YOKOGAWA	DU100-11	12BB23175	Dec 9, 2011

Personnel List:
A. Lowry
Nathaniel Ramos

Relative Humidity: 50 %
Ambient Temperature: 23 °C

Southwest Research Institute
Chemistry and Chemical Engineering Division
Department of Fire Technology

INSTRUMENTATION RECORD SHEET

Client: Battelle PNNL

Project No.: **01.13295.12.008**

Test Date: Nov 17, 2011

Test ID: ASTM E-1354

Critical Equipment	**Manufacturer**	**Model Number**	**Serial Number**	**Next Cal Due**
BALANCE	METTLER TOLEDO	B3001	1117512563	Nov 25, 2011
CH4 FLOW METER	BRONKHORST	F-112AC-AAD-33-V	M7205789A	May 17, 2012
CALIPER	MITUTOYO	CD-8"PS	06616887	Feb 9, 2012
CLASS F WEIGHT	UNKNOWN	N/A	12451	May 16, 2012
DATA LOGGER	OMEGA	OM-73	0510201028	Apr 19, 2012
SCHMIDT-BOELTER	MEDTHERM CORPOR	GTW-10-32-485A	99161	Mar 11, 2012

Personnel List:
Alan K Lowry

Relative Humidity: **50 %**
Ambient Temperature: **23 °C**

Mayces, Tom

From: Ehnstrom, Mark R.
Sent: Tuesday, November 29, 2011 8:12 AM
To: Tom Mayces (tom.mayces@swri.edu)
Cc: Lopez, Priscilla
Subject: Div.01

Mr. Mayces,

Yesterday I did surveillance on the Div. 01 project that you started last week. I think the client is Bechtel. Here is the information for the test I witnessed yesterday:

Flowmeters:

Asst # 012391	Cal. Date 1/20/11	Cal Due 12/20/11
012392	8/23/11	8/23/12

Radiometer:

011860	3/11/11	3/11/12

Pressure Transducer:

012828	9/22/11	9/22/12

Scale:

004688	1/29/11	12/29/11

0-6" Caliper

011786	11/21/11	11/21/12

The test performed was a "Flaming IMO Part 2 Test". It was a 20 minute test and because of the material provided by the client, it was a E662 compliant test. I didn't know if you were going to do a surveillance report or not.

Best regards,

Mark

SOUTHWEST RESEARCH INSTITUTE

CLIENT:	Battelle Memorial Ins. PNNL
TASK ORDER#:	111111-12
SRR#:	46069
SDG#:	479235
VTSR:	11/11/2011
PROJECT #:	13295.12.00X

SAMPLE RECEIPT, TASK ORDER
&
CHAIN OF CUSTODY

Sample Receipt

Sample Receipt Number: 46069

Southwest Research Institute | VTSR: 11/11/11 | Time: 08:30:00

Project: 13295.12.00X
Case #: 167930
Client: BATTELLE PNNL

Manager: HORTON, GENE
Logged in by: DROMAN
Creation Date: 11/11/11

Notes

Samples were received intact at 22.0 °C (No ice).
Fed Ex #404742686287

Parameters: Analysis/located on Task Order.

See chain-of-custody as part of the SRR system for more information.

Phases:
001 - admin
007 - drg
008 - Fire Tech
*** DROMAN Nov 11 2011 9:43AM ***

System ID	Customer ID	CED	Matrix	Containers	Special Reqs.
479235	RESORCINOL-FORMALDEHYDE POLYMER		Liquid	1	

Containers: 1 **Samples: 1**

These documents are associated with this receipt: 109898[COC for SRR 46069], 109900[MSDS for SRR 46069], 109148[RFP #190709], 109444[Tech Prop], 109445[Cost Prop], 109773[Contract 167930]

Thermometer: 027
Temperature: 22.0

Client: BATTELLE PNNL SR#: 46069 FRM-002

Page Printed: 16 Dec 2011 16:14:14
ver (9/12/2011)

Southwest Research Institute

Laboratory Task Order

TO #: 111111-12 Revision: 1

SDG: 479235
VTSR: 11/11/11
RFP: 190709

SRR #'s: 46069
Client(s): BATTELLE PNNL

Project(s): 13295.12.00X
Manager(s): HORTON, GENE
To PM: 11/28/11
To QA: 12/13/11
To Client: 12/14/11

Instructions

BATTELLE MEMORIAL INSTITUTE - PACIFIC NORTHWEST NATIONAL LABORATORY. (BATTELLE PNNL)
RFP 190709. CONTRACT #167930

17-DAY TAT FOR PRELIMINARY RESULTS.
33-DAY TAT FOR FINAL DATA PACKAGE TO CLIENT.

1 OVERALL SAMPLE (1 CONTAINERS) RECEIVED ON 11/11/11 FOR THE REQUESTED ITEMS UNDER RFP 190709.

PLEASE CONTACT GENE HORTON X3457 WITH ANY ADDITIONAL QUESTIONS OR ALSO SEE THE RFP.

BRIEF SUMMARY OF REQUIREMENTS:
RESIN FLASH IGNITION TEMPERATURE (FIT) CONE CALORIMETER & HOT-AIR FURNACE METHOD ASTM D 1929
CRITICAL HEAT FLUX (PERFORMED PRIOR TO REGULAR 1354 TEST) CONE CALORIMETER ASTM E 1354
HEAT RELEASE RATES, MASS LOSS RATES CALCULATED THERMAL RESPONSE PARAMETER (TRP)□CONE CALORIMETER ASTM E 1354
EFFECTIVE HEATS OF COMBUSTION VALUES OBTAINED THROUGH THERMAL DECOMPOSITION MICROSCALE COMBUSTION CALORIMETER ASTM D 7309-07A, METHOD A
NET CALORIFIC VALUE OR NET HEAT OF COMBUSTION VS. TOTAL HEAT OF COMBUSTION. VALUES OBTAINED THROUGH OXIDATIVE DECOMPOSITION MICROSCALE COMBUSTION CALORIMETER ASTM D 7309-07B METHOD B
SOOT AND SMOKE YIELD FTIR ASTM E800C 2
PARTICLE SIZE DISTRIBUTION OF GENERATED PARTICLES PARTICLE CAPTURE DURING ADDITIONAL TEST ASTM 662
VISIBLE SMOKE DEVELOPMENT SPECIFIC OPTICAL DENSITY OF SMOKE ASTM E 662
SAMPLE PREPARATION ALL TESTS

****

CLIENT CONTACT IS MS. RENEE RUSSELL 509.372.4708, RENEE.RUSSELL@PNL.GOV; PROJECT MANAGER REID PETERSON 509.375.2464.
HASQARD REQUIREMENTS. SEE ALSO SOW 58008.

EMAIL PRELIMINARY RESULTS TO:
RENEE.RUSSELL@PNNL.GOV; REID.PETERSON@PNNL.GOV; MICHAEL SCHWEIGER MIKE.SCHWEIGER@PNNL.GOV.

ALSO INCLUDE A COPY OF THE E-MAIL FOR THE PRELIM RESULTS IN THE DATA PACKAGE FOR THE FINAL REPORT. THIS SERVES AS DOCUMENTATION THAT THE TAT WAS MET.

FINAL HARDCOPY / CD TO (FULL RAW DATA PACKAGE):
BATTELLE PNNL FOR US DOE
790 6TH STREET
RICHLAND, WA 99354
ATTN: PO #167930, RENEE RUSSELL K6-24
REVISION 1, DRMZ 11/14/11: TASK ORDER REVISED TO CORRECT THE DATE RECEIVED REFERENCED UNDER THE INSTRUCTIONS TO 11/11/11.

Documents Related to this task order: 109898[COC for SRR 46069], 109900[MSDS for SRR 46069], 109148[RFP #190709], 109444[Tech Prop], 109445[Cost Prop], 109773[Contract 167930]

Deliverables --> Hard Copy: -YES- EDD: no PDF: -YES-

Test: ARCH_DRG Holding: 365 days from CED
Section: DATA REPORTING **Archive a copy for records/billing.** Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	CED	Method Date
479235		1	Liquid	RESORCINOL-FORMALDEHYDE POLYMER		

Test: ASTM D1929 Holding: 30 days from VTSR
Section: FT ADMIN **Standard Test Method For Determining Ignition Temperature Of Plastics** Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	VTSR	Method Date
479235		1	Liquid	RESORCINOL-FORMALDEHYDE POLYMER	11 Nov 11	11 Dec 11

Southwest Research Institute

Laboratory Task Order

TO #: 111111-12 Revision: 1

SDG: 479235
VTSR: 11/11/11
RFP: 190709

SRR #'s: 46069
Client(s): BATTELLE PNNL

Project(s): 13295.12.00X
Manager(s): HORTON, GENE
To PM: 11/28/11
To QA: 12/13/11
To Client: 12/14/11

Test: ASTM D7309
Section: FT ADMIN

Holding: 14 days from VTSR

Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry

Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	VTSR	Method Date
479235		1	Liquid	RESORCINOL-FORMALDEHYDE POLYMER	11 Nov 11	25 Nov 11

Test: ASTM E1354
Section: FT ADMIN

Holding: 30 days from VTSR

Standard Test Method For Heat And Visible Smoke Release Rates For Materials And Products Using An Oxygen Consumption Calorimeter (Cone Calorimeter)

Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	VTSR	Method Date
479235		1	Liquid	RESORCINOL-FORMALDEHYDE POLYMER	11 Nov 11	11 Dec 11

Test: ASTM E662
Section: FT ADMIN

Holding: 30 days from VTSR

Standard Test Method For Specific Optical Density Of Smoke Generated By Solid Materials (Smoke Chamber)

Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	VTSR	Method Date
479235		1	Liquid	RESORCINOL-FORMALDEHYDE POLYMER	11 Nov 11	11 Dec 11

Test: ASTM E800
Section: FT ADMIN

Holding: 30 days from VTSR

Standard Guide For Measurement Of Gases Present Or Generated During Fires (Gas Sampling Protocol)

Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	VTSR	Method Date
479235		1	Liquid	RESORCINOL-FORMALDEHYDE POLYMER	11 Nov 11	11 Dec 11

109898

General Sample Chain of Custody

Ver (8/25/2011)

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

SRR #:	46069	Thermometer:	027
Project	13295.12.00X	Temperature:	22.0
CASE:	167930	Airbill #:	404742686287
Customer:	BATTELLE PNNL	Logged in by:	DROMAN
Samples Received:	Nov 11 2011 8:30AM	Logged in:	Nov 11 2011 9:43AM
Manager:	HORTON, GENE		
Disposal Instructions:	Contact Eugen Horton for sample disposal authorization. *** DROMAN Nov 11 2011 9:43AM ***		

System ID	Customer ID	CED	Matrix	# Cont	Special Requirements	Sample Condition
479235	RESORCINOL-FORMALDEHYDE POLYMER		Liquid	1		Intact

	Date	Time
Reliquished by (Print/Signature): FED EX	11/11/11	0830
Received by (Print/Signature): DINO ROMAN / [signature]	11/11/11	0830
Reliquished by (Print/Signature):		
Received by (Print/Signature):		
Reliquished by (Print/Signature):		

Southwest Research Institute

Traffic Report

Sample Custodian Signature: ______________________

Project: 13295.12-00X

Case: 167930 / SDG: ____________

Sample Receipt: 46069

Airbill: 404742686287

1. Custody Seal — Not Present or Damaged
2. Chain of Custody — Not Present
3. Sample Tags — Not Present
   Sample Tag Numbers — Not on COC
4. SMO Forms — Not Present

Date Received	Time Received	COC Record	SMO Sample #	Corresponding		Traffic Rpt, Tags, COC Agree	Sample Condition
				Sample Tag #	SwRI #		
11/11/11	08:30:00	SwRI COC	RESORCINOL-FORMALDEHYDE POLYMER	None	479235	YES	Intact

Client: BATTELLE PNNL

WO: 46069

FRM-217

SAMPLE LOG-IN SHEET

Lab Name	
Southwest Research Institute	Page 1 of 1
Received By (Print Name): DINO ROMAN	Log-in Date: 11/11/2011
Received By (Signature): DR	

Case Number	Sample Delivery Group No.	SAS Number
167930	NA	NA

Remarks: 13295.12.00X

				Corresponding		Remarks: Condition of Sample Shipment, etc
			EPA Sample #	Sample Tag #	Assigned Lab #	
1.	Custody Seal(s)	Present/(Absent*) Intact/Broken	RESORCINOL-FOR MALDEHYDE POLYMER	None	479235	Intact
2.	Custody Seal Nos.	NA				
3.	Chain-of Custody Records	Present/(Absent*)				
4.	Traffic Reports or Packing Lists	Present/(Absent*)				
5.	Airbill	Airbill/Sticker (Present)/Absent*				
6.	Airbill No.	404742686287				
7.	Sample Tags	Present/(Absent*)				
	Sample Tag Numbers	Listed/(Not) listed on Chain of Custody				
8.	Sample Condition	(Intact)/Broken*/ Leaking				
9.	Cooler Temperature	22.0C				
10.	Does Information on custody records, traffic reports, and sample tags agree?	(Yes)/No*				
11.	Date Received at Lab	11/11/2011				
12.	Time Received	08:30:00				

Sample Transfer

Fraction	Fraction
Fire Tech.	
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By	By
DINO ROMAN	
On	On
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* Contact SMO and attach record of resolution

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ASTM D 1929 – 96 (RE-APPROVED 2001), *STANDARD TEST METHOD FOR DETERMINING IGNITION TEMPERATURE OF PLASTICS*

MATERIAL ID: *RESORCINOL-FORMALDEHYDE POLYMER*

FINAL REPORT (Revised)
Consisting of 10 Pages

SwRI® Project No. 01.13295.12.008a
Test Dates: November 29, and December 1–3 and 5, 2011
Report Date: February 2, 2012

Prepared for:

Battelle Memorial Institute
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1.0 INTRODUCTION

ASTM D 1929, *Standard Test Method for Determining Ignition Temperature of Plastics,* covers a laboratory determination of the spontaneous ignition temperature (SIT) and flash ignition temperature (FIT) of plastics using a hot-air furnace. The hot-air ignition furnace consists primarily of an electrical heating unit and specimen holder. The furnace tube is a vertical tube with an inside diameter of 100 ± 5 mm and a length of 230 ± 20 mm, made of ceramic that will withstand at least 750 °C. The inner ceramic tube, with an inside diameter of 75 ± 5 mm, a length of 230 ± 20 mm, and a thickness of approximately 3 mm, is placed inside the furnace tube and positioned 20 ± 2 mm above the furnace floor on spacer blocks. The pilot flame is located immediately above the opening. The test apparatus is shown in Figure 1.

Testing was performed on the test samples in accordance with the quality assurance requirements of the DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents, Volumes 1 and 4 (HASQARDS), latest revision. In accordance with the HASQARDs, a positive, a negative, and duplicate tests were run, in addition to the one standard test. The positive tests were performed on PMMA (Poly methyl methacrylate). This is a know standard used in testing and it was selected as the positive standard. The negative tests were performed on marinate board, which is a non-combustible material. This material is often used as a substrate or backer board in standard fire testing. It does not react to flame or heat except to lose moisture.

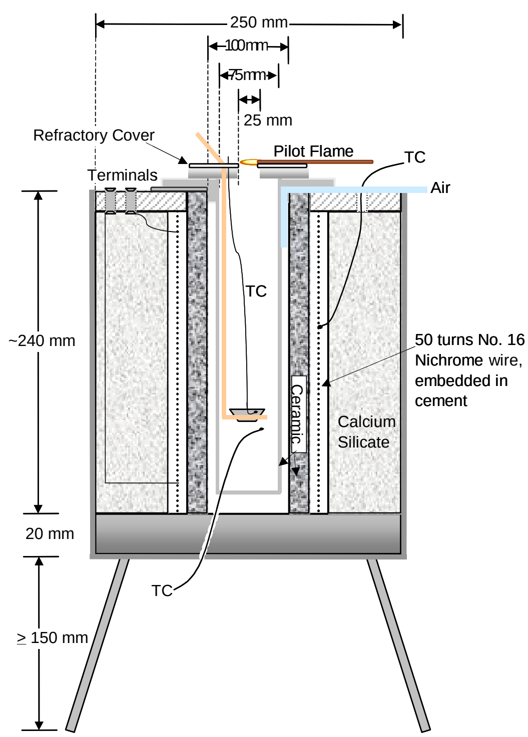

Figure 1. Schematic of Southwest Research Institute (SwRI) Hot-Air Furnace.

SIT is the minimum temperature at which the self-heating properties of the specimen lead to ignition or ignition occurs of itself, under specified test conditions, in the absence of any additional flame ignition source. The lowest air temperature at which the specimen ignites during a 10-min period is recorded as the spontaneous ignition temperature.

FIT is the minimum temperature at which, under specified test conditions, sufficient flammable gases are emitted to ignite momentarily upon application of a small external pilot flame. The lowest air temperature at which a flash is observed during a 10-min period is recorded as the flash ignition temperature.

2.0 SAMPLE IDENTIFICATION AND PREPARATION

Battelle Memorial Institute, located in Richland, Washington, provided a material identified as *Resorcinol-Formaldehyde Polymer* for testing in accordance with ASTM D 1929. A description of the material provided by the client can be found in Table 1. On November 11, 2011, SwRI received one 20-L bottle of specimen described in the table below.

TABLE 1. SAMPLE DESCRIPTION PROVIDED BY BATTELLE MEMORIAL INSTITUTE.

Material ID	Description	Color
Resorcinol-Formaldehyde Polymer	Micro Bead	Red

The samples provided consisted of a micro bead material dispersed in water. The material was tested in worst case scenario, which is with the water removed. This process was achieved by first filtering the material, and then drying it in an oven. The sample was dried for a minimum of 10 h at 60 °C ± 3 °C (140 °F ± 5 °F) prior to testing. After drying, the sample consisted of only the micro bead material; the beads became statically charged in the absence of the water medium and; therefore, they were placed in a closed container after drying, in an effort not to lose any beads. Each sample was then conditioned in a controlled environment maintained at 23 °C ± 3 °C and 50% ± 5% relative humidity until just prior to testing, but for not less than 40 h prior to testing.

3.0 RESULTS

Testing was conducted November 29, and December 1–3 and 5, 2011. Table 2 contains the results for the material provided by Battelle Memorial Institute. Test results are accurate to ± 5 °C, and are presented in detail at the end of this report. These test results relate only to the behavior of test specimens under the particular conditions of the test. They are not intended to be used, and shall not be used, to assess the potential fire hazards of a material in use.

TABLE 2. IGNITION TEMPERATURE DATA.

Material ID	SIT	FIT
Resorcinol-Formaldehyde Polymer	615 °C	600 °C
Resorcinol-Formaldehyde Polymer (Duplicate Run)	610 °C	590 °C
Marinite, Negative Standard	NA	NA
PMMA, Positive Standard	420 °C	NA

APPENDIX A

ASTM D 1929 TEST DATA SHEETS

(CONSISTING OF 6 PAGES)

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ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 3 and 5, 2011	*Date Prepared by SwRI:*	Prepared within 48h test
Material ID:*	PMMA, Positive Standard	*Color:*	Black
Description:*	Solid Black Composite	*Average Sample Mass:*	3.01 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 420

** Information/instructions provided by the Client*

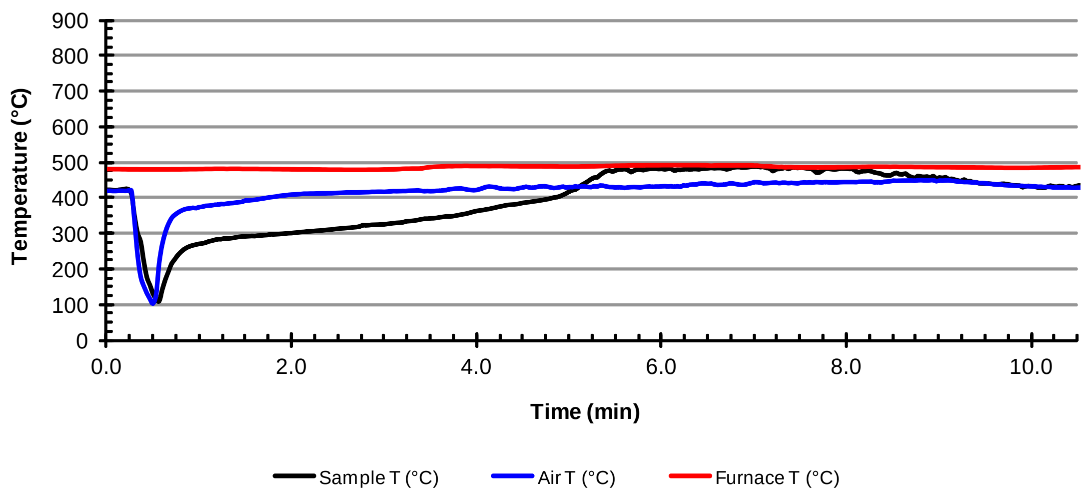
RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-337PNN008F65	3.15	0.75	2.40	417	410	467	422	407	474	No
11-337PNN008F45	2.87	0.00	2.87	423	**420**	479	434	429	484	Yes

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-337PNN008F65	0:36	NA	NA	None	Yes	None	Yes	10:36
11-337PNN008F45	0:34	NA	5:15	None	Yes	None	None	10:34

Positive Standard SIT Test



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ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowery	*Receipt Date:*	In stock
Test Date(s):	December 2, 2011	*Date Prepared by SwRI:*	Prepared within 48h test
Material ID:*	Marinate	*Color:*	White
Description:*	Solid Fire resistant backer board	*Average Sample Mass:*	2.89 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : NA

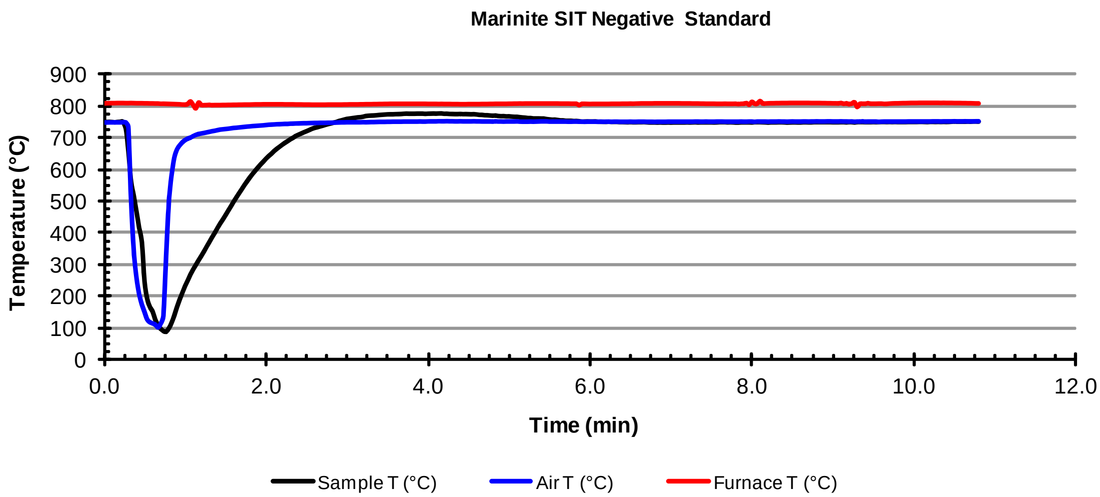
** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN008E1S	2.89	2.62	0.27	750	**750**	809	751	753	809	NO

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN008E1S	0:47	NA	NA	None	NO	None	None	10:00



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ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	Nathaniel Ramos	*Receipt Date:*	November 11, 2011
Test Date(s):	November 29, 2011	*Date Prepared by SwRI:*	Prepared within 48h test
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 615

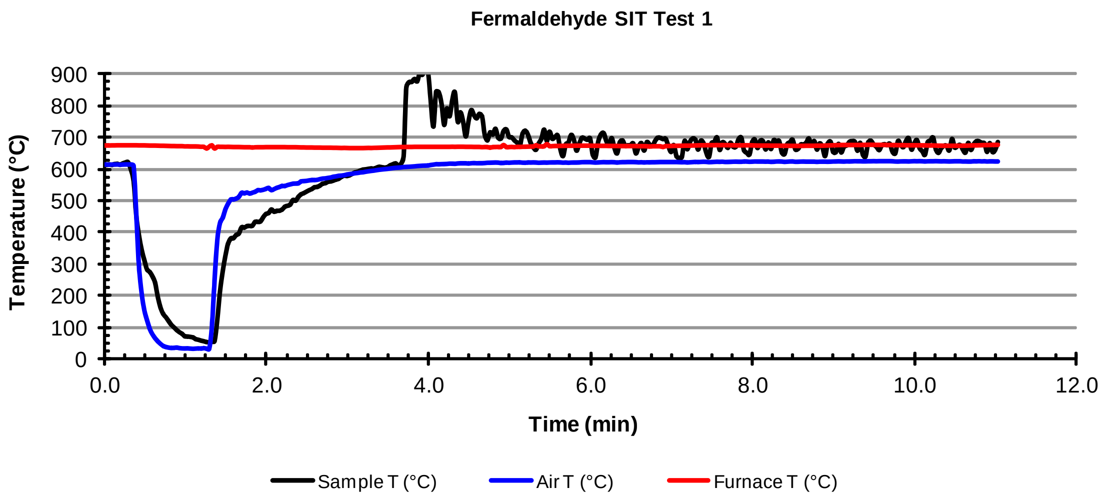
** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-333PNN00A75	3.00	1.31	1.69	609	605	603	633	614	662	No
11-333PNN00A65	3.00	1.24	1.76	615	**615**	675	670	626	678	Yes
11-333PNN00A55	3.00	1.25	1.75	630	625	684	676	635	687	Yes

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-333PNN00A75	1:56	NA	0:00	None	Yes	None	None	10:00
11-333PNN00A65	1:23	NA	2:21	None	Yes	None	None	10:00
11-333PNN00A55	1:42	NA	2:35	None	Yes	None	None	10:00



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ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	Nathaniel Ramos	*Receipt Date:*	November 11, 2011
Test Date(s):	December 1, 2011	*Date Prepared by SwRI:*	Prepared within 48h test
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

** Information/instructions provided by the Client*

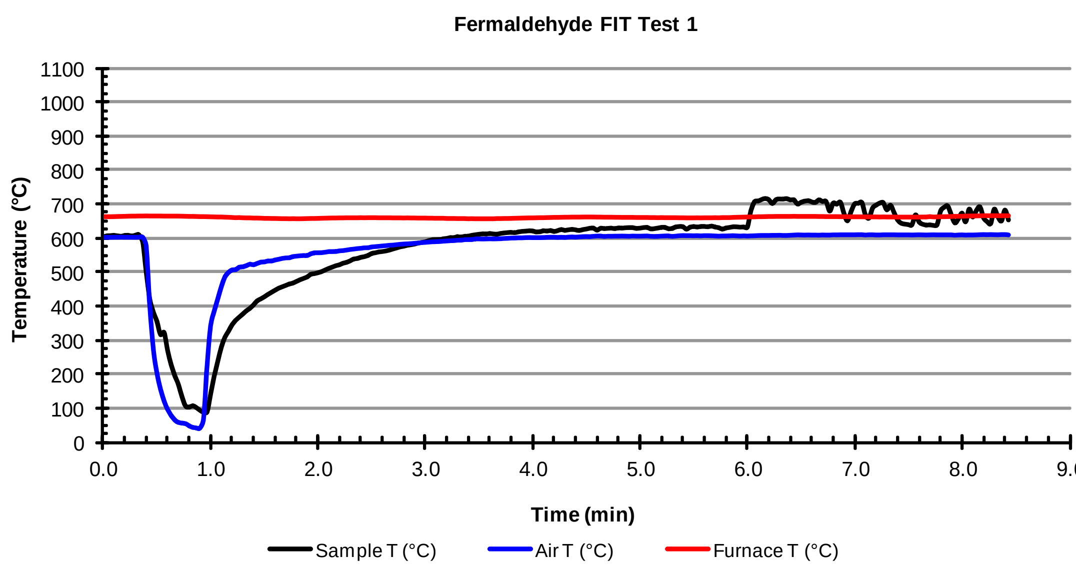
FLASH IGNITION TEMPERATURE (°C) : 600

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN00B4F	3.00	1.53	1.47	591	590	649	628	598	648	No
11-335PNN00B1F	3.00	1.66	1.34	603	**600**	660	653	608	663	Yes

FLASH IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Type (min:s)	Observed Soot (min:s)	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN00B4F	0:40	NA	NA	None	Yes	None	None	10:40
11-335PNN00B1F	0:59	NA	Flaming at 5:15	None	Yes	None	None	8:26



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ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 2, 2011	*Date Prepared by SwRI:*	Prepared within 48h test
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 610

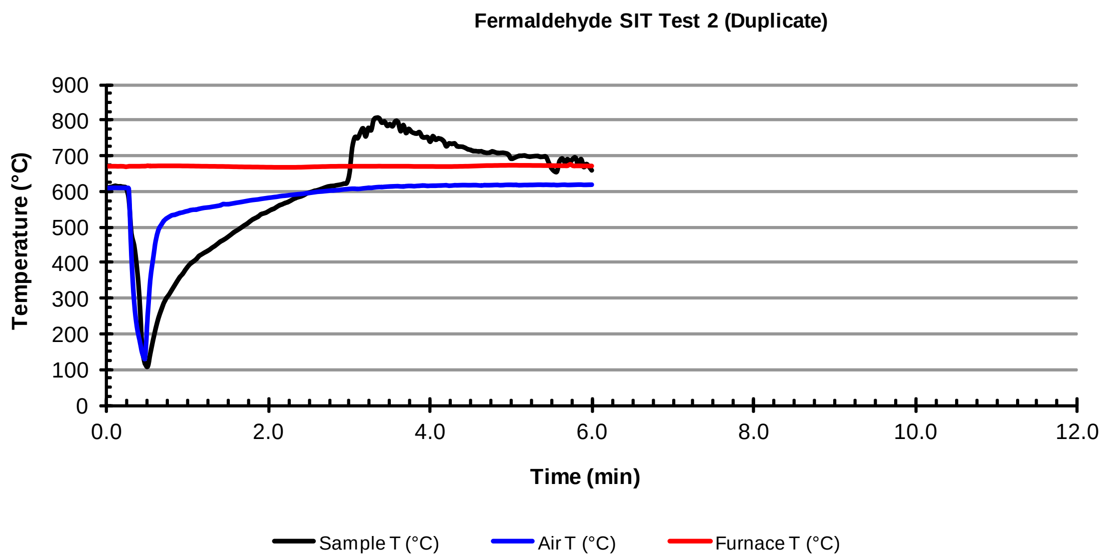
** Information/instructions provided by the Client*

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN00D1S	3.00	1.54	1.46	603	600	658	640	609	658	No
11-336PNN00D2S	3.00	1.76	1.24	611	**610**	669	659	619	670	Yes

SPONTANEOUS IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Time (min:s)	Observed Soot	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN00D1S	0:34	NA	NA	None	Yes	None	None	10:34
11-336PNN00D2S	0:31	NA	2:34	None	Yes	None	None	6:00



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ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	A. Lowry	*Receipt Date:*	November 11, 2011
Test Date(s):	December 2, 2011	*Date Prepared by SwRI:*	Prepared within 48h test
Material ID:*	Resorcinol-Formaldehyde Polymer	*Color:*	Red
Description:*	Small Beads	*Average Sample Mass:*	3.00 g

** Information/instructions provided by the Client*

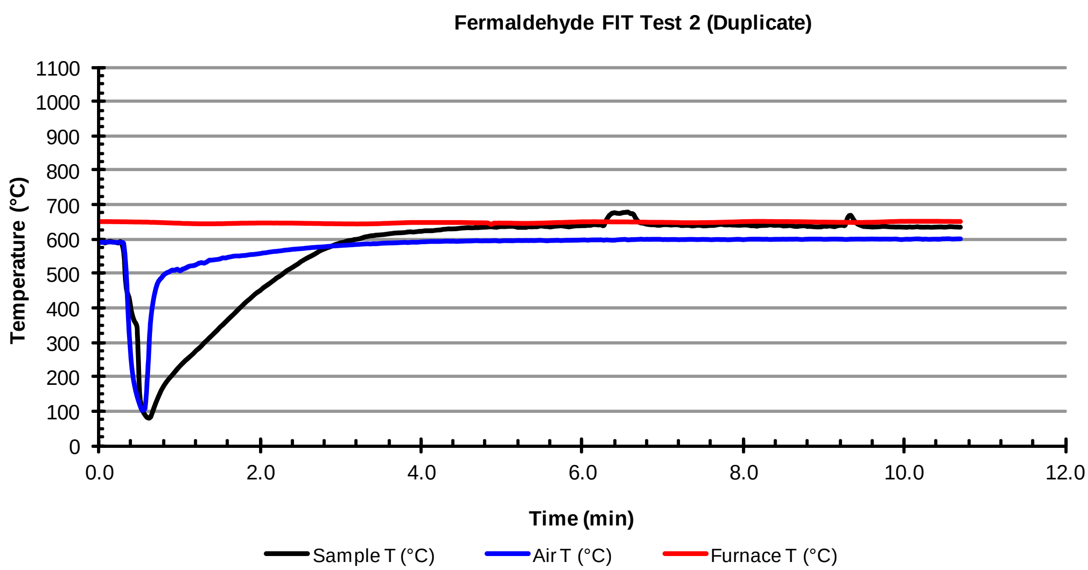
FLASH IGNITION TEMPERATURE (°C) : 590

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
11-336PNN00C3F	3.00	1.57	1.43	599	580	639	622	590	641	Yes
11-335PNN00C2F	3.00	1.45	1.55	589	**590**	649	633	600	649	Yes
11-335PNN00C1F	3.00	1.40	1.60	576	600	659	639	611	660	No

FLASH IGNITION OBSERVATIONS

	Insertion Time (min:s)	Observed Smoke (min:s)	Combustion Type (min:s)	Observed Soot (min:s)	Observed Charring	Observed Melt	Observed Bubbling	Total Test Time (min:s)
11-336PNN00C3F	0:40	NA	NA	None	Yes	None	None	10:40
11-335PNN00C2F	0:41	NA	Flaming at 5:39	None	Yes	None	None	10:42
11-335PNN00C1F	0:41	NA	Flaming at 5:15	None	Yes	None	None	10:42



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