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WTP-RPT-220, Rev 0

U.S. DEPARTMENT OF ENERGY

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

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

D Kim
MJ Schweiger
RA Peterson

September 2012

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Fire Safety Tests for Cesium-Loaded 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-11-00004, 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 ______________________ 9/18/12 ______________

Dean E. Kurath, Manager — Date

WTP R&T Support Project

Testing Summary

A preliminary 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 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 resolve this question, the fire properties of the SRF resin were measured by Southwest Research Institute (SwRI) through a subcontract managed by Pacific Northwest National Laboratory (PNNL).

The results of initial fire safety tests on the SRF resin were documented in a previous report (PNNL-21321). The present report summarizes the results of additional tests performed by SwRI on the cesium-loaded SRF resin. The efforts by PNNL were limited to summarizing the test results provided by SwRI into one consolidated data report. The as-received SwRI report is attached to this report in the Appendix A. Where applicable, the precision and bias of each test method, as given by each American Society for Testing and Materials (ASTM) standard procedure, are included and compared with the SwRI test results of the cesium-loaded SRF resin.

Objectives

The objectives for the fire safety tests were:

- to measure the thermal conductivity and heat capacity for sodium-form SRF resin,
- to perform thermo-gravimetric analysis (TGA) with mass spectrometry of the nonradioactive cesium spiked resin offgas up to the auto ignition temperature and determine constituents of the offgas, and
- to repeat ignition test using resin loaded with nonradioactive cesium.

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

The 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
• Measure the thermal conductivity and heat capacity for sodium-form SRF resin.	Yes	The thermal conductivity of the cesium-loaded SRF resin was measured in accordance with ASTM D5930-09 and ASTM D5334-08. The thermal conductivity (λ in W/[mK]) as a function of temperature (T in °C) between 22 and 90°C was expressed by $\lambda = 0.000273T + 0.0769$. The specific heat capacity of the cesium-loaded SRF resin was measured by differential scanning calorimetry (DSC) in accordance with ASTM E1269-11. The average specific heat capacity ranged from 1.434 J/(gK) at 40°C to 2.001 J/(gK) at 90°C.
• Perform TGA with mass spectrometry of the nonradioactive cesium spiked resin offgas up to the auto ignition temperature and determine constituents.	Partially Yes	From the TGA combined with DSC analysis, an endothermic transition near 102°C appeared with a total weight loss of approximately 25%. Small exothermic transitions occurred around 387°C and 619°C with a large exothermic transition near 864°C with corresponding weight losses of approximately 30%, 5%, and 20%, respectively. Because SwRI did not have a TGA setup with mass spectrometer, the sample was run with TGA setup integrated with Fourier transform infrared (FTIR) for offgas analyses. The result of FTIR analyses showed that the weight loss peak at 102°C was linked to H_2O while all other peaks at higher temperature were linked to CO_2 gas.
• Repeat ignition test using resin loaded with nonradioactive cesium.	Yes	The flash ignition temperature (FIT) and spontaneous ignition temperature (SIT) of the cesium-loaded SRF resin were determined in accordance with ASTM D1929-96. The average values of FIT and SIT from duplicate tests were 555°C and 610°C, respectively. The FIT is 40°C lower than the previous result for the SRF resin without cesium and SIT is only 3°C lower, which is well below the repeatability limits of 11 to 31°C.

Table S.2. Test Exception

Test Exception Number	Description of Test Exception
24590-PTF-TEF-RT-11-00004, Rev 0	This test exception was issued by Bechtel National, Inc. (BNI) on December 21, 2011. This test exception incorporates the approved Request for Technology Development (RTD), 24590-WTP-RTD-RT-11-0008_Rev_000, which requests data on thermal conductivity, heat capacity, and thermal analysis of the SRF resin and repeat of the ignition tests using the cesium-loaded SRF 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 thermal conductivity and heat capacity measurements of sodium-form SRF resin.	This success criterion was met. The thermal conductivity (λ in W/[mK]) of the cesium-loaded SRF resin as a function of temperature (T in °C) between 22 and 90°C was expressed by $\lambda = 0.000273T + 0.0769$. The average thermal conductivity was 0.083 W/(mK) at 25°C, 0.088 W/(mK) at 45°C, and 0.102 W/(mK) at 90°C. The average specific heat capacity of the cesium-loaded SRF resin ranged from 1.434 J/(gK) at 40°C to 2.001 J/(gK) at 90°C.
2. Provide test results for TGA with mass spectrometry of the nonradioactive cesium spiked resin offgas up to the auto ignition temperature and the determined constituents.	This success criterion was partially met. From the TGA-DSC analysis, an endothermic transition near 102°C appeared with a total weight loss of approximately 25%. Small exothermic transitions occurred around 387°C and 619°C and a large exothermic transition near 864°C with corresponding weight losses of approximately 30%, 5%, and 20%, respectively. Because SwRI did not have a TGA setup with mass spectrometer, the sample was run with TGA-FTIR setup for offgas analyses. The result of FTIR analyses showed that the weight loss peak at 102°C was linked to H_2O while all other peaks at higher temperature were linked to CO_2 gas.
3. Provide test results for the spontaneous ignition temperature using resin loaded with nonradioactive cesium.	This success criterion was met. For the cesium-loaded SRF resin, the FIT was 555°C, and SIT was 610°C.

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. The SRF resin was loaded with cesium by SwRI according to the procedure supplied by PNNL.

Table S.4. Test-Condition Summary

Test Conditions	Were Test Conditions Followed?
1. The ignition temperature of the resin loaded with non-radioactive cesium will be determined along with the thermal conductivity and heat capacity of the resin in sodium-form. A TGA with mass spectrometry of the resin offgas up to the auto ignition temperature will also be performed and the constituents determined.	The FIT and SIT were determined in accordance with ASTM D1929-96, the thermal conductivity was measured in accordance with ASTM D5930-09 and ASTM D5334-08, and the specific heat capacity was measured by DSC in accordance with ASTM E1269-11. A TGA-DSC was performed along with FTIR to determine the gaseous constituents.

Discrepancies and Follow-On Tests

No discrepancies were observed.

Acknowledgments

The authors thank the Southwest Research Institute (Eugene Horton and Radonna Spies, 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.
CXP	Cesium Ion Exchange Process System
DOE	U.S. Department of Energy
DSC	differential scanning calorimetry
FIT	flash ignition temperature
FTIR	Fourier transform infrared
GGRF	ground gel resorcinol formaldehyde
NIST	National Institute of Standards and Technology
NSSP	non-steady-state probe
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
R&T	research and technology
RF	resorcinol formaldehyde
RTD	request for technology development
SIT	spontaneous ignition temperature
SRF	spherical resorcinol formaldehyde
SwRI	Southwest Research Institute
TGA	thermo-gravimetric analysis
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WTPSP	Waste Treatment Plant Support Project

Symbols

C	calibration constant
I	current flowing through heater wire
L	length of heated needle
Q	heat input per unit length of heater
r	repeatability limit
R	reproducibility limit
R_W	total resistance of heater wire
S	slop obtained from temperature versus $\ln(t)$ or $\ln[1/(t - t_1)]$
t	time from the beginning of heating
t_1	heating time
T	temperature
ΔT	temperature rise from time zero
λ	thermal conductivity

Contents

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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, the fire 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). The results of initial fire safety tests on the SRF resin were documented in a previous report (PNNL-21321). The present report summarizes the results of additional tests performed by SwRI on the cesium-loaded SRF resin.

Section 2.0 details the basis of the PNNL Quality Assurance (QA) Program as applied to the WTP quality requirements. 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 a summary of all test results. Section 6.0 provides a list of pertinent references. An as-received SwRI report is included in the Appendix A.

2.0 Quality Assurance

The PNNL 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 tests on the cesium-loaded SRF resin. Table 3.1, presented at the end of this section, summarizes the ASTM standard test procedures used by SwRI for this testing.

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. The SRF resin was loaded with cesium according to the procedure supplied by PNNL (Appendix B). For all tests described in this report, the sample was filtered and then dried for 2 to 12 hr 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.

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

The flash ignition temperature (FIT) and spontaneous ignition temperature (SIT) of the cesium-loaded 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 initial 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 initial air temperature at which the specimen ignites during a 10-min period is recorded as the SIT.

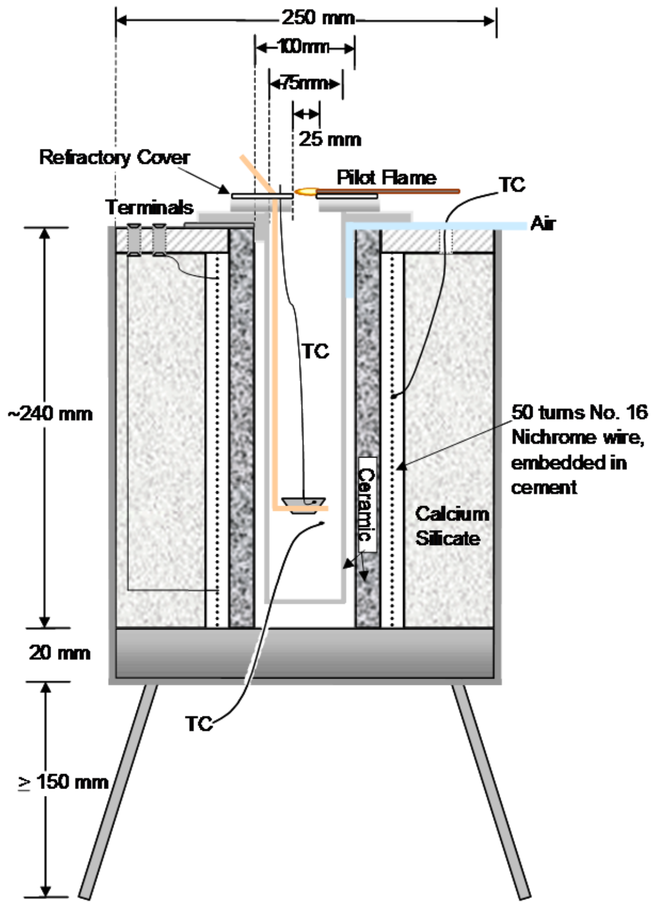

Figure 3.1. Schematic of Southwest Research Institute Hot-Air Furnace

3.3 Thermal Conductivity

The thermal conductivity of the cesium-loaded SRF resin was measured using the method as described in ASTM D5930-09, *Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique* and ASTM D5334-08, *Standard Test Method for Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure*. The Hukseflux TP02 Non-Steady-State Probe (NSSP) was used for the analysis. A Global Specialties 1305 Dual Output DC Power Supply (0-32V, 5A) was used to provide a constant current of 0.2A to the probe. Measurement Computing's USB-TEMP with TracerDAQ® was utilized to acquire the time and temperature at 1 s intervals during the duration of the test. The test material was placed into a glass cylindrical vessel with a diameter of 55 mm and a length of 200 mm. The thermal conductivity probe was inserted into the material and temperature was monitored to ensure thermal equilibrium had been reached prior to beginning the test. The program was set up to monitor the stabilized temperature for 1 min prior to beginning the test. The time the power remained on (180 s) was the "heating phase". Once the power was turned off, the temperature was acquired for an additional 6 min for the "cooling phase".

The NSSP principle relies on a unique property of a line source. After a short transient period the temperature rise is given as:

$$\Delta T = \frac{Q}{4\pi\lambda}\ln(t) \quad 0 < t \le t_1 \tag{1}$$

$$\Delta T = \frac{Q}{4\pi\lambda}\ln\left(\frac{t}{t - t_1}\right) \quad t > t_1 \tag{2}$$

where ΔT is temperature rise from time zero (K), Q is heat input per unit length of heater (W/m), λ is thermal conductivity [W/(mK)], t is time from the beginning of heating (s), and t_1 is heating time (s).

The thermal conductivity is calculated from the slope of the line representing the temperature versus ln(t) or ln[t /(t - t_1)]. A graph was constructed for each set of data (heating and cooling). The early and late portions of the test were not used in the slope analysis. For the heating phase, the slope was calculated from the ln(t) range of 2 to 3, which corresponds to 7 to 148 s. For the cooling phase, the slope was calculated for the ln[t /(t - t_1)] range of 0.5 to 3, which corresponds to 189 to 457 s. The thermal conductivity for the heating and cooling phases was calculated from the equations:

$$\lambda = \frac{CQ}{4\pi S} \tag{3}$$

$$Q = \frac{I^2 R_W}{L} \tag{4}$$

where C is calibration constant and S is slope obtained from temperature versus ln(t) or ln[t /(t - t_1)], I is current flowing through heater wire (A), R_W is total resistance of heater wire, and L is length of heated needle (m). The final conductivity reported was calculated as the average of the heating and cooling phase thermal conductivities.

The thermal conductivity probe was calibrated before use with freshly prepared Agar water (2.5 g Agar and 500 mL deionized water). The calculated thermal conductivity differed slightly from Agar water's published thermal conductivity of 0.607 W/(mK); therefore, the calibration factor, C, was 0.91659. Freshly purchased and opened glycerol was used for the calibration verifications. The thermal conductivity for the initial and final verifications was 100% of published value for glycerol.

3.4 Specific Heat Capacity

The specific heat capacity of the cesium-loaded SRF resin was measured by differential scanning calorimetry (DSC) in accordance with ASTM E1269-11, *Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry*. The test consists of heating the sample in a controlled atmosphere through a temperature region of interest. The difference in heat flow in the sample compared to a reference material and a blank pan are used to calculate the specific heat capacity.

Specific heat capacity measurement was performed using a Seiko DSC 210. The instrument was purged with 50 mL/min of nitrogen throughout the testing. For the DSC temperature program, the temperature was held isothermally at 25°C for 4 min; the temperature was then ramped at 20°C/min to 100°C and held for 5 min. The thermal curve was collected from each analytical run for sample, reference material, and blank pan. The thermal curve for the blank aluminum pan was collected to perform the blank subtraction. The DSC software performs the blank pan subtraction from samples and reference material automatically. The blank subtracted sample and reference material files were imported into the specific heat capacity calculation program. The program generated a specific heat capacity curve along with a table of specific heat capacities for the temperature range from 40°C to 90°C at 1°C interval for which sample heating rate was stable. Sapphire disk from the Perkin Elmer Specific Heat Kit (PN# 02190136) was used as the reference material. The instrument response was standardized against the Perkin Elmer sapphire disk and all sample results were reported using this standardization.

3.5 DSC-TGA-FTIR

Thermal and infrared analysis of the cesium-loaded SRF resin was performed using a TA Instruments Q600 SDT Simultaneous differential scanning calorimetry-thermo-gravimetric analysis (DSC-TGA) Heat Flow Analyzer and a Nicolet Magna-IR 560 Fourier Transform Infrared (FTIR) Analyzer. Two instruments were connected with a Nicolet TGA Interface. This allowed real time infrared analysis of the evolved gases from the thermal decomposition or volatilization of the sample.

DSC-TGA analysis was performed on a 14.39 mg sample placed in an alumina pan. The sample was heated from room temperature to 1000°C at a ramp rate of 20°C/min. An air purge flow rate of 60 mL/min was used to sweep the evolved gases through a heated transfer line (180°C) and then through the heated flow cell (225°C) of the TGA interface. The Nicolet Magna-IR 560 monitored the composition of the gas.

Table 3.1. Standard Procedures Used by Southwest Research Institute

Main Analysis/Property	Test Equipment(s)	Standard Procedures used by SwRI
Flash ignition temperature and spontaneous ignition temperature	Hot-air ignition furnace	ASTM D1929-96, *Standard Test Method for Determining Ignition Temperature of Plastics*
Thermal conductivity [W/(mK)]	Non-steady-state probe with transient line-source technique	ASTM D5930-09, *Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique* ASTM D5334-08, *Standard Test Method for Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure*
Specific heat capacity[J/(gK)]	Differential scanning calorimeter	ASTM E1269-11, *Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry*
Thermal and infrared analyses	Differential scanning calorimeter-thermo-gravimetric analyzer with Fourier transform Infrared analyzer	Not applicable

4.0 Results

The results of the cesium-loaded SRF resin fire tests reported by SwRI are summarized in this section. Where applicable, the precision and bias of each test method as given in 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 cesium-loaded 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 is 0°C for SIT and 10°C for FIT, which is within the range of the repeatability limits for FIT (8 to 13°C). 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 available. It is noted that the FIT is 40°C lower than the previous results for the SRF resin without cesium although SIT is only 3°C lower, which is well below the repeatability limits of 11 to 31°C.

Table 4.1. Flash Ignition Temperature and Spontaneous Ignition Temperature of Cesium-Loaded SRF Resin

Property	FIT (°C)	SIT (°C)
Initial Run	550	610
Duplicate Run	560	610
Average	555	610
Difference	10	0
Precision estimates[(a)]		
r	8–13	11–31
R	27–117	47–103
Negative and positive standards		
Marinite (negative standard)	No ignition	No ignition
PMMA (positive standard)	390	480

(a) Determined from interlaboratory tests involving seven laboratories on six polymeric materials with triplicate tests (ASTM D1929-96).

4.2 Thermal Conductivity Results

Table 4.2 summarizes thermal conductivities of the cesium-loaded SRF resin measured at three temperatures with three measurements at a minimum at each temperature.

Table 4.2. Thermal Conductivity with Temperature

Temperature (°C)	Thermal Conductivity [W/(mK)]	Average Thermal Conductivity [W/(mK)]
22	0.0855	0.0834
22	0.0816	
22	0.0860	
22	0.0806	
45	0.0872	0.0882
45	0.0899	
45	0.0874	
90	0.1010	0.1018
90	0.1021	
90	0.1023	

The thermal conductivity was plotted against temperature and a linear least-square fit was established as shown in Figure 4.1. The thermal conductivity at various temperatures between 22 and 90°C can be estimated from the following linear regression equation:

$$\lambda = 0.000273T + 0.0769 \quad (22°\text{C} \le T \le 90°\text{C}) \tag{5}$$

where λ is thermal conductivity [W/(mK)] and T is temperature (°C).

The results of an interlaboratory study involving this test method used for Ottawa sand and paraffin wax indicated a measurement precision of ± 10% and ± 15%, respectively, with a tendency to a positive bias (higher value) over the known values for the materials studied (ASTM D5334-08).

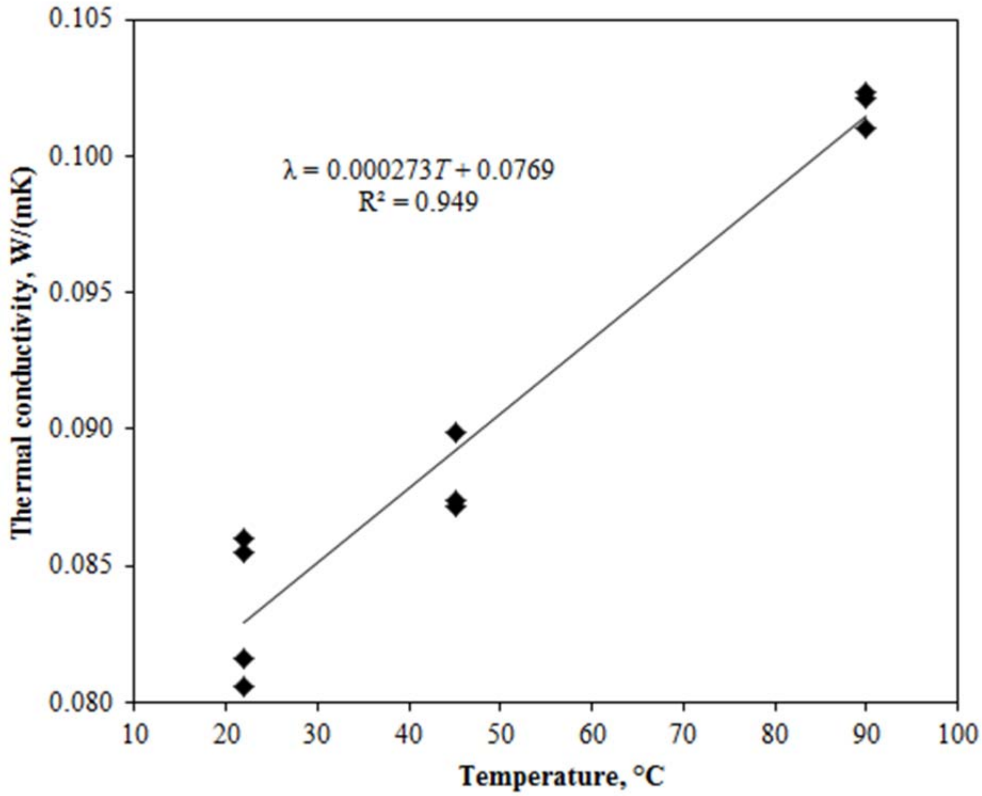

Figure 4.1. Thermal Conductivity as a Function of Temperature

4.3 Specific Heat Capacity Results

Table 4.3 summarizes the results of specific heat capacity measurements following the standard procedure ASTM E1269-11 for temperatures between 40°C and 90°C. Table 4.3 summarizes the results of duplicate tests and their average and Figure 4.2 shows the specific heat capacity as a function of temperature curves for duplicates tests and average.

The precision of the test method was determined from interlaboratory trials involving seven laboratories on three different solid materials (biphenyl ether, National Institute of Standards and Technology (NIST) linear polyethylene, and indium metal) over the temperature range from 40°C to 80°C. The relative repeatability limit was 6.2% and relative reproducibility limit was 8.4%. The relative difference from duplicate tests in this study ranged from 1.9% (at 40°C) to 3.7% (at 90°C), which is smaller than the relative repeatability limit of 6.2%.

The bias of the test method was obtained from the above mentioned interlaboratory study by comparing the mean specific heat capacity at 67°C to the literature value: the relative difference from the corresponding literature value was +0.95% for biphenyl ether, -1.1% for NIST linear polyethylene, and +0.8% or +1.8% for indium metal from two different references.

Table 4.3. Specific Heat Capacity Test Results of SRF Resin

T, C	Specific Heat Capacity [J/(gK)] 1st run	Duplicate	Average
40	1.420	1.447	1.434
41	1.432	1.459	1.446
42	1.442	1.469	1.456
43	1.450	1.478	1.464
44	1.459	1.486	1.473
45	1.468	1.495	1.482
46	1.477	1.505	1.491
47	1.487	1.516	1.502
48	1.494	1.525	1.510
49	1.501	1.532	1.517
50	1.509	1.541	1.525
51	1.515	1.549	1.532
52	1.523	1.560	1.542
53	1.531	1.569	1.550
54	1.538	1.576	1.557
55	1.545	1.585	1.565
56	1.554	1.594	1.574
57	1.562	1.603	1.583
58	1.569	1.609	1.589
59	1.577	1.616	1.597
60	1.585	1.624	1.605
61	1.595	1.634	1.615
62	1.604	1.644	1.624
63	1.615	1.655	1.635
64	1.624	1.666	1.645
65	1.633	1.676	1.655
66	1.643	1.688	1.666
67	1.653	1.699	1.676
68	1.664	1.712	1.688
69	1.675	1.724	1.700
70	1.687	1.736	1.712
71	1.699	1.748	1.724
72	1.713	1.763	1.738
73	1.727	1.780	1.754
74	1.741	1.794	1.768
75	1.755	1.809	1.782
76	1.770	1.826	1.798
77	1.787	1.845	1.816
78	1.804	1.864	1.834
79	1.818	1.879	1.849
80	1.830	1.893	1.862
81	1.844	1.908	1.876
82	1.858	1.923	1.891
83	1.871	1.938	1.905
84	1.885	1.953	1.919
85	1.898	1.968	1.933
86	1.912	1.984	1.948
87	1.926	1.998	1.962
88	1.941	2.013	1.977
89	1.954	2.026	1.990
90	1.965	2.037	2.001

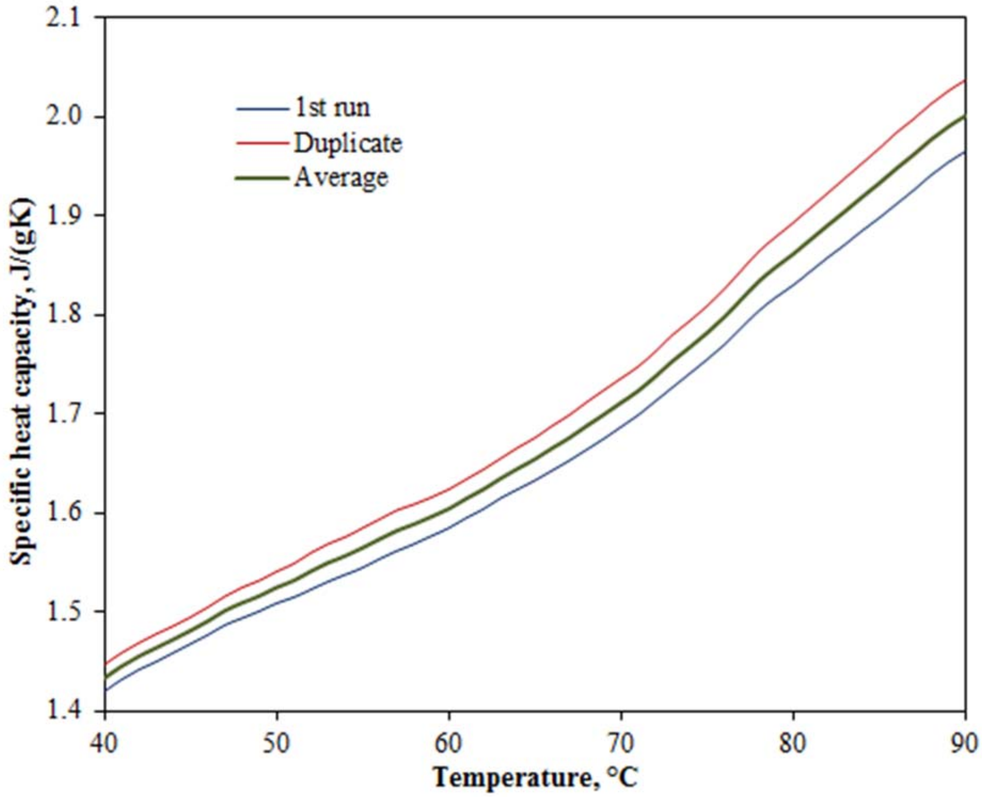

Figure 4.2. Specific Heat Capacity as a Function of Temperature

4.4 DSC-TGA-FTIR Results

Figure 4.3 shows the thermal behavior of the sample as displayed with weight change and heat flow plotted as a function of temperature. From the DSC analysis, the heat flow curve shows an endothermic transition near 102°C. This endotherm corresponded to gradual weight loss between room temperature and roughly 300°C with a total weight loss of approximately 25%. Small exothermic transitions occur around 387°C and 619°C and a large exothermic transition takes place near 864°C. Each of these exotherms corresponded to weight losses of approximately 30%, 5%, and 20%, respectively.

Figure 4.4 displays the weight change curve with the derivative overlaid. In Figure 4.5 the weight change, its derivative, and the heat flow curves are overlaid. The derivative of weight change aids in distinguishing some subtle transitions as shown in Figure 4.5. The first weight loss peak at 91°C may be due to moisture. Major weight loss peaks occur at 356°C and 866°C. Other smaller peaks appear at 340, 365, 375, 614 and 624°C.

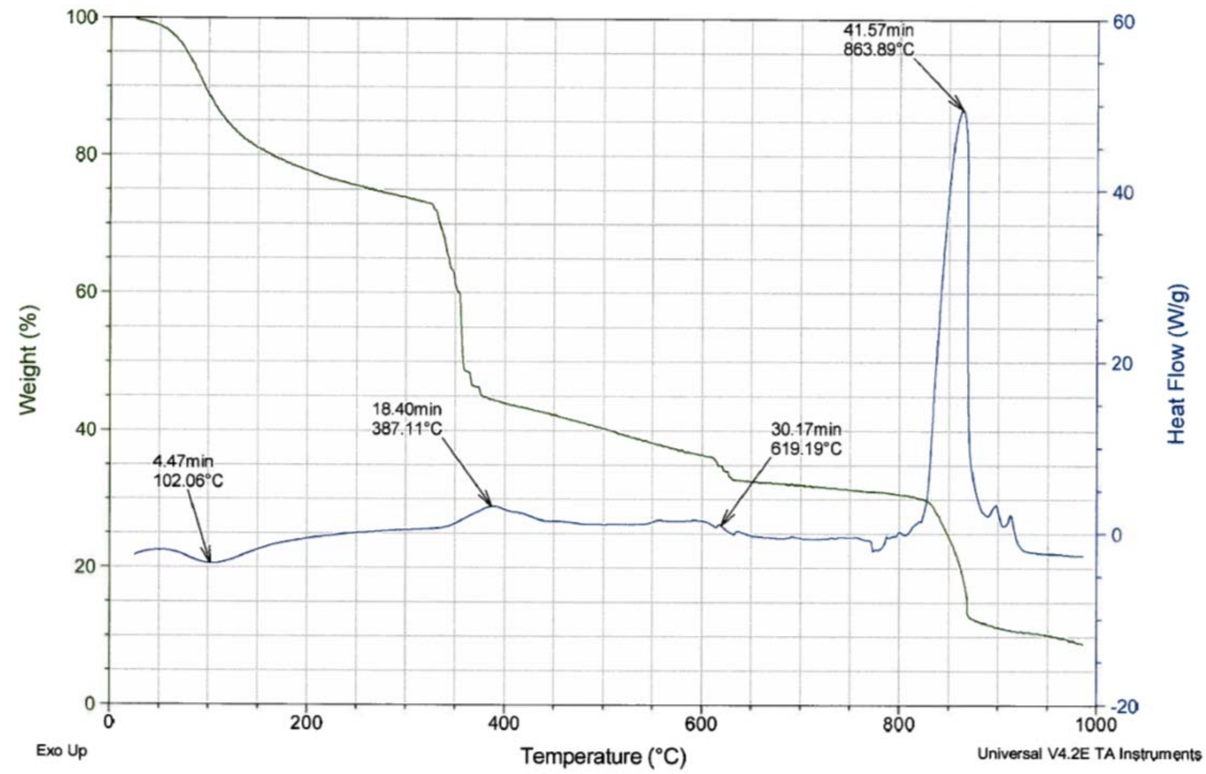

Figure 4.3. Weight Change from TGA and Heat Flow from DSC of the Cesium-Loaded SRF Resin

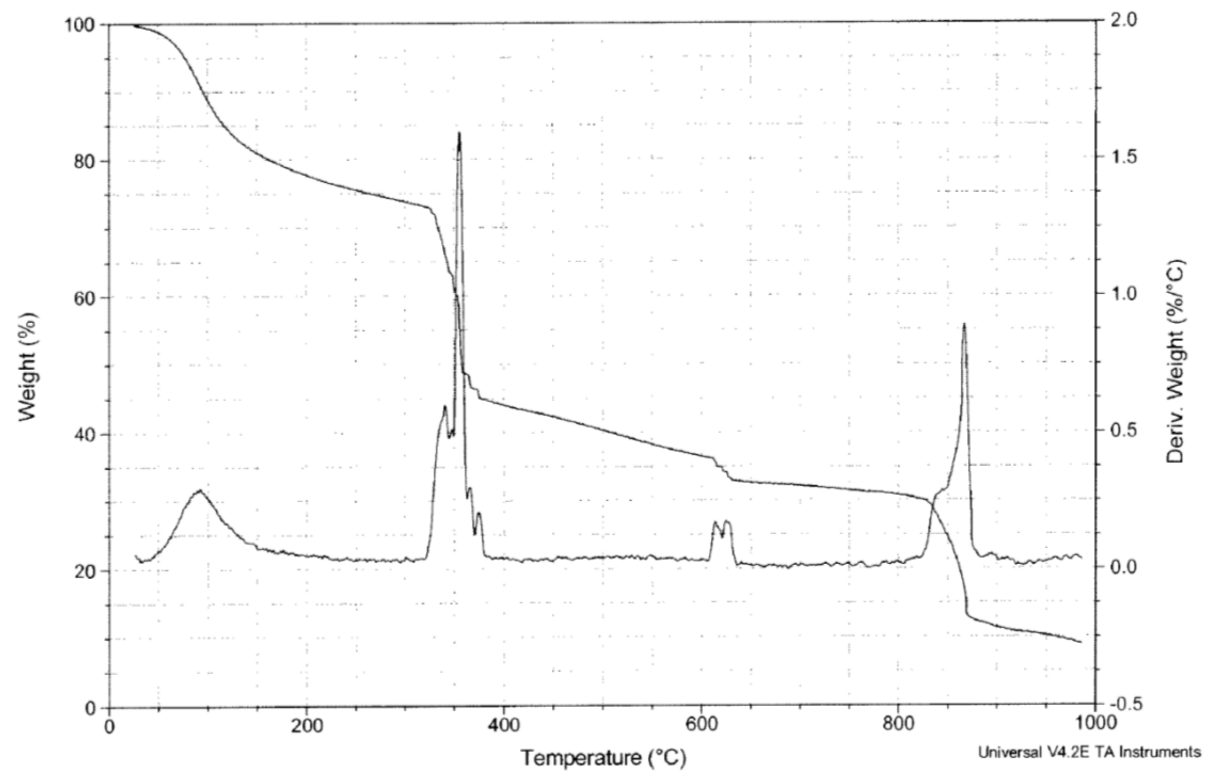

Figure 4.4. Weight Change and Its Derivative from TGA of the Cesium-Loaded SRF Resin

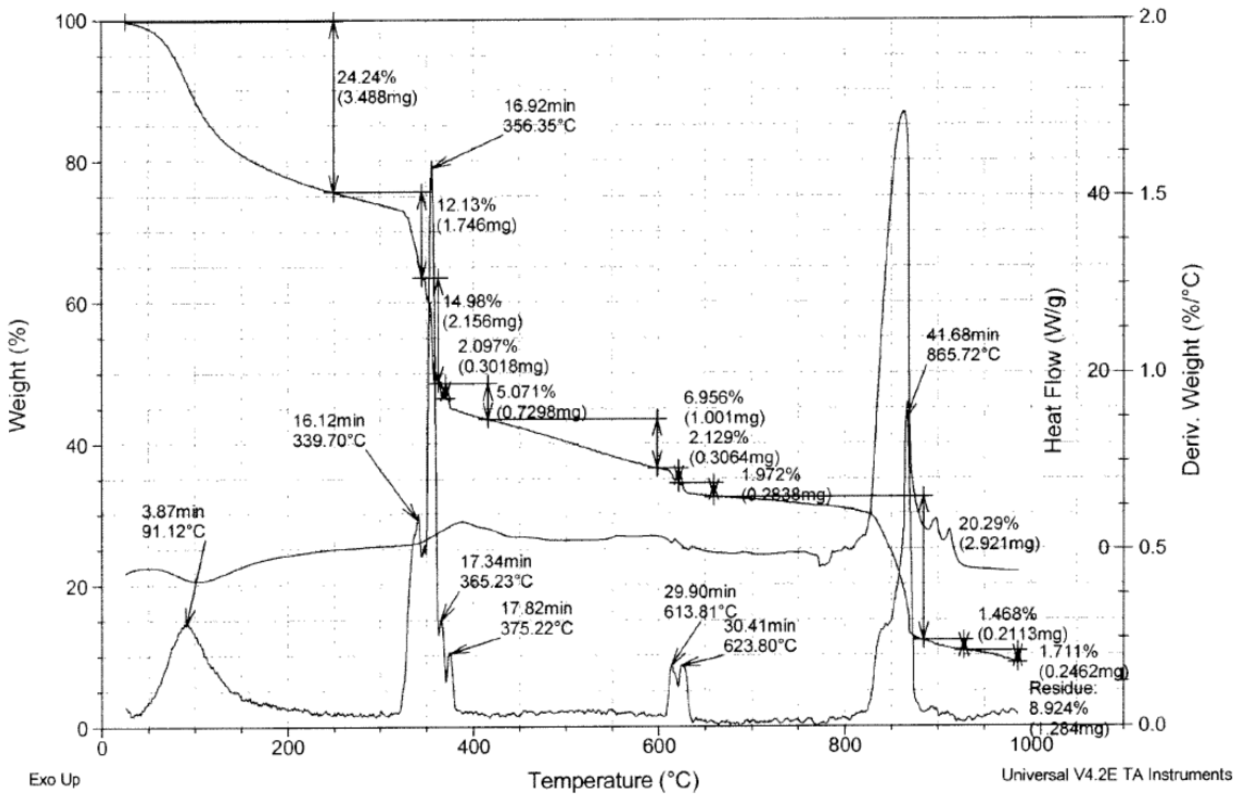

Figure 4.5. Overlay of Weight Change, Its Derivative, and Heat Flow

FTIR analysis utilized a Gram-Schmidt reconstruction to plot the change in infrared intensity as a function of time is shown in Figure 4.6. This was used to correlate infrared spectra with thermal events occurring during the temperature ramp on the TGA. The labeled peaks correspond closely with the derivative curve peaks shown in the TGA plots. Table 4.4 summarizes the FTIR analysis results produced by adding the data in a selected time range for the major peaks in Figure 4.6. The FTIR spectra collected from 2.95-3.89 min (84-103°C) show noisy H_2O peaks at the wavenumber range of 4000-3400 cm^{-1} and 2000-1300 cm^{-1}. All other spectra collected from the peaks approximately centered at 391, 575, 811, 863, and 931°C show CO_2 peaks at 3900-3500 cm^{-1}, 2400-2200 cm^{-1}, and 750-600 cm^{-1}. No peaks indicative of hydrocarbons or CO gas were observed.

For information purposes, the DSC-TGA was performed using a N_2 purge of 100 mL/min instead of air. Figure 4.7 shows the DSC-TGA results performed under N_2 purge and Figure 4.8 compares the TGA curves from tests under air and N_2. Under the N_2 purge the sample does not show the major weight loss peaks observed at 356 and 866°C but rather shows continuous weight loss from 150°C to the end of the test. A slight weight increase observed at 750°C from the test under N_2 is likely an experimental noise.

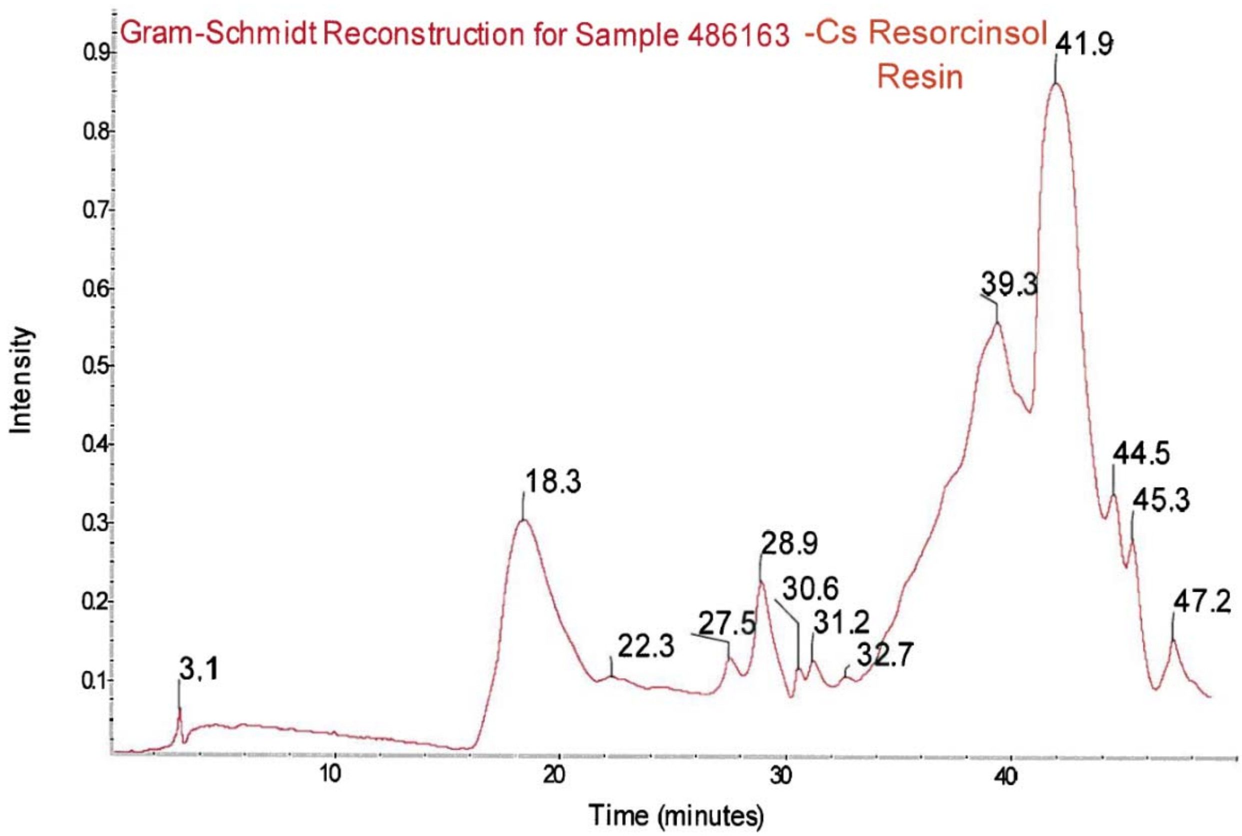

Figure 4.6. Gram-Schmidt Reconstruction for FTIR of the Cesium-Loaded SRF Resin

Table 4.4. Summary of FTIR Analysis Results on the Cesium-Loaded SRF Resin

Gram-Schmidt Reconstruction			
Peak time (min)	Time range (min)	Temperature range (°C)	FTIR Results
3.1	2.95 - 3.89	84 - 103	Peaks at 4000-3400 cm^{-1} and 2000-1300 cm^{-1}, representing H_2O
18.3	16.50 - 19.18	355 - 409	Peaks at 3900-3500 cm^{-1}, 2400-2200 cm^{-1}, and 750-600 cm^{-1}, representing CO_2
27.5	27.09 - 28.03	567 - 586	
39.3	37.02 - 40.10	765 - 827	
41.9	40.77 - 43.86	840 - 902	
45.3	44.13 - 46.14	908 - 948	

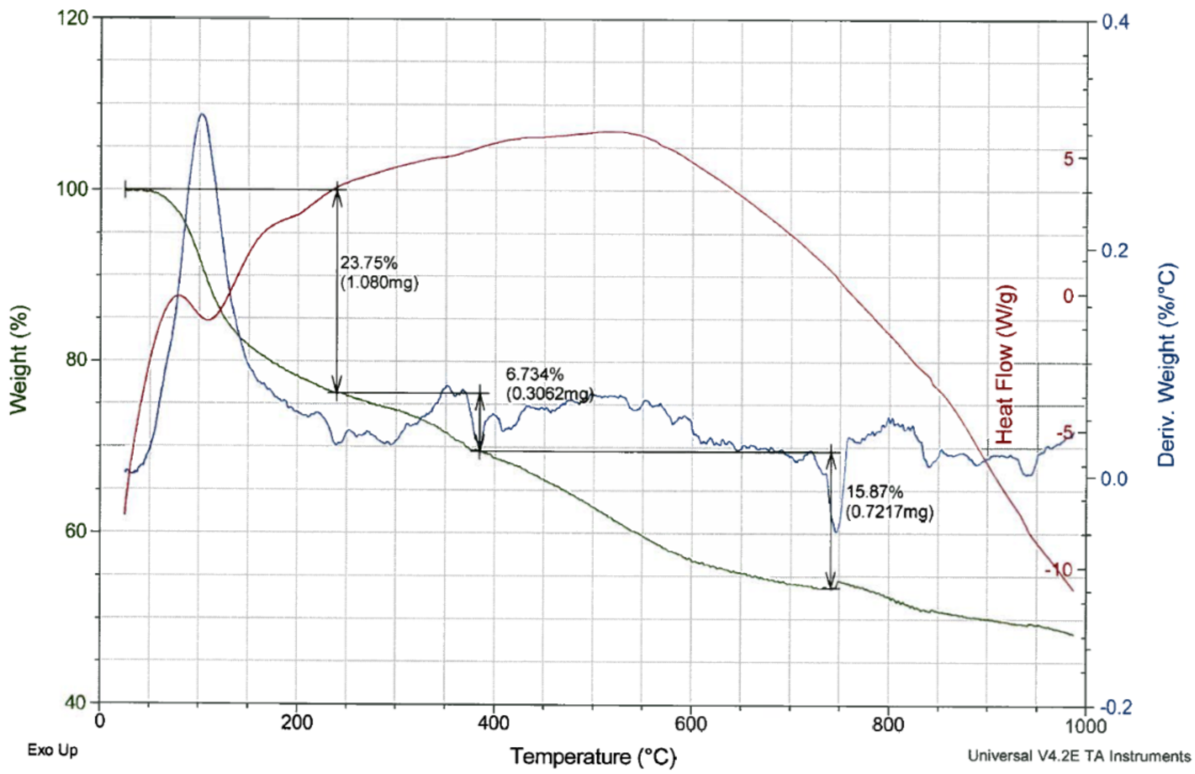

Figure 4.7. DSC-TGA in N_2 for the Cesium-Loaded SRF Resin

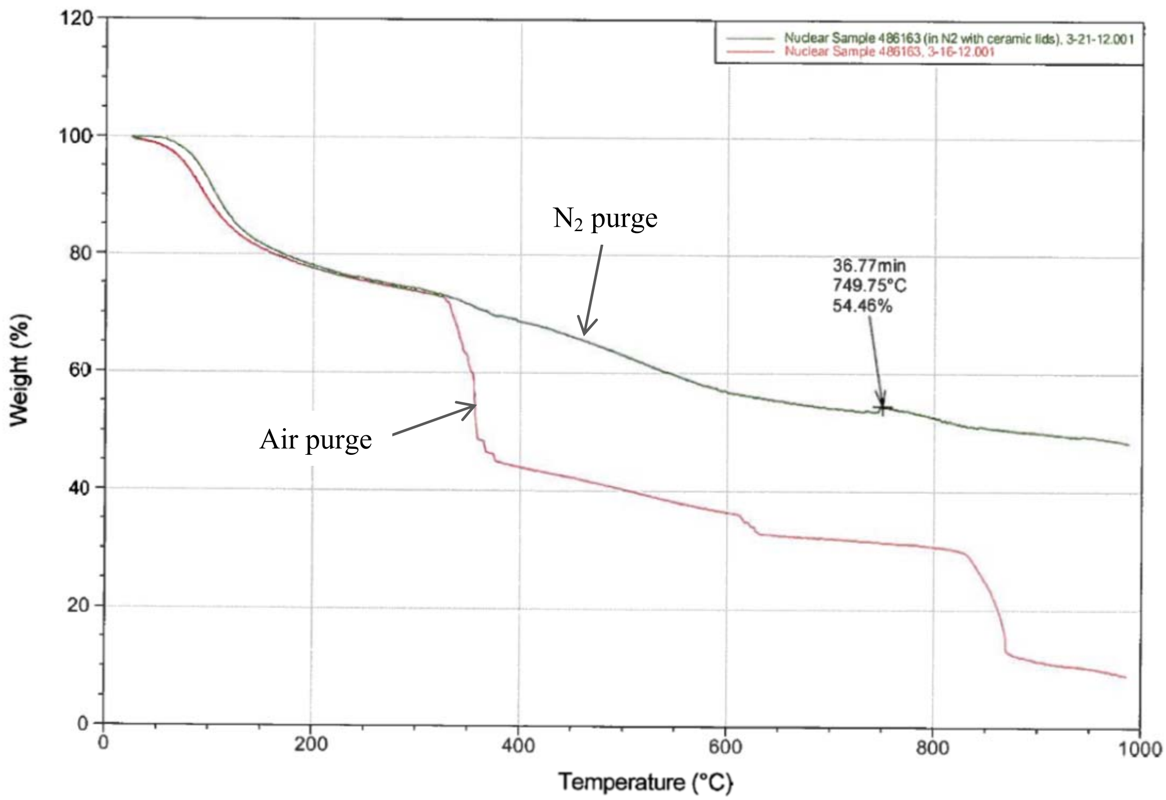

Figure 4.8. Comparison of DSC-TGA Curves in Air and N_2 for the Cesium-Loaded SRF Resin

5.0 Summary

The average FIT and SIT of the cesium-loaded SRF resin were 555°C and 610°C, respectively, which were 40°C (for FIT) and 3°C (for SIT) lower than the previous results for the SRF resin without cesium. The thermal conductivity (λ in W/[mK]) of the cesium-loaded SRF resin as a function of temperature (T in °C) between 22 and 90°C was expressed by a linear regression equation, $\lambda = 0.000273T + 0.0769$. The average specific heat capacity of the cesium-loaded SRF resin ranged from 1.434 J/(gK) at 40°C to 2.001 J/(gK) at 90°C. From the TGA-DSC analysis, an endothermic transition near 102°C appeared with a total weight loss of approximately 25%. Small exothermic transitions occurred around 387°C and 619°C and a large exothermic transition near 864°C with corresponding weight losses of approximately 30%, 5%, and 20%, respectively. The result of FTIR analyses showed that the weight loss peak at 102°C is linked to H_2O while all other peaks at higher temperature were linked to CO_2 gas.

6.0 References

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

ASTM D5334-08. 2008. *Standard Test Method for Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure*, ASTM International, West Conshohocken, PA.

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Not Publically Available

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24590-PTF-TEF-RT-11-00004, 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 Report

SOUTHWEST RESEARCH INSTITUTE®

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Chemistry and Chemical Engineering Division
Department of Analytical and Environmental Chemistry

May 22, 2012

Battelle Memorial Institute - PNNL
MSIN K6-24
790 6th Street
Richland, Washington 99352

Attn: Ms. Renee Russell

Subject:	Purchase Order No.:	167930
	SDG Number:	486163
	SwRI Project No.:	13295.12.00X
	SwRI Task Order Number:	120319-5
	SwRI Sample Receipt Number:	46112
	Samples Received:	11.11.11, 02.15.12
	Required Analysis:	Various/ See RFP 190709

Dear Ms Russell:

Please find the enclosed revised results for the sample and additional volume received on the above referenced date. Please note that this revision reflects updates to the Narrative and the FTIR Section. Should you have any questions, please do not hesitate to call me at 210-522-3242.

Sincerely,

Radonna Spies
Group Leader

APPROVED:

Michael J. Dammann
Director

RS: aa

Encl

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Chemistry and Chemical Engineering
Department of Analytical and Environmental Chemistry

April 23, 2012

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

Attention: Renee Russell

Subject:	Purchase Order No.:	167930
	SDG Number:	486163
	SwRI Project No.:	13295.12.00X
	SwRI Task Order No.:	120319-5
	SwRI Sample Receipt No.:	46112
	Samples Received:	11/11/11, 02/15/12
	Required Analysis:	Various / See RFP 190709

Dear Ms. Russell,

Please find the enclosed results for the sample and additional volume received on the above referenced dates. Should you have any questions, please feel free to contact me at (210) 522-3242, radonna.spies@swri.org.

Best Regards,

Radonna Spies
Group Leader

APPROVED:

Michael J. Dammann
Director

RS:dr

SOUTHWEST RESEARCH INSTITUTE

CLIENT: Battelle Memorial Ins. PNNL

TASK ORDER#: 120319-5

SRR#: 46112

SDG#: 486163

VTSR: 120319-5

PROJECT #: 13295.12.008

NARRATIVE

000002 rev 1
M 5.22.18

Client: Battelle Memorial PNNL
SDG: 486163
SwRI Project Number: 13295.12.008
SwRI Task Order Number: 120319-5

NARRATIVE

The spherical resorcinol formaldehyde resin was loaded with cesium according to the procedure described in Appendix B of the SOW. In addition to a thermal gravimetric off-gas analysis, the thermal conductivity and specific heat capacity were measured. The resin was dried at 95 °C prior to testing.

Thermal Conductivity

The thermal conductivity of the resin was measured using the transient heat method as described in ASTM D5334. The Hukseflux TP02 Non-Steady-State Probe (NSSP) for thermal conductivity measurement was used for the analysis. A Global Specialties 1305 Dual Output DC Power Supply (0-32V, 5A) was used to provide a constant current of 0.200 A to the probe when initiated. Measurement Computing's USB-TEMP with TracerDAQ® was utilized to acquire the time and temperature at 1 second intervals during the duration of the test. The material was place in to glass cylindrical vessel having a diameter of 55 mm and a length of 200 mm. The thermal conductivity probe was inserted into the material and temperature monitored to ensure thermal equilibrium had been reached prior to beginning the test. The program was setup to monitor the stabilized temperature for 1 minute prior to beginning the test. The power was turned on 1 minute into the test and turned off 4 minutes into the test. The time the power remained on (180 s) was the "heating phase". Once the power was turned off, the temperature was acquired for an additional 6 minutes; this was the 'cooling phase".

The NSSP principle relies on a unique property of a line source: after a short transient period the temperature rise, ΔT, only depends on heater power, Q, and medium thermal conductivity, λ:

$$\Delta T = (Q / 4 \pi \lambda)(\ln t) \qquad 0<t<t_1$$
$$\Delta T = (Q / 4 \pi \lambda)(\ln t/(t-t_1)) \qquad t>t_1$$

Where:
ΔT = temperature rise from time zero (K)
Q = heater input per unit length of heater (W/m)
t = time from the beginning of heating (s)
t_1 = heating time

Therefore, by measuring the heater power (Q) and recording temperature (T) versus time (t), the thermal conductivity (λ) can be calculated since the temperature rise varies linearly with the logarithm of time. The thermal conductivity is equivalent to the slope of the line representing the temperature vs ln t for the heating phase, and the ln(t/(t-t1)) for the cooling phase. A graph was constructed for each set of data (heating and cooling). The data graphed was evenly space with the logarithm of time (x-axis). The early and late portions of the test were not used in the slope analysis. For the heating phase, the slope was calculated from the ln(t) range of 2 to 5, which corresponds to 7 - 148 seconds. For the cooling phase, the slope was calculated for the ln(t/(t-t1)) range of 0.5 to 3, which corresponds to 189 - 457 seconds. The thermal conductivity for the heating and cooling phases was calculated using the equation below, and the final thermal conductivity reported was calculated using the average of the heating and cooling phase thermal conductivities.

$$\lambda = (CQ/4 \pi S)$$

Where:
$Q = I2\ (R/L) = (EI/L)$
Q = heat input (W/m)
C = calibration constant
λ = thermal conductivity [W/(m·K)]
S = slope used to compute thermal conductivity
t = time (s)
I = current flowing through heater wire (A)
R = total resistance of heater wire (ohm)
L = length of heated needle (m), and
E = measured voltage (V)

The thermal conductivity of the sample was measured in triplicate, at a minimum, and at three temperatures: 22 °C (ambient), 45 °C and 90 °C. The average thermal conductivity at each temperature was plotted and a linear best-fit trendline was established. Therefore, the thermal conductivity can be closely estimated using the following linear regression equation:

$$\text{Thermal Conductivity, } \lambda = (0.0003 \times T) + 0.0767$$

Where:
T = temperature in °K or °C

The thermal conductivity probe was calibrated before use with freshly prepared Agar water (2.5 g Agar / 500mL deionized water). The calculated thermal conductivity differed slightly from water's published thermal conductivity of 0.607 W/mK; therefore, the calibration factor, C, was adjusted to 0.91659.

Freshly purchased and opened glycerol was used for the calibration verifications. The thermal conductivity for the initial and final verifications calculated to 100% of published value for glycerol.

Specific Heat Capacity

Specific heat capacity, Cp, was measured by differential scanning calorimetry (DSC) in accordance with ASTM E1269 and the instrument manufacturer's instructions. The test consists of heating the sample at a controlled atmosphere through a temperature region of interest. The difference in heat flow into the sample compared to a reference material and a blank pan are used to calculate the specific heat capacity. Samples were analyzed using a Seiko DSC 210. The instrument was purged with 50 mL/min of nitrogen throughout the testing. For the DSC temperature program, the temperature was held isothermally at 25 °C for 4 minutes; the temperature was then ramped to 100 °C and held for 5 minutes. The thermal curve was collected for each analytical run. The thermal curve for the blank aluminum pan was collected to perform the blank subtraction. The DSC software contains a specific heat capacity program that performs the blank pan subtraction from samples and reference standards automatically. The blank subtracted sample and standard files were imported into the Cp calculation program. The program generated a specific heat capacity curve (Cp vs Temp) along with a table of specific heat capacities from 40-90 °C at 1 °C intervals.

Sapphire disk (PN# 0219 1483) from the Perkin Elmer Specific Heat Kit (PN #0219 0136) was used as the standard material. The specific heat capacity of the sapphire disk was verified against NIST SRM 720 with the following results:

Perkin Elmer Sapphire Disk (0219 1483)

Temp. °C	Published Cp J/g°K	Calculated Cp J/g°K	% Rec
46.85	0.8194	0.76	92.8%
56.85	0.8380	0.782	93.3%
66.85	0.8556	0.798	93.3%
76.85	0.8721	0.809	92.8%
86.85	0.8878	0.809	91.1%

The specific heat capacity of the sample was determined in duplicate. The RPDs of the heat capacities for the sample and duplicate at 40 °C and 90 °C were 1.88% and 3.55%, respectively.

DSC-TGA-FTIR

Thermal and infrared analysis on the Cs loaded resorcinol resin was accomplished using a TA Instruments Q600 SDT Simultaneous DSC-TGA Heat Flow Analyzer and a Nicolet Magna-IR 560 Fourier Transform Infrared Analyzer (FT-IR). The two instruments were connected with a Nicolet TGA Interface. This allowed the real time infrared analysis of the evolved gases from the thermal decomposition or volatilization of the sample material.

TGA analysis was performed on a 14.39 mg sample quantity placed in a ceramic alumina pan. The sample was heated from room temperature to 1000°C at a ramp rate of 20°C/min. An air purge gas flow rate of 60 ml/min was used to sweep the evolved gases through a heated transfer line (180°C) and then through the heated flow cell (225°C) of the TGA Interface. The Nicolet Magna-IR 560 monitored the composition of the gas in real time.

The thermal behavior of the sample was displayed with weight % and heat flow plotted as a function of temperature. From the TGA/DSC analysis (Figure 1) the heat flow curve shows an endothermic transition near 100 °C. Small exothermic transitions occur around 387 °C and 620° C. A large exotherm takes place near 864 °C. Each of these exotherms corresponded to weight losses of approximately 30%, 5% and 20%, respectively.

The weight loss curve with the derivative curve overlay is presented in Figure 2. The derivative curve aids in distinguishing some subtle transitions.

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M 5-22-12

Client: Battelle Memorial PNNL
SDG: 486163
SwRI Project Number: 13295.12.008
SwRI Task Order Number: 120319-5

Figure 3 is the weight loss curve with the derivative curve and heat flow curve overlaid. The first weight loss at around 91 °C may be due to moisture or volatiles. Major weight losses occur at approximately 350 °C and 850 °C. Other smaller disassociations appear around 340, 365, 375, 614, and 624 °C.

FTIR analysis utilized a Gram-Schmidt reconstruction to plot (Figure 4) the change in infrared intensity as a function of time. This was used to correlate infrared spectra with thermal events occurring during the temperature ramp on the TGA. The labeled peaks correspond closely with the derivative curve peaks shown in the TGA plots.

The linked spectrum (Figure 5) is the first spectrum collected near the start of the experiment. Subsequent "Coadded Spectrums" are single spectrums produced by coadding data in a selected time region of the Gram-Schmidt reconstruction profile. Coadded spectrums were chosen to relate to the TGA thermal events and time frames.

As the sample approached 100 °C, the FTIR spectra collected from 2.95-3.89 min (Figure 6) shows noisy moisture peaks appearing to develop in the 3500-4000 cm-1 and the 1300-2000 cm-1 ranges. In Figure 7, the FTIR acquisition during the first major weight loss at 350 °C (16.50-19.18 min) shows peaks at 2350 cm-1 and 670 cm which are attributed to carbon dioxide (CO_2). The following co-added spectrum at 27-09-28.03 min (Figure 8) contained the same CO_2 peaks but at lower intensity. Figures 9 and 10 are spectra collected during the second major weight loss event at 850 °C (37.02-40.10 min and 40.77-43.86 min, respectively). In addition to the large peaks CO_2 peak at 2350 cm-1, numerous peaks in the 3500-4000 cm-1 and 620-720 cm-1 ranges are present and are indicative of CO_2. During the analysis, no peaks indicitive of hydrocarbons (2800-3200 cm-1) were observed.

TGA analysis in Nitrogen

For informational purposes, the thermogravimetric analysis was repeated using a nitrogen purge of 100 mL/min instead of air and covering the sample containers.

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the laboratory manager or his/her designee, as verified by the following signature. This report shall not be reproduced except in full without the written approval of SwRI."

Group Leader ____________________ **Date** 05/22/12

SOUTHWEST RESEARCH INSTITUTE
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

SAMPLE RECEIPT, TASK ORDER & CHAIN OF CUSTODY

Southwest Research Institute

Sample Receipt

Sample Receipt Number: 46112

Revision: 3

VTSR: 11/11/11

Time: 08:30:00

Project: 13295.12.008
Proposal #: 01-63774
Client: Battelle Memorial Institute

This Receipt was Revised Apr 23 2012 4:05PM

Manager: HORTON, GENE
Logged in by: KBill
Creation Date: 11/17/11

Notes

Container 1: Test Sample
Container 2: Extra

Project is Nuclear Safety Related, 10 CFR 50, Part 21, and Appendix B. For Div 01 contact Joann Boyd @ X2169 or M. Valenti @ X3682. Have SPQP (Latest Revision) available at the time of prep or analysis of samples. All personnel must be QA Nuclear Certified.

POC: Eugene Horton X3457.

Disposal Instructions: Contact Eugene Horton for sample disposal authorization.

*** KBill Nov 17 2011 1:58PM ***

Revised to add additional sample SWRI delivered at 10:50AM on 02/15/2012.
*** KBill Feb 15 2012 1:51PM ***

System ID	Customer ID	CED	Matrix	Containers	Special Reqs.
486163	(2/15) CS Loaded Resorcinol Resin			2	
479505	Resorcinol-Formaldehyde Polymer			2	
Containers: 4				**Samples: 2**	

These documents are associated with this receipt: 109148[RFP #190709], 109444[Tech Prop], 109445[Cost Prop], 109773[Contract 167930], 109898[COC for SRR 46069], 109900[MSDS for SRR 46069], 110146[Sample Pic], 113577[(2/15) Sample Pic], 115168[Form 170]

Thermometer:
Temperature:

Client: Battelle Memo... SR#: 46112

Page Printed: 23 Apr 2012 16:06:43
ver (9/12/2011)

FRM-002

Southwest Research Institute

Laboratory Task Order

TO #: 120319-5 Revision: 2

SDG: 486163
VTSR: 11/11/11, 02/15/12
CASE: 167930

SRR #'s: 46112
Client(s): Battelle Memorial Institute

Project(s): 13295.12.008
Manager(s): HORTON, GENE
To PM: 03/08/12
To QA: 03/09/12
To Client: 03/09/12

Instructions

• MEASURE THE SODIUM-FORM RF RESIN BED CONDUCTIVITY AND CAPACITY.
HEAT CAPACITY VIA DSC ASTM E1269
THERMAL CONDUCTIVITY VIA ASTM D5334/D5930
• PERFORM A TGA (THERMAL GRAVIMETRIC ANALYSIS) WITH MASS SPECTROMETRY OF THE RESIN OFFGAS UP TO THE AUTO IGNITION TEMPERATURE AND DETERMINE CONSTITUENTS.
THIS WILL BE ACHIEVED IN SEVERAL STEPS SINCE SWRI DOES NOT HAVE A TGA-MS SETUP. WE DO HAVE A TGA-FTIR SETUP. THE SAMPLE WILL FIRST BE RUN VIA TG/DTA-FTIR (THERMAL GRAVIMETRIC / DIFFERENTIAL THERMAL ANALYSIS- FOURIER TRANSFORM INFRARED SPECTROMETRY) THIS TECHNIQUE WILL ALLOW US TO DETERMINE THE MASS LOSS VS. TEMPERATURE DATA WHILE MEASURING ANY THERMAL TRANSITIONS, AND THE FTIR WILL ALLOW IDENTIFICATION OF THE GASSES RELEASED DURING COMBUSTION. THESE TESTS WILL BE DONE USING AIR AS THE ATMOSPHERIC GAS. AFTER REVIEWING THE DATA, IF SUFFICIENT EVIDENCE IS OBTAINED THAT A MASS SPECTROMETRY ANALYSIS OF THE OFF GAS WOULD BE BENEFICIAL, A SECOND RUN WILL BE PERFORMED AND GRAB GAS SAMPLES, TRAPPED VIA SUMMA CANISTER AT THE APPROPRIATE INFLECTION POINT(S) OF THE TGA CURVE WILL BE TAKEN. THESE SAMPLES WILL BE RUN VIA GC AND/OR GC-MS FOR EXPECTED SPECIES.
REVISION 1, DRMZ 04/20/12: TASK ORDER REVISED TO ADD ASTM D1929, IGNITION TEST, ALONG WITH A NOTE INDICATING ORIGINAL SAMPLE RECEIVED ON 11/11/11 AND ADDITIONAL VOLUME ON 02/15/12.

REVISION 2, DRMZ 04/23/12: TASK ORDER REVISED TO INDICATE THE INITIAL RESORCINOL-FORMALDEHYDE POLYMER SAMPLE (LAB SYSTEM ID 479235) WAS RECEIVED AND LISTED IN SAMPLE RECEIPT REPORT # 46069. WHEN THE SAMPLE WAS TRANSFERRED TO FIRE TECH, THAT SECTION CREATED SRR 46112 TO NOTATE IT/TRACK IT UNDER THEIR SYSTEM AND ALSO TO LIST THE ADDITIONAL SAMPLE RECEIVED ON 02/15/12.

Documents Related to this task order: 109148[RFP #190709], 109444[Tech Prop], 109445[Cost Prop], 109773[Contract 167930], 109898[COC for SRR 46069], 109900[MSDS for SRR 46069], 110146[Sample Pic], 113577[(2/15) Sample Pic], 115168[Form 170]

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

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
486163		1		(2/15) CS Loaded Resorcinol Resin	11 Nov 11	11 Dec 11

Test: DSC_SwRI Holding: 180 days from CED
Section: WETCHEM **Differential Scanning Calorimetry** Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	CED	Method Date
486163		1		(2/15) CS Loaded Resorcinol Resin		

Test: WET-MISC Holding: 60 days from CED
Section: WETCHEM **Any miscellaneous wetchem test.** Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	CED	Method Date
486163		1		(2/15) CS Loaded Resorcinol Resin		

Test: WET-SOLID Holding: 60 days from CED
Section: WETCHEM **Any miscellaneous wetchem solid test.** Cnt: 1

System ID	Type	Cont	Matrix	Customer ID	CED	Method Date
486163		1		(2/15) CS Loaded Resorcinol Resin		

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
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

ASTM D 1929 – 96 (Re-approved 2011), Standard Test Method for Determining Ignition Temperature of Plastics

Total Page Count:
Fraction: ASTM Pages: 010001–010010
AA 4.23.12

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®

6220 CULEBRA RD. 78238-5166 • P.O. DRAWER 28510 78228-0510 • SAN ANTONIO, TEXAS, USA • (210) 684-5111 • WWW.SWRI.ORG
EMISTRY AND CHEMICAL ENGINEERING DIVISION
FIRE TECHNOLOGY DEPARTMENT
WWW.FIRE.SWRI.ORG
FAX (210) 522-3377

ASTM D 1929 – 96 (Re-approved 2011), *STANDARD TEST METHOD FOR DETERMINING IGNITION TEMPERATURE OF PLASTICS*

MATERIAL ID: *RESORCINOL FORMALDEHYDE RESIN*

FINAL REPORT
Consisting of 10 Pages

SwRI® Project No. 01.13295.12.008g
Test Date: February 17, 20-24, 2012
Report Date: April 23, 2012

Prepared for:

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

Submitted by:

Eugene Horton
Senior Engineering Technologist
Material Flammability Section

Approved by:

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

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

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.

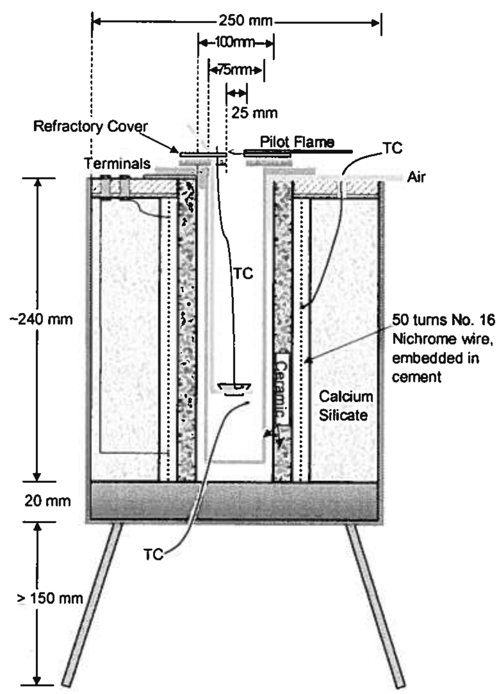


Figure 1. Schematic of Southwest Research Institute (SwRI) Hot-Air Furnace.

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 Resin* for testing in accordance with ASTM D 1929. On November 11, 2011, SwRI received approximately 20 liters of the specimen. further described in Table 1. On February 15, 2012, the Material Flammability Section received 1 bottle of Cesium loaded *Resorcinol Formaldehyde Resin* processed by our chemistry department, per instructions from the client.

TABLE 1. SAMPLE DESCRIPTION PROVIDED BY BATTELLE MEMORIAL INSTITUTE.

Material ID	Description*	Color*	Received Mass*	Nominal Tested Mass*
Resorcinol Formaldehyde Resin	Micro beads loaded with Cesium	Red	563.84 g	3 g

* Measured by SwRI personnel.

Upon receipt, the samples were placed in a controlled environment maintained at 23 °C ± 2 °C (73 °F ± 5 °F) and 50 % ± 5% relative humidity for not less than 40 h prior to testing. SwRI personnel weighed the material to a specimen weight of ~3 g on the day of testing

3.0 RESULTS

Testing was conducted February 17, 20-24, 2012. 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. Two known material standards were tested to confirm equipment operation against known parameters. These results are also reported in Table 2. 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 Resin with Cesium	610 °C	550 °C
	1130 °F	1022 °F
PMMA	480 °C	390 °C
	896 °F	734 °F
Marinite	Did Not Burn	Did Not Burn

SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	February 15, 2012
Test Date(s):	February 24, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	Resorcinol- Formaldehyde Polymer (Cesium loaded)	*Color:*	Red
Description:*	Small beads	*Average Sample Mass:*	3.00 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 610

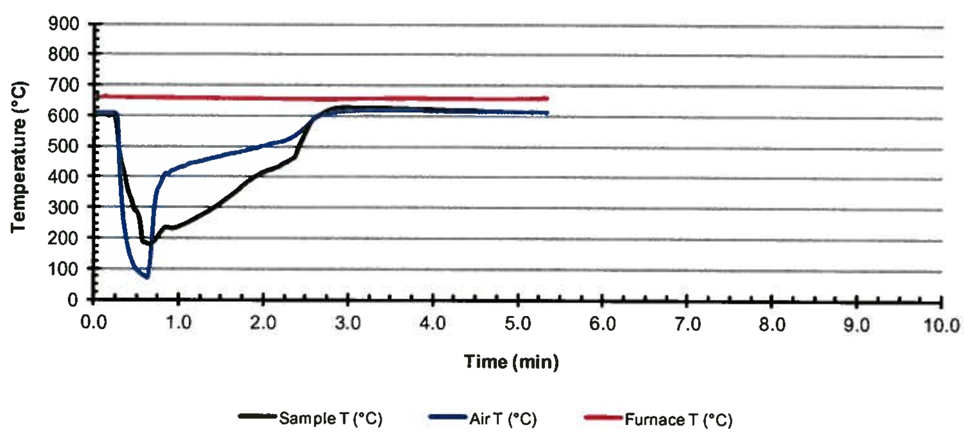
RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
12-055BAT008N1S	3.00	0.87	2.13	598	600	650	598	598	648	No
12-055BAT008N2S	3.00	0.99	2.01	604	610	661	613	614	659	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)
12-055BAT008N1S	:38	2:14	NA	No	Yes	No	No	10:38
12-055BAT008N2S	0:41	1:49	2:01	No	Yes	No	No	5:20

Resorcinol-Formaldehyde Polymer - Cesium Loaded



Note: Containment screen placed over the specimen holder

SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	A. Lowry	*Receipt Date:*	February 15, 2012
Test Date(s):	February 24, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	Resorcinol- Formaldehyde Polymer (Cesium loaded)	*Color:*	Red
Description:*	Small beads	*Average Sample Mass:*	3.00 g

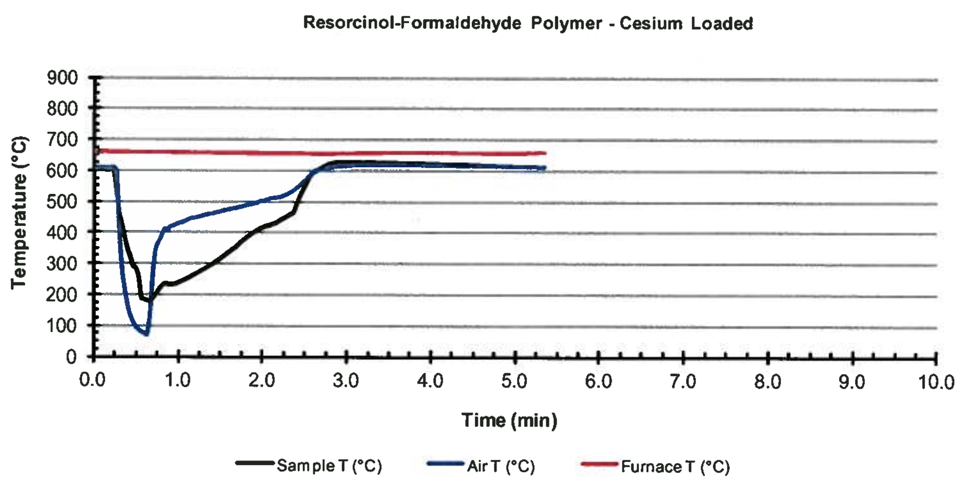
FLASH IGNITION TEMPERATURE (°C) : 550

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
12-055BAT008K8F	3.00	1.43	1.57	552	550	601	579	564	600	Yes
12-054BAT008K5F	3.00	1.41	1.59	540	540	591	552	548	590	No
12-054BAT008K6F	3.00	1.24	1.76	590	590	641	599	599	639	No
12-055BAT008K7F	3.00	1.40	1.60	574	570	620	594	583	616	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)
12-055BAT008K8F	0:43	NO	Flaming at 2:35	NO	Yes	NO	NO	5:00
12-054BAT008K5F	0:57	NO	NO	NO	Yes	NO	NO	10:58
12-054BAT008K6F	0:45	1:47	Flaming Flash	NO	Yes	NO	NO	10:46
12-055BAT008K7F	0:46	NO	Flaming Flash	NO	Yes	NO	NO	5:00



Note: Containment screen placed over the specimen holder

SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	February 15, 2012
Test Date(s):	February 24, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	Resorcinol- Formaldehyde Polymer (Cesium loaded)	*Color:*	Red
Description:*	Small beads	*Average Sample Mass:*	3.00 g

SPONTANEOUS IGNITION TEMPERATURE (°C) : 610

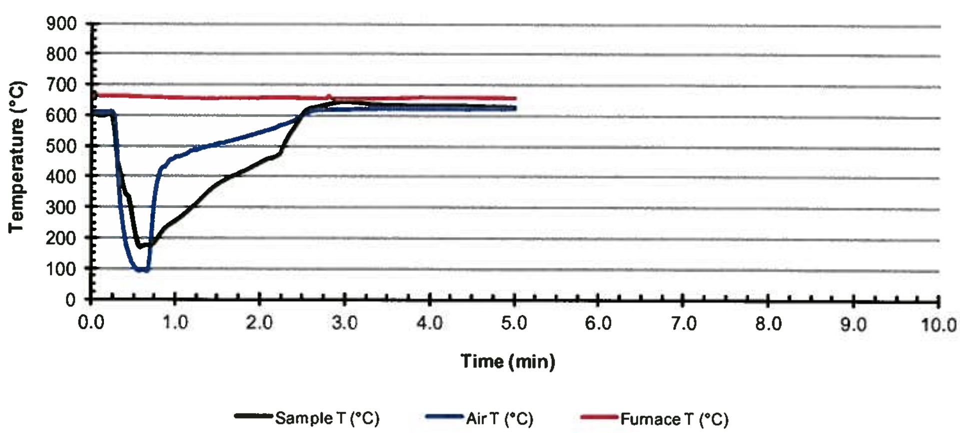
RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
12-055BAT008L2S	3.00	1.41	1.59	605	610	661	628	623	659	Yes
12-055BAT008L3S	3.00	0.96	2.04	597	600	650	599	602	651	NO
12-055BAT008L1S	3.00	1.32	1.68	619	620	672	637	632	671	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)
12-055BAT008L2S	:44	1:28	1:48	NO	Yes	NO	NO	5:00
12-055BAT008L3S	0:47	1:58	NA	NO	Yes	NO	NO	10:48
12-055BAT008L1S	:43	1:35	1:43	NO	Yes	NO	NO	5:00

Resorcinol-Formaldehyde Polymer



NOTE: Duplicate Test Run
Screen placed over sample cup

SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	A. Lowry	*Receipt Date:*	February 15, 2012
Test Date(s):	February 24, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	Resorcinol- Formaldehyde Polymer (Cesium loaded)	*Color:*	Red
Description:*	Small beads	*Average Sample Mass:*	3.00 g

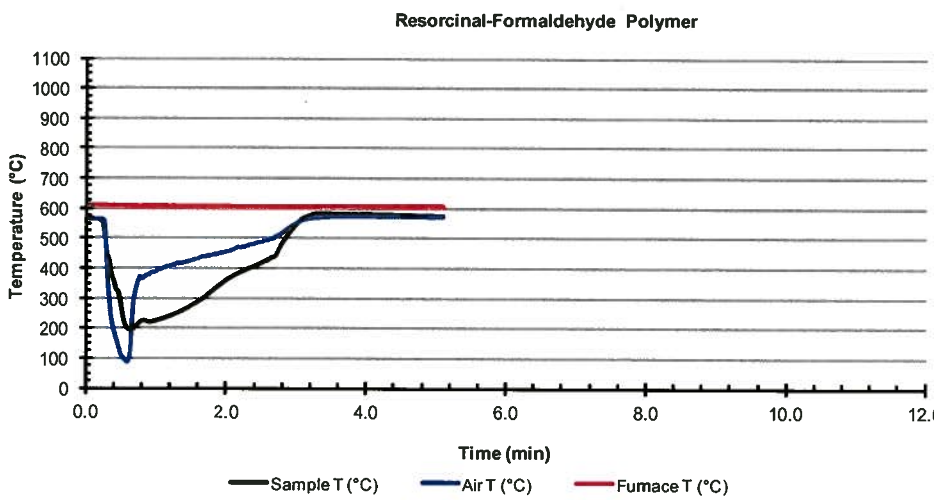
FLASH IGNITION TEMPERATURE (°C) : 560

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
12-055BAT008M3F	3.00	0.95	2.05	569	560	611	575	570	607	Yes
12-055BAT008M2F	3.00	0.92	2.08	554	550	600	549	550	600	NO
12-055BAT008M1F	3.00	1.00	2.00	540	540	590	540	540	588	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 (min:s)
12-055BAT008M3F	0:40	NO	Flaming at 2:20	NO	Yes	NO	NO	5:06
12-055BAT008M2F	0:38	NO	NA	NO	Yes	NO	NO	10:38
12-055BAT008M1F	:44	NO	NA	NO	Yes	NO	NO	10:44



NOTE: Duplicate Test Run
Screen placed over sample cup

SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	February 15, 2012
Test Date(s):	February 17, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	PMMA	*Color:*	Black
Description:*	Solid Block	*Average Sample Mass:*	3.07 g
		Average Sample Mass:	3.07 g

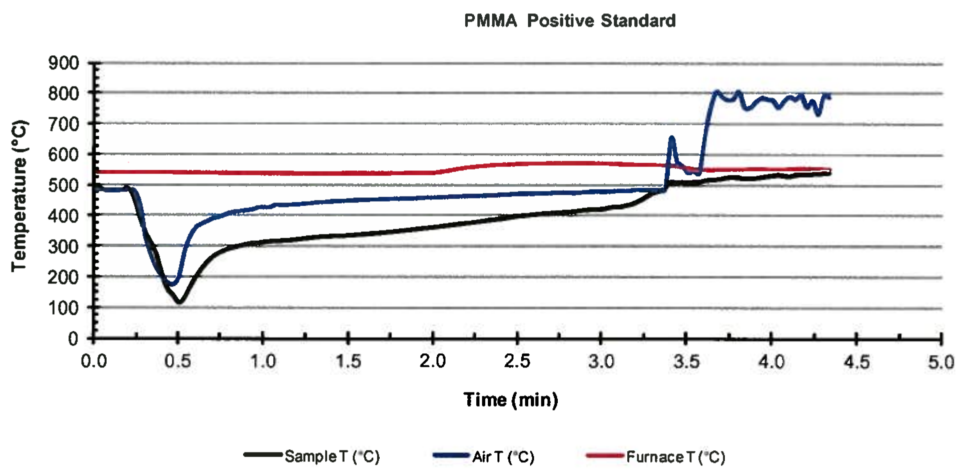
SPONTANEOUS IGNITION TEMPERATURE (°C) : 480

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
12-048BAT008G1S	3.15	0.64	2.51	414	410	466	415	408	465	No
12-048BAT008G2S	3.19	0.49	2.70	419	420	476	427	421	480	No
12-048BAT008G3S	2.94	0.07	2.87	430	430	486	447	436	486	No
12-048BAT008G4S	2.95	0.01	2.94	442	440	496	448	449	499	No
12-048BAT008G5S	3.00	0.72	2.28	489	480	536	540	784	551	Yes
12-048BAT008G6S	3.19	0.00	3.19	474	470	525	471	477	529	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)
12-048BAT008G1S	0:35	NO	NA	NO	NO	Yes	Yes	10:36
12-048BAT008G2S	0:42	NO	NA	NO	NO	Yes	Yes	10:42
12-048BAT008G3S	0:34	NO	NA	NO	NO	Yes	Yes	10:34
12-048BAT008G4S	0:34	NO	NA	NO	NO	Yes	Yes	10:34
12-048BAT008G5S	0:33	NO	Flaming at 2:49	NO	NO	Yes	Yes	4:20
12-048BAT008G6S	0:33	NO	NA	NO	NO	Yes	Yes	10:34



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - FLASH IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Flash
Operator:	A. Lowry	*Receipt Date:*	February 15, 2012
Test Date(s):	February 20-22, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	PMMA	*Color:*	Black
Description:*	Solid Block	*Average Sample Mass:*	3.08 g

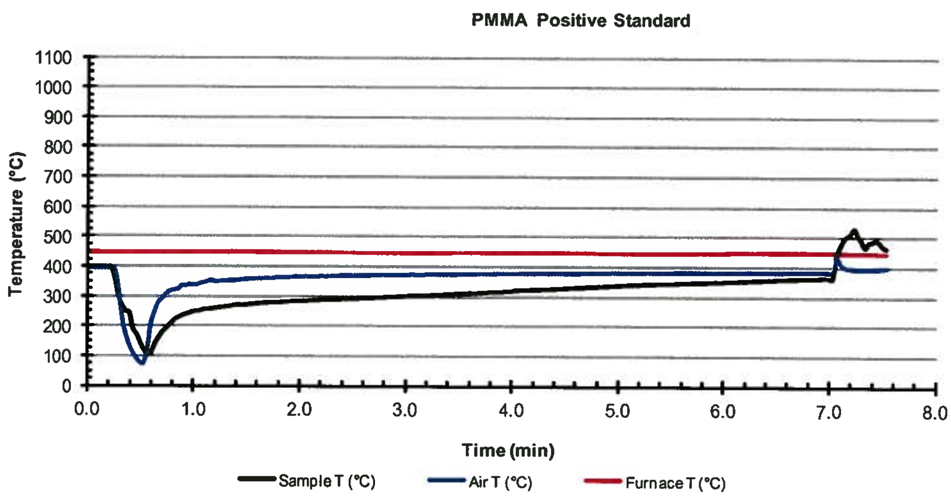
FLASH IGNITION TEMPERATURE (°C) : 390

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
12-051BAT008H1F	3.19	1.66	1.53	436	440	497	463	502	504	Yes
12-052BAT008H2F	3.19	2.22	0.97	435	430	490	534	427	489	Yes
12-052BAT008H3F	3.07	2.13	0.94	420	420	475	352	421	476	Yes
12-052BAT008H4F	3.19	2.25	0.94	414	410	463	386	411	463	Yes
12-052BAT008H5F	3.10	1.94	1.16	402	400	454	490	405	455	Yes
12-052BAT008H6F	2.98	1.72	1.26	395	390	450	463	395	447	Yes
12-052BAT008H7F	2.84	1.29	1.55	384	380	435	369	373	434	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)
12-051BAT008H1F	0:42	NO	Flaming at 2:10	NO	NO	Yes	Yes	4:20
12-052BAT008H2F	0:45	NO	Flaming at 3:01	NO	NO	Yes	Yes	4:24
12-052BAT008H3F	0:40	NO	Flaming at 3:22	NO	NO	Yes	Yes	4:40
12-052BAT008H4F	0:35	NO	Flaming at 3:59	NO	NO	Yes	Yes	5:00
12-052BAT008H5F	0:37	NO	Flaming at 5:15	NO	NO	Yes	Yes	6:20
12-052BAT008H6F	0:40	NO	Flaming at 6:22	NO	NO	Yes	Yes	7:32
12-052BAT008H7F	0:35	NO	NA	NO	NO	Yes	Yes	10:36



SOUTHWEST RESEARCH INSTITUTE

ASTM D 1929 TEST DATA SHEET - SPONTANEOUS IGNITION

Client:	Battelle Memorial Institute	*Ignition Type:*	Spontaneous
Operator:	A. Lowry	*Receipt Date:*	NA
Test Date(s):	February 22, 2012	*Date Prepared by SwRI:*	Prior to Testing
Project Number:	01.13295.12.008g		
Material ID:*	Marinite	*Color:*	White
Description:*	Solid block	*Original Thickness:*	20 mm
		Average Sample Mass:	3.17 g

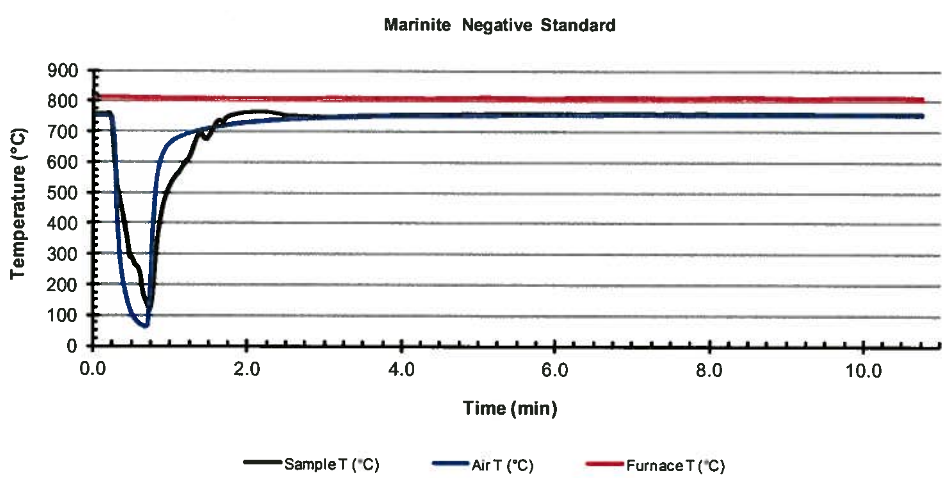
SPONTANEOUS IGNITION TEMPERATURE (°C) : NA

RESULTS

Test ID	Initial Mass (g)	Final Mass (g)	Mass Loss Loss (g)	Initial Temperature (°C) Sample	Air	Furnace	Final Temperature (°C) Sample	Air	Furnace	Ignition
12-053BAT008I1S	3.17	2.65	0.52	760	750	813	759	755	811	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)
12-053BAT008I1S	0:46	NA	NA	None	None	None	None	10:46



SOUTHWEST RESEARCH INSTITUTE

CLIENT: Battelle Memorial Ins. PNNL

TASK ORDER#: 120319-5

SRR#: 46112

SDG#: 486163

VTSR: 120319-5

PROJECT #: 13295.12.008

WETCHEM ANALYSIS

Total Page Count: 020001 –

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SOUTHWEST RESEARCH INSTITUTE
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

RAW DATA

SOUTHWEST RESEARCH INSTITUTE
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

Thermal Conductivity Data

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

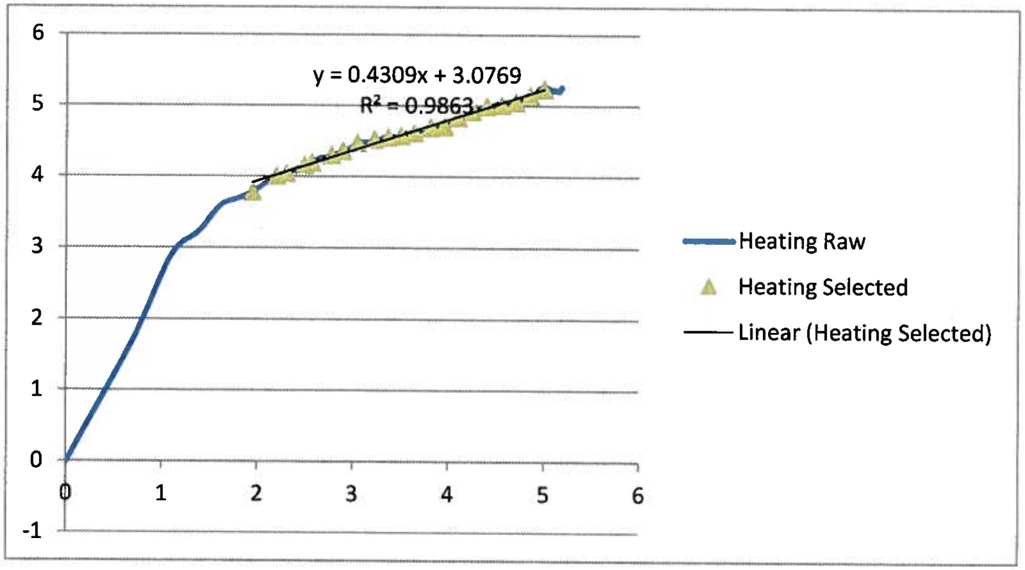
Sample ID: **Water Calibration 031612 (22-01-WCS6)**

	Heating	Cooling	Average
Slope:	0.4309	0.4315	0.4312
Therm Cond	**0.663**	**0.662**	**0.662**
TV	0.607	0.607	0.607
% Rec	109%	109%	109%

Current:	0.2	A
R:	89.73	ohms/m
Q:	3.5892	W/m
C:	1	Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time	ln(t)	Temp
2	7	1.9459101	3.7853
2.15	9	2.1972246	4.0095
2.3	10	2.3025851	4.0518
2.45	12	2.4849066	4.1648
2.6	13	2.5649494	4.2025
2.75	16	2.7725887	4.3091
2.9	18	2.8903718	4.3642
3.05	21	3.0445224	4.4772
3.2	25	3.2188758	4.5277
3.35	29	3.3672958	4.5644
3.5	33	3.4965076	4.5791
3.65	38	3.6375862	4.6204
3.8	45	3.8066625	4.6994
3.95	52	3.9512437	4.7123
4.1	60	4.0943446	4.8408
4.25	70	4.2484952	4.9308
4.4	81	4.3944492	4.9969
4.55	95	4.5538769	5.0125
4.7	110	4.7004804	5.052
4.85	128	4.8520303	5.153
5	148	4.9972123	5.242

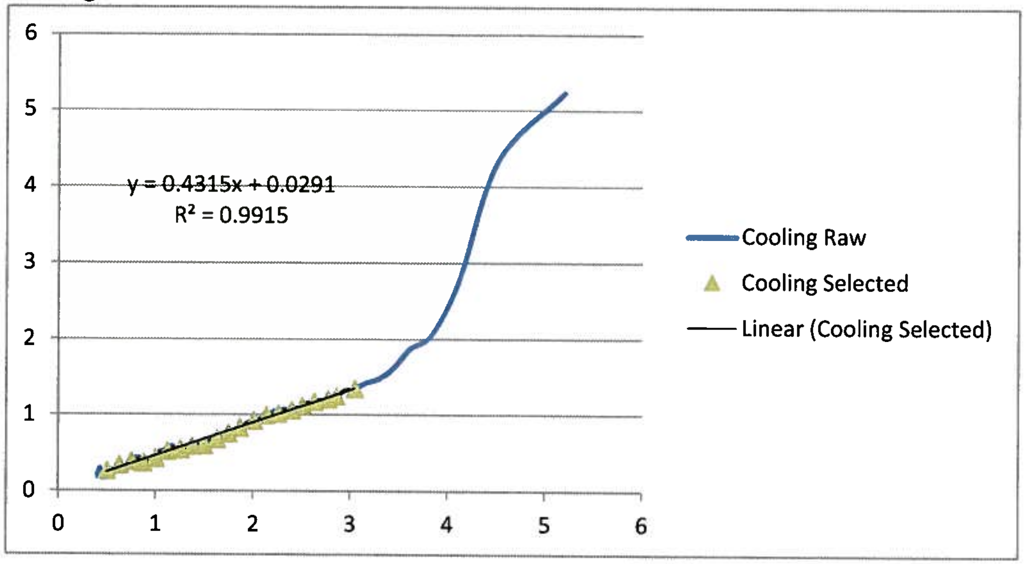
020004

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **Water Calibration 031612 (22-01-WCS6)**

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time	ln(t/(t-t1))	Temp
0.5	457	0.5006659	0.2712
0.625	387	0.6257059	0.3477
0.75	341	0.7504781	0.392
0.875	309	0.8735289	0.3726
1	285	0.9985288	0.4335
1.125	267	1.1213405	0.5275
1.25	252	1.252763	0.5506
1.375	241	1.3739231	0.5948
1.5	232	1.4954937	0.604
1.625	224	1.6274564	0.687
1.75	218	1.7469089	0.7617
1.875	213	1.8647846	0.8382
2	208	2.0053336	0.9285
2.125	204	2.1400662	0.9994
2.25	201	2.2587825	1.0206
2.375	198	2.3978953	1.0658
2.5	196	2.5055259	1.1211
2.625	194	2.6288008	1.18
2.75	192	2.7725887	1.2123
2.875	191	2.8543782	1.251
3	189	3.0445224	1.3486

020005

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **Glycerine ICV 031612**

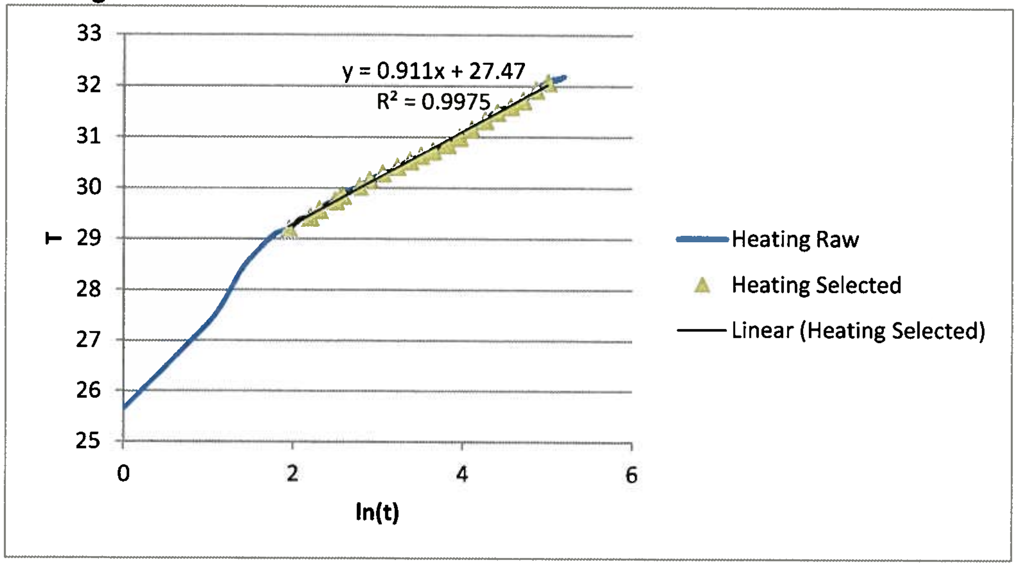
Actual Test Temperature: 22 °C

	Heating	Cooling	Average
Slope:	0.911	0.879	0.895
Therm Cond	0.287	0.298	0.293
TV	0.292	0.292	0.292
%Rec	98.4%	102%	100%

Current:	0.2	A
R:	89.73	ohms/m
Q:	3.5892	W/m
C:	0.91659	Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	29.1958
2.15	9	2.1972246	29.4337
2.3	10	2.3025851	29.5761
2.45	12	2.4849066	29.7488
2.6	13	2.5649494	29.8535
2.75	16	2.7725887	30.039
2.9	18	2.8903718	30.163
3.05	21	3.0445224	30.2943
3.2	25	3.2188758	30.4228
3.35	29	3.3672958	30.5403
3.5	33	3.4965076	30.644
3.65	38	3.6375862	30.7357
3.8	45	3.8066625	30.8605
3.95	52	3.9512437	31.0027
4.1	60	4.0943446	31.1651
4.25	70	4.2484952	31.3393
4.4	81	4.3944492	31.4934
4.55	95	4.5538769	31.6108
4.7	110	4.7004804	31.7235
4.85	128	4.8520303	31.9307
5	148	4.9972123	32.0865

020006

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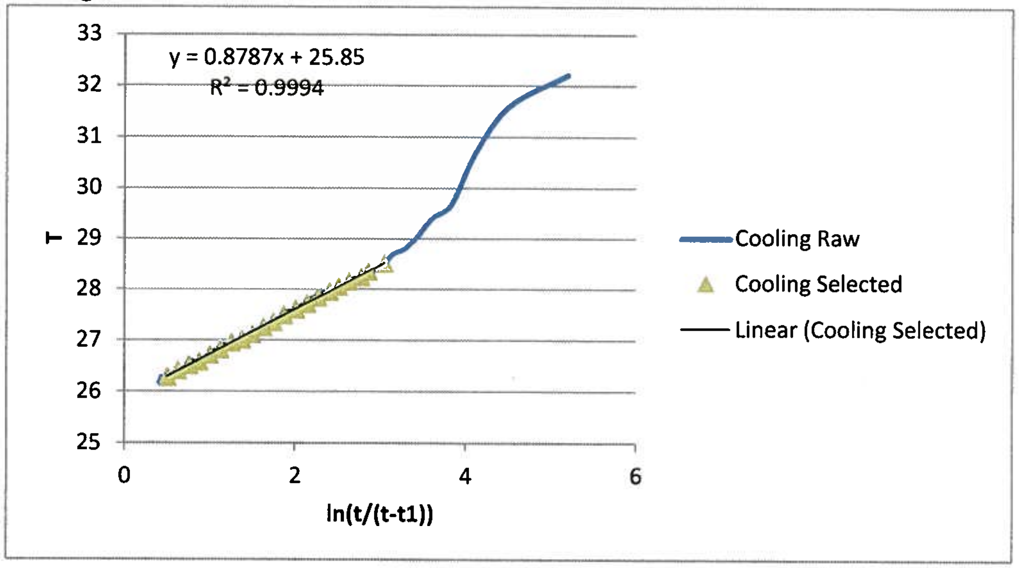
Thermal Conductivity - ASTM D5334/D5930

Sample ID: **Glycerine ICV 031612**

Actual Test Temperature: 22 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	26.2925
0.625	387	0.6257059	26.4234
0.75	341	0.7504781	26.5229
0.875	309	0.8735289	26.5938
1	285	0.9985288	26.7292
1.125	267	1.1213405	26.8342
1.25	252	1.252763	26.9696
1.375	241	1.3739231	27.0294
1.5	232	1.4954937	27.1307
1.625	224	1.6274564	27.2651
1.75	218	1.7469089	27.3691
1.875	213	1.8647846	27.5063
2	208	2.0053336	27.6167
2.125	204	2.1400662	27.7318
2.25	201	2.2587825	27.8523
2.375	198	2.3978953	27.9683
2.5	196	2.5055259	28.0639
2.625	194	2.6288008	28.166
2.75	192	2.7725887	28.2543
2.875	191	2.8543782	28.3638
3	189	3.0445224	28.5275

020007

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

SwRI ID: **486163_25** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 22 °C

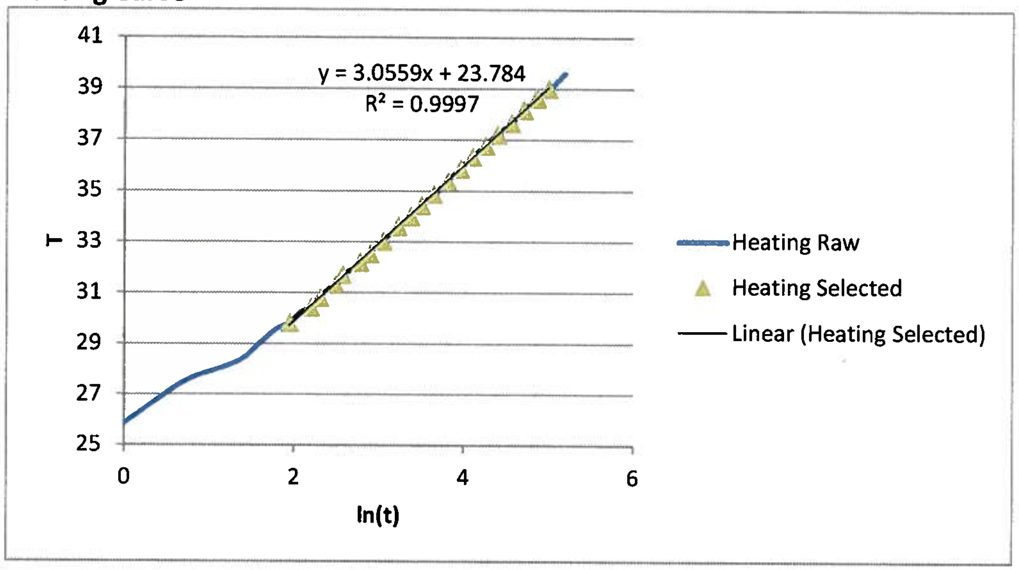
	Heating	Cooling	Average
Slope:	3.0559	3.0711	3.0635
Therm Cond	0.0857	0.0852	0.0855

Current:	0.2	A
R:	89.73	ohms/m
Q:	3.5892	W/m
C:	0.91659	Cal Factor

Heating	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.0855

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	29.8388
2.15	9	2.1972246	30.4641
2.3	10	2.3025851	30.8385
2.45	12	2.4849066	31.3283
2.6	13	2.5649494	31.718
2.75	16	2.7725887	32.2194
2.9	18	2.8903718	32.5446
3.05	21	3.0445224	33.0309
3.2	25	3.2188758	33.625
3.35	29	3.3672958	34.0184
3.5	33	3.4965076	34.4529
3.65	38	3.6375862	34.8826
3.8	45	3.8066625	35.3897
3.95	52	3.9512437	35.8958
4.1	60	4.0943446	36.3559
4.25	70	4.2484952	36.7903
4.4	81	4.3944492	37.23
4.55	95	4.5538769	37.6695
4.7	110	4.7004804	38.1873
4.85	128	4.8520303	38.6419
5	148	4.9972123	39.0217

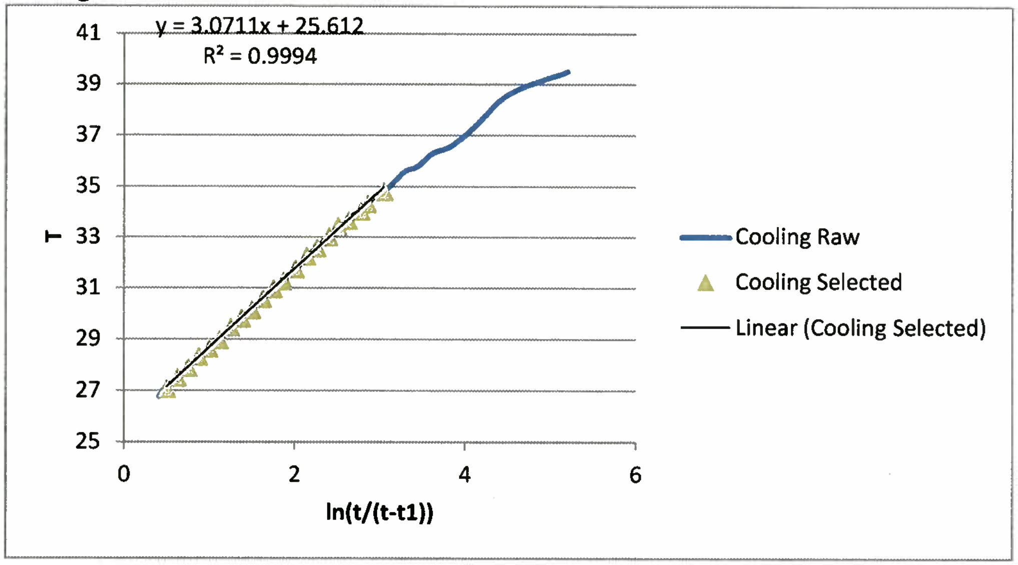
020008

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

SwRI ID: **486163 25** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 22 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	27.0976
0.625	387	0.6257059	27.5192
0.75	341	0.7504781	27.9057
0.875	309	0.8735289	28.3491
1	285	0.9985288	28.6599
1.125	267	1.1213405	29.0055
1.25	252	1.252763	29.4898
1.375	241	1.3739231	29.8425
1.5	232	1.4954937	30.1694
1.625	224	1.6274564	30.61
1.75	218	1.7469089	30.9908
1.875	213	1.8647846	31.3082
2	208	2.0053336	31.7703
2.125	204	2.1400662	32.2781
2.25	201	2.2587825	32.6079
2.375	198	2.3978953	33.0374
2.5	196	2.5055259	33.4457
2.625	194	2.6288008	33.6864
2.75	192	2.7725887	34.0541
2.875	191	2.8543782	34.3404
3	189	3.0445224	34.8287

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_25d**   Client ID: (2/15) Cs Loaded Resorcinol Resin   Actual Test Temperature: 22 °C

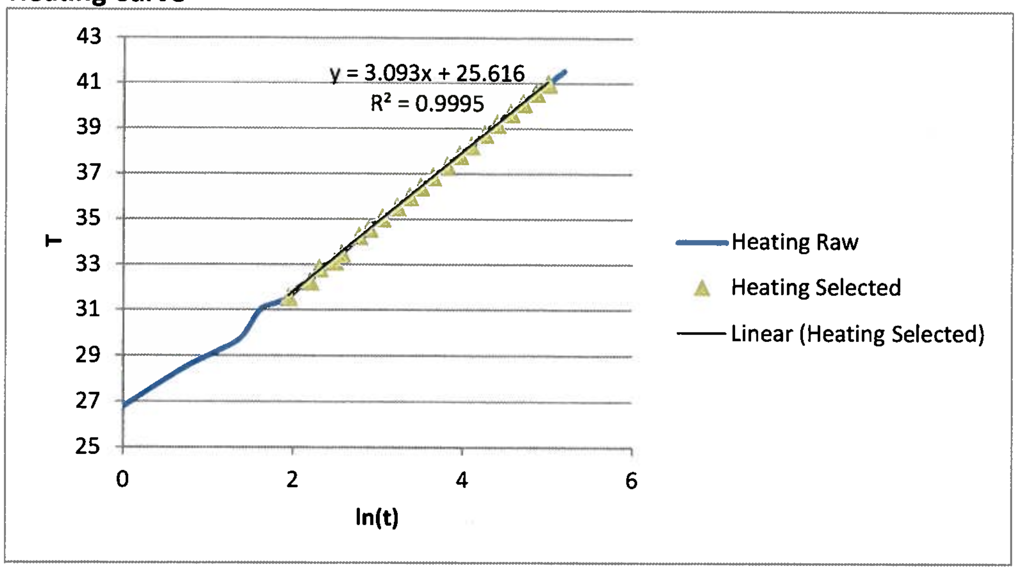
	Heating	Cooling	Average
Slope	3.093	3.327	3.210
Therm Cond	0.0846	0.0787	0.0816

Current:	0.2	A
R:	89.73	ohms/m
Q:	3.5892	W/m
C:	0.91659	Cal Factor

Heating	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.0816

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	31.5902
2.15	9	2.1972246	32.2832
2.3	10	2.3025851	32.874
2.45	12	2.4849066	33.1771
2.6	13	2.5649494	33.4883
2.75	16	2.7725887	34.3016
2.9	18	2.8903718	34.6171
3.05	21	3.0445224	35.0915
3.2	25	3.2188758	35.5794
3.35	29	3.3672958	36.026
3.5	33	3.4965076	36.4587
3.65	38	3.6375862	36.9058
3.8	45	3.8066625	37.3683
3.95	52	3.9512437	37.8634
4.1	60	4.0943446	38.2607
4.25	70	4.2484952	38.7864
4.4	81	4.3944492	39.2317
4.55	95	4.5538769	39.6923
4.7	110	4.7004804	40.1327
4.85	128	4.8520303	40.5975
5	148	4.9972123	41.0166

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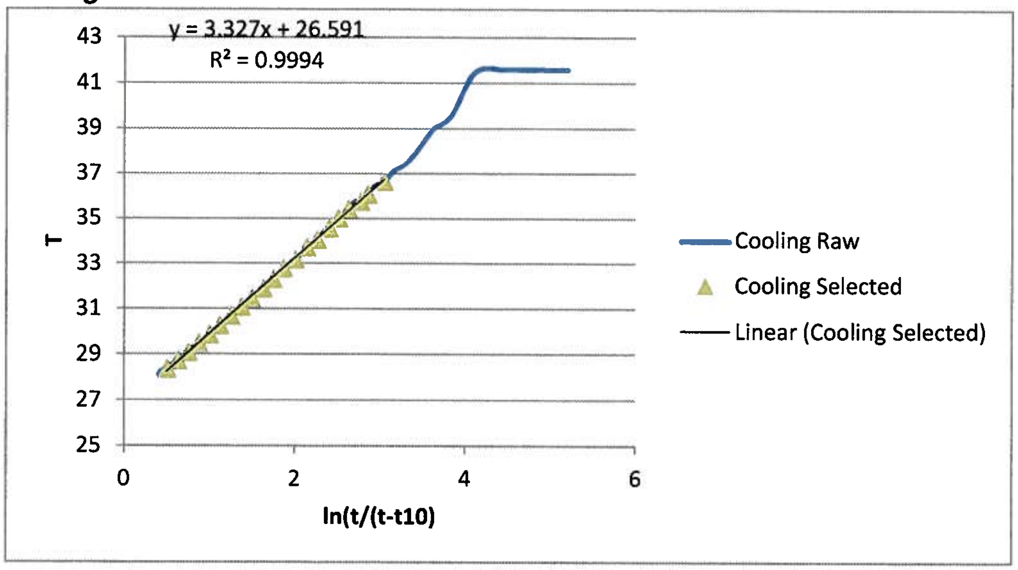
Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 25d** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 22 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	28.372
0.625	387	0.6257059	28.748
0.75	341	0.7504781	29.1027
0.875	309	0.8735289	29.5071
1	285	0.9985288	29.8799
1.125	267	1.1213405	30.2904
1.25	252	1.252763	30.7172
1.375	241	1.3739231	31.1098
1.5	232	1.4954937	31.4702
1.625	224	1.6274564	31.9139
1.75	218	1.7469089	32.3382
1.875	213	1.8647846	32.8009
2	208	2.0053336	33.2202
2.125	204	2.1400662	33.7612
2.25	201	2.2587825	34.1006
2.375	198	2.3978953	34.599
2.5	196	2.5055259	35.0505
2.625	194	2.6288008	35.4534
2.75	192	2.7725887	35.7768
2.875	191	2.8543782	36.0991
3	189	3.0445224	36.6705

020011

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Thermal Conductivity - ASTM D5334/D5930

Sample ID: 486163_25t   Client ID: (2/15) Cs Loaded Resorcinol Resin   Actual Test Temperature: 22 °C

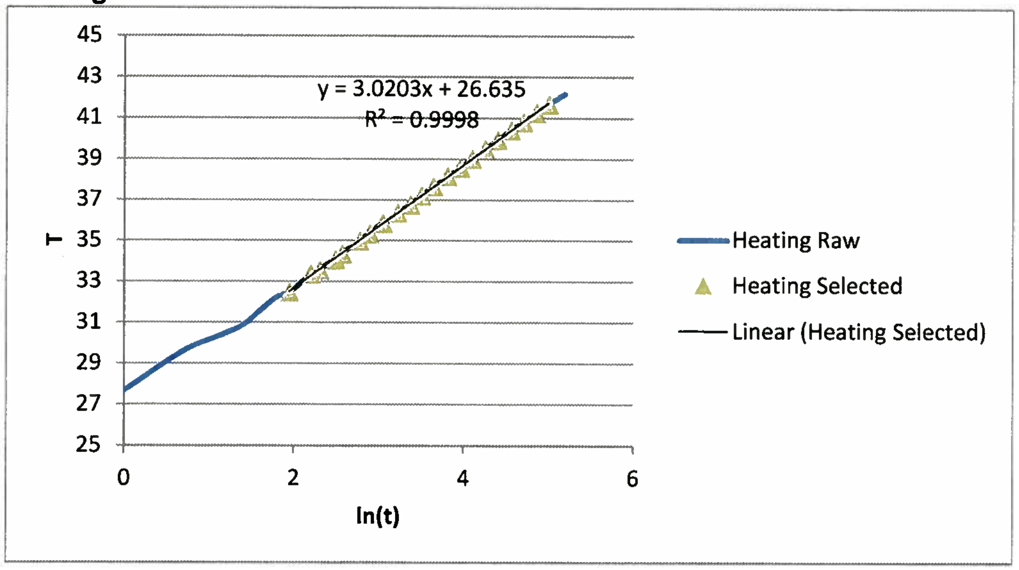
	Heating	Cooling	Average
Slope	3.0203	3.0655	3.0429
Therm Cond	0.0867	0.0854	0.0860

Current: 0.2 A
R: 89.73 ohms/m
Q: 3.5892 W/m
C: 0.91659 Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.0860

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	32.4917
2.15	9	2.1972246	33.379
2.3	10	2.3025851	33.5593
2.45	12	2.4849066	34.0736
2.6	13	2.5649494	34.3498
2.75	16	2.7725887	34.9769
2.9	18	2.8903718	35.3397
3.05	21	3.0445224	35.8577
3.2	25	3.2188758	36.3644
3.35	29	3.3672958	36.7714
3.5	33	3.4965076	37.2129
3.65	38	3.6375862	37.6671
3.8	45	3.8066625	38.1739
3.95	52	3.9512437	38.5902
4.1	60	4.0943446	39.0164
4.25	70	4.2484952	39.5026
4.4	81	4.3944492	39.9322
4.55	95	4.5538769	40.4071
4.7	110	4.7004804	40.8062
4.85	128	4.8520303	41.2544
5	148	4.9972123	41.6742

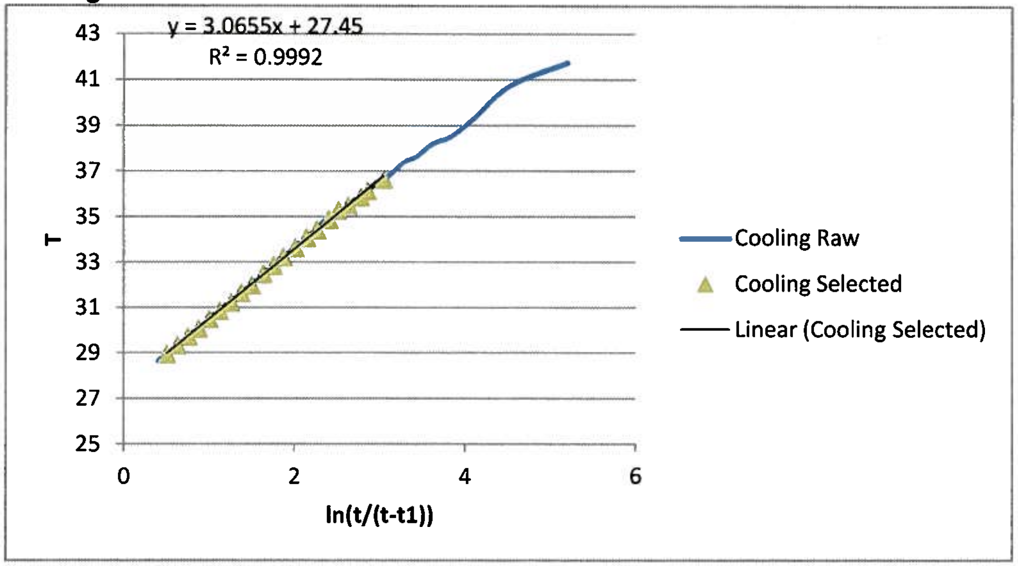
020012

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_25t** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 22 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	28.9609
0.625	387	0.6257059	29.3533
0.75	341	0.7504781	29.7345
0.875	309	0.8735289	30.0761
1	285	0.9985288	30.5048
1.125	267	1.1213405	30.86
1.25	252	1.252763	31.2388
1.375	241	1.3739231	31.6578
1.5	232	1.4954937	31.997
1.625	224	1.6274564	32.4882
1.75	218	1.7469089	32.8554
1.875	213	1.8647846	33.23
2	208	2.0053336	33.6492
2.125	204	2.1400662	34.0928
2.25	201	2.2587825	34.4267
2.375	198	2.3978953	34.8701
2.5	196	2.5055259	35.2731
2.625	194	2.6288008	35.485
2.75	192	2.7725887	35.8558
2.875	191	2.8543782	36.1389
3	189	3.0445224	36.6218

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 25g** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 22 °C

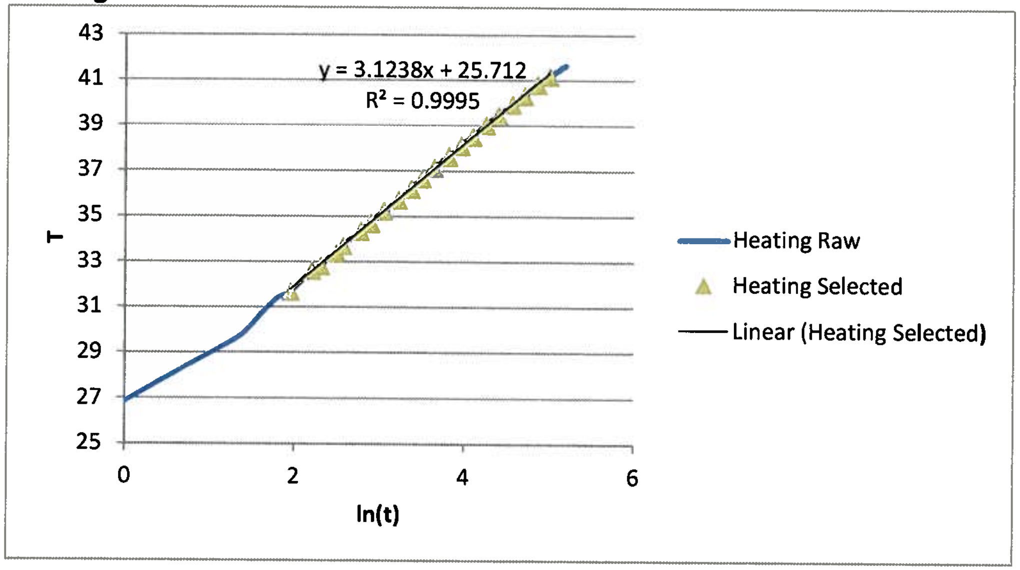
	Heating	Cooling	Average
Slope	3.1238	3.372	3.2479
Therm Cond	0.0838	0.0776	0.0806

Current:	0.2	A
R:	89.73	ohms/m
Q:	3.5892	W/m
C:	0.91659	Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.0806

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	31.6879
2.15	9	2.1972246	32.6217
2.3	10	2.3025851	32.8287
2.45	12	2.4849066	33.4028
2.6	13	2.5649494	33.7277
2.75	16	2.7725887	34.3835
2.9	18	2.8903718	34.7428
3.05	21	3.0445224	35.2592
3.2	25	3.2188758	35.7707
3.35	29	3.3672958	36.24
3.5	33	3.4965076	36.7137
3.65	38	3.6375862	37.1634
3.8	45	3.8066625	37.6567
3.95	52	3.9512437	38.1325
4.1	60	4.0943446	38.5379
4.25	70	4.2484952	39.0452
4.4	81	4.3944492	39.454
4.55	95	4.5538769	39.9499
4.7	110	4.7004804	40.3192
4.85	128	4.8520303	40.8076
5	148	4.9972123	41.1784

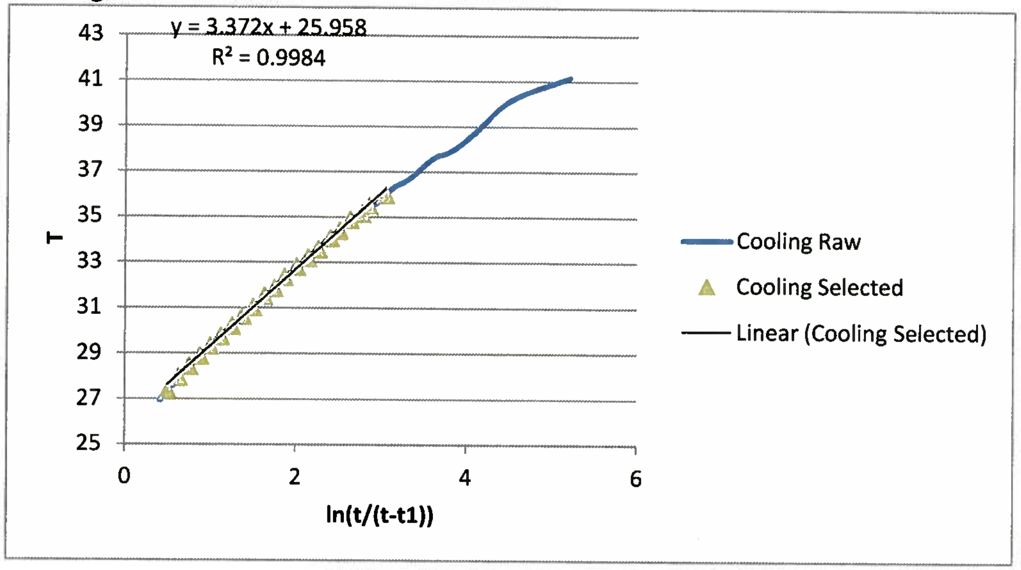
020014

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 25g** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 22 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	27.3702
0.625	387	0.6257059	27.9851
0.75	341	0.7504781	28.457
0.875	309	0.8735289	28.914
1	285	0.9985288	29.358
1.125	267	1.1213405	29.7769
1.25	252	1.252763	30.2342
1.375	241	1.3739231	30.6739
1.5	232	1.4954937	31.0427
1.625	224	1.6274564	31.5252
1.75	218	1.7469089	31.8827
1.875	213	1.8647846	32.3831
2	208	2.0053336	32.8273
2.125	204	2.1400662	33.2174
2.25	201	2.2587825	33.6036
2.375	198	2.3978953	34.0886
2.5	196	2.5055259	34.4077
2.625	194	2.6288008	34.8996
2.75	192	2.7725887	35.1765
2.875	191	2.8543782	35.5036
3	189	3.0445224	36.0032

020015

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_45** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 45 °C

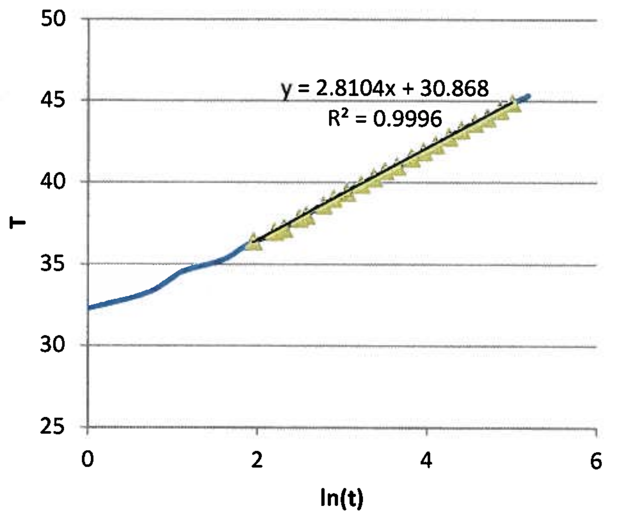
	Heating	Cooling	Average
Slope:	2.8104	3.1927	3.00155
Therm Cond	0.0932	0.0820	0.0872

Current: 0.2 A
R: 89.73 ohms/m
Q: 3.5892 W/m
C: 0.91659 Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.0872

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	36.4343
2.15	9	2.1972246	37.0439
2.3	10	2.3025851	37.2254
2.45	12	2.4849066	37.8447
2.6	13	2.5649494	38.0207
2.75	16	2.7725887	38.6422
2.9	18	2.8903718	39.0566
3.05	21	3.0445224	39.4335
3.2	25	3.2188758	39.9347
3.35	29	3.3672958	40.3368
3.5	33	3.4965076	40.7597
3.65	38	3.6375862	41.0504
3.8	45	3.8066625	41.5428
3.95	52	3.9512437	41.9407
4.1	60	4.0943446	42.365
4.25	70	4.2484952	42.8344
4.4	81	4.3944492	43.2785
4.55	95	4.5538769	43.6682
4.7	110	4.7004804	44.0134
4.85	128	4.8520303	44.4924
5	148	4.9972123	44.9422

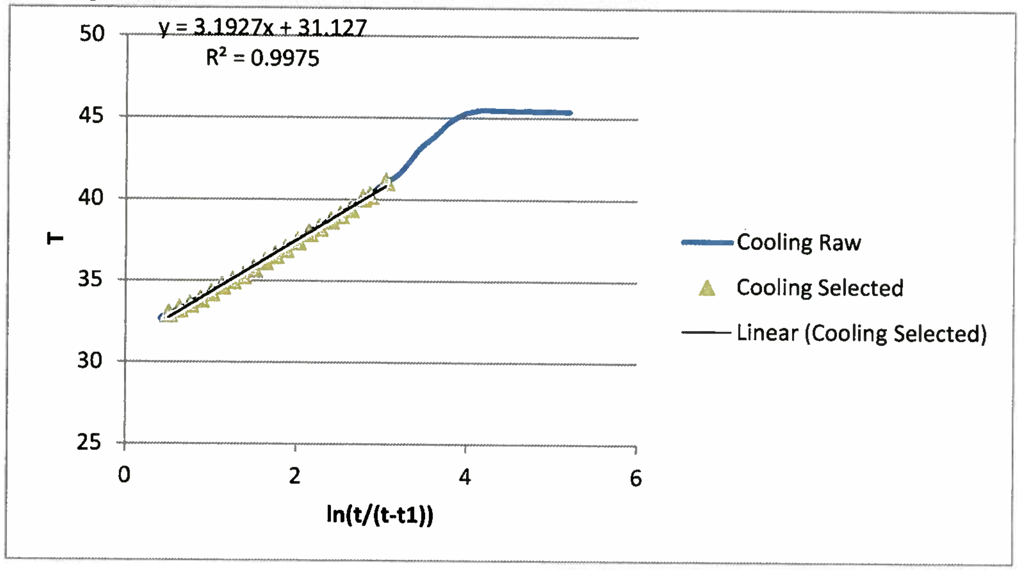
020016

Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_45** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 45 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	32.997
0.625	387	0.6257059	33.2917
0.75	341	0.7504781	33.5878
0.875	309	0.8735289	33.8904
1	285	0.9985288	34.2848
1.125	267	1.1213405	34.7076
1.25	252	1.252763	35.0408
1.375	241	1.3739231	35.3531
1.5	232	1.4954937	35.7624
1.625	224	1.6274564	36.2172
1.75	218	1.7469089	36.6053
1.875	213	1.8647846	36.9968
2	208	2.0053336	37.4601
2.125	204	2.1400662	37.9716
2.25	201	2.2587825	38.3079
2.375	198	2.3978953	38.7431
2.5	196	2.5055259	39.0827
2.625	194	2.6288008	39.4513
2.75	192	2.7725887	40.0984
2.875	191	2.8543782	40.2794
3	189	3.0445224	41.146

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Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 45d**   Client ID: (2/15) Cs Loaded Resorcinol Resin   Actual Test Temperature: 45 °C

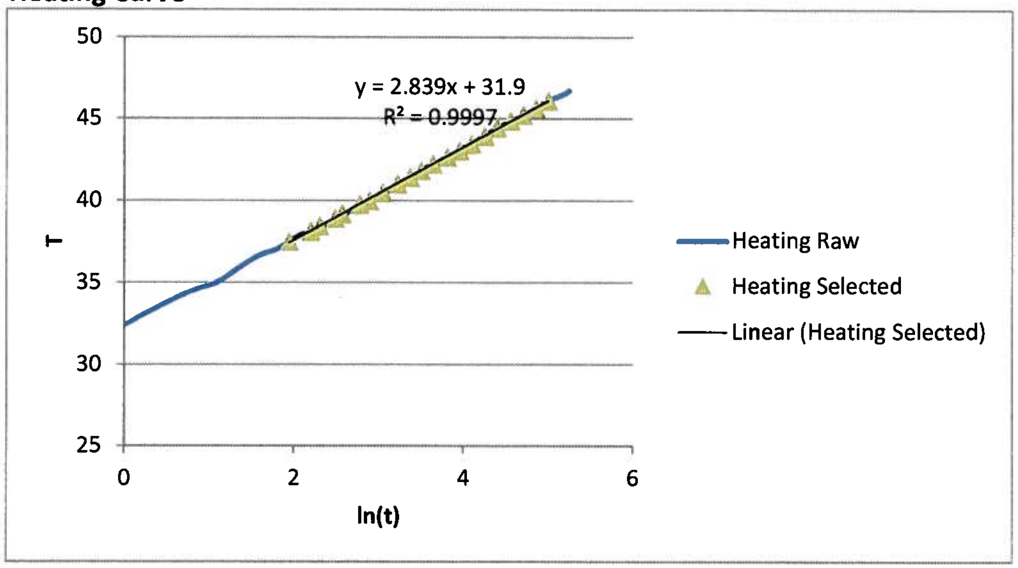
	Heating	Cooling	Average
Slope:	2.839	2.9849	2.91195
Therm Cond	0.0922	0.0877	0.0899

Current	0.2	A
R	89.73	ohms/m
Q	3.5892	W/m
C	0.91659	Cal Factor

Heating:	On (s)	Off (s)	Delta
	50	240	190

Thermal Conductivity, W/(m·K) = 0.0899

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time	ln(t)	T, °C
2	7	1.9459101	37.5256
2.15	9	2.1972246	38.1181
2.3	10	2.3025851	38.4662
2.45	12	2.4849066	38.9353
2.6	13	2.5649494	39.1966
2.75	16	2.7725887	39.7901
2.9	18	2.8903718	40.0039
3.05	21	3.0445224	40.4962
3.2	25	3.2188758	41.038
3.35	29	3.3672958	41.4315
3.5	33	3.4965076	41.8421
3.65	38	3.6375862	42.2599
3.8	45	3.8066625	42.6783
3.95	52	3.9512437	43.0674
4.1	60	4.0943446	43.4748
4.25	70	4.2484952	43.954
4.4	81	4.3944492	44.4621
4.55	95	4.5538769	44.8861
4.7	110	4.7004804	45.2409
4.85	128	4.8520303	45.6622
5	148	4.9972123	46.1089

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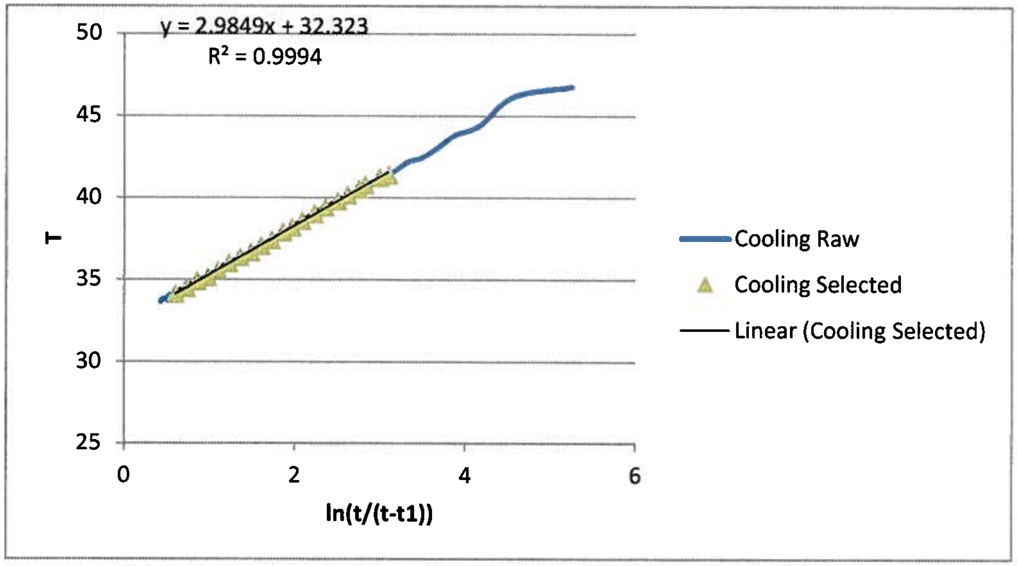
Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 45d** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 45 °C

Cooling Curve



	Beg	End	Delta
Range	0.6	3.1	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.6	421	0.6002151	34.1379
0.725	368	0.7262994	34.4853
0.85	332	0.8493079	34.9108
0.975	305	0.9753796	35.1504
1.1	285	1.0986123	35.5756
1.225	269	1.2252635	36.016
1.35	256	1.3555227	36.3701
1.475	246	1.4799798	36.6699
1.6	238	1.6010697	37.0559
1.725	231	1.7288456	37.4363
1.85	225	1.8607523	37.9061
1.975	221	1.9641755	38.1932
2.1	217	2.0840605	38.595
2.225	213	2.2257979	39.0287
2.35	210	2.3513753	39.4248
2.475	207	2.4995054	39.808
2.6	205	2.6149598	40.179
2.725	203	2.7482566	40.5191
2.85	202	2.823361	40.7928
2.975	200	2.9957323	41.2464
3.1	199	3.0960802	41.4164

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Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 45t**   Client ID: (2/15) Cs Loaded Resorcinol Resin   Actual Test Temperature: 45 °C

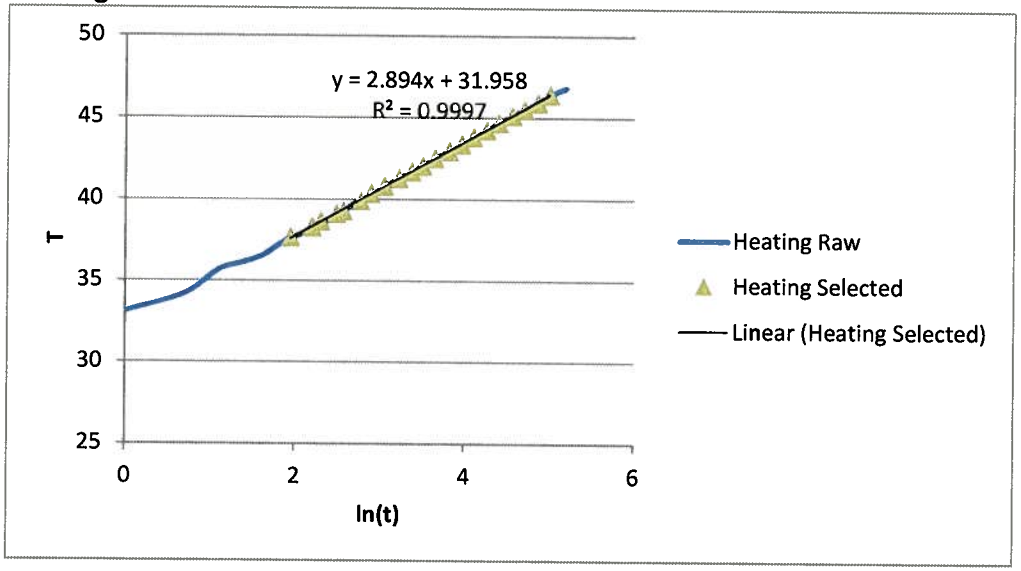
	Heating	Cooling	Average
Slope:	2.894	3.0993	2.99665
Therm Cond	0.0905	0.0845	0.0874

Thermal Conductivity, W/(m·K) = 0.0874

Current: 0.2 A
R: 89.73 ohms/m
Q: 3.5892 W/m
C: 0.91659 Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	37.6623
2.15	9	2.1972246	38.2949
2.3	10	2.3025851	38.6411
2.45	12	2.4849066	39.1166
2.6	13	2.5649494	39.2595
2.75	16	2.7725887	39.9049
2.9	18	2.8903718	40.3745
3.05	21	3.0445224	40.8264
3.2	25	3.2188758	41.3009
3.35	29	3.3672958	41.7117
3.5	33	3.4965076	42.0649
3.65	38	3.6375862	42.5115
3.8	45	3.8066625	42.968
3.95	52	3.9512437	43.389
4.1	60	4.0943446	43.8244
4.25	70	4.2484952	44.2616
4.4	81	4.3944492	44.7205
4.55	95	4.5538769	45.1454
4.7	110	4.7004804	45.5218
4.85	128	4.8520303	45.9348
5	148	4.9972123	46.4469

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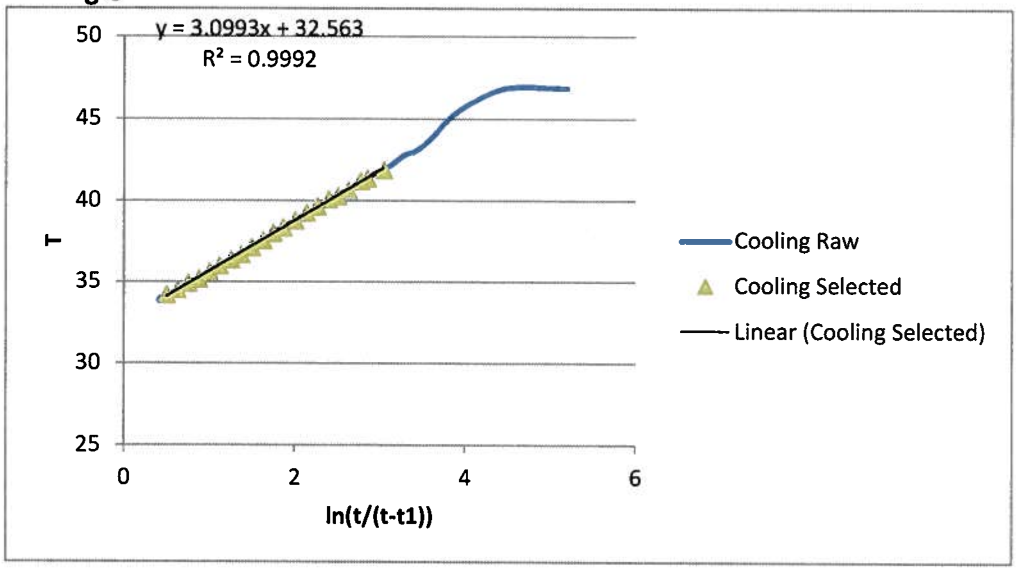
Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_45t** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 45 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	34.2172
0.625	387	0.6257059	34.5369
0.75	341	0.7504781	34.9266
0.875	309	0.8735289	35.1855
1	285	0.9985288	35.6335
1.125	267	1.1213405	36.0145
1.25	252	1.252763	36.3719
1.375	241	1.3739231	36.7084
1.5	232	1.4954937	37.1484
1.625	224	1.6274564	37.5763
1.75	218	1.7469089	38.0222
1.875	213	1.8647846	38.3603
2	208	2.0053336	38.7921
2.125	204	2.1400662	39.2711
2.25	201	2.2587825	39.6606
2.375	198	2.3978953	40.0944
2.5	196	2.5055259	40.2981
2.625	194	2.6288008	40.661
2.75	192	2.7725887	41.2282
2.875	191	2.8543782	41.3773
3	189	3.0445224	41.9069

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Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 90** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 90 °C

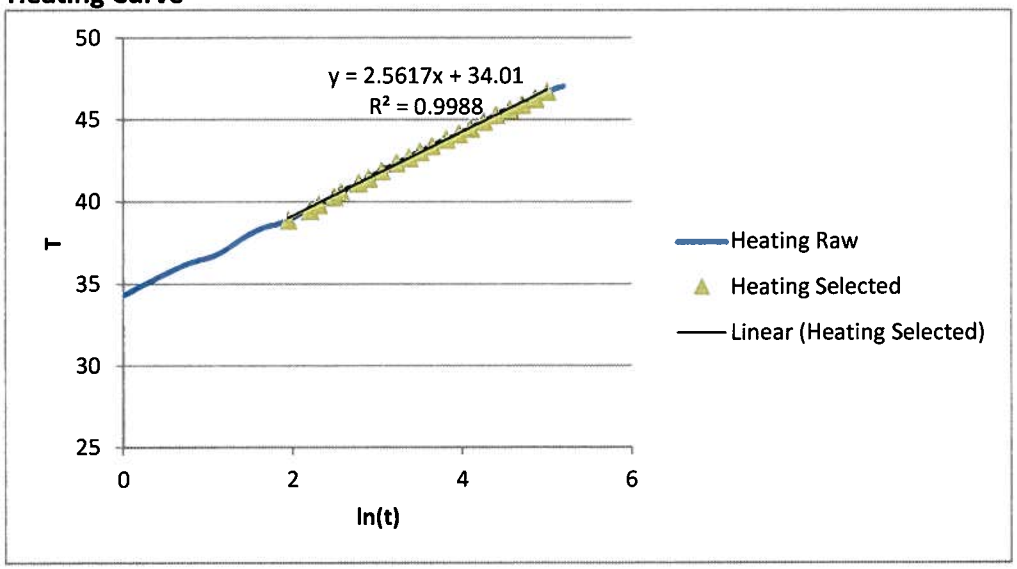
	Heating	Cooling	Average
Slope:	2.5617	2.6203	2.591
Therm Cond	0.1022	0.0999	0.1010

Current: 0.2 A
R: 89.73 ohms/m
Q: 3.5892 W/m
C: 0.91659 Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.1010

Heating Curve



Range	Beg	End	Delta
	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	38.8924
2.15	9	2.1972246	39.488
2.3	10	2.3025851	39.8538
2.45	12	2.4849066	40.3153
2.6	13	2.5649494	40.5899
2.75	16	2.7725887	41.1508
2.9	18	2.8903718	41.4517
3.05	21	3.0445224	41.8834
3.2	25	3.2188758	42.3919
3.35	29	3.3672958	42.7015
3.5	33	3.4965076	43.0525
3.65	38	3.6375862	43.4154
3.8	45	3.8066625	43.83
3.95	52	3.9512437	44.1506
4.1	60	4.0943446	44.5079
4.25	70	4.2484952	44.915
4.4	81	4.3944492	45.3194
4.55	95	4.5538769	45.6608
4.7	110	4.7004804	45.9283
4.85	128	4.8520303	46.3014
5	148	4.9972123	46.743

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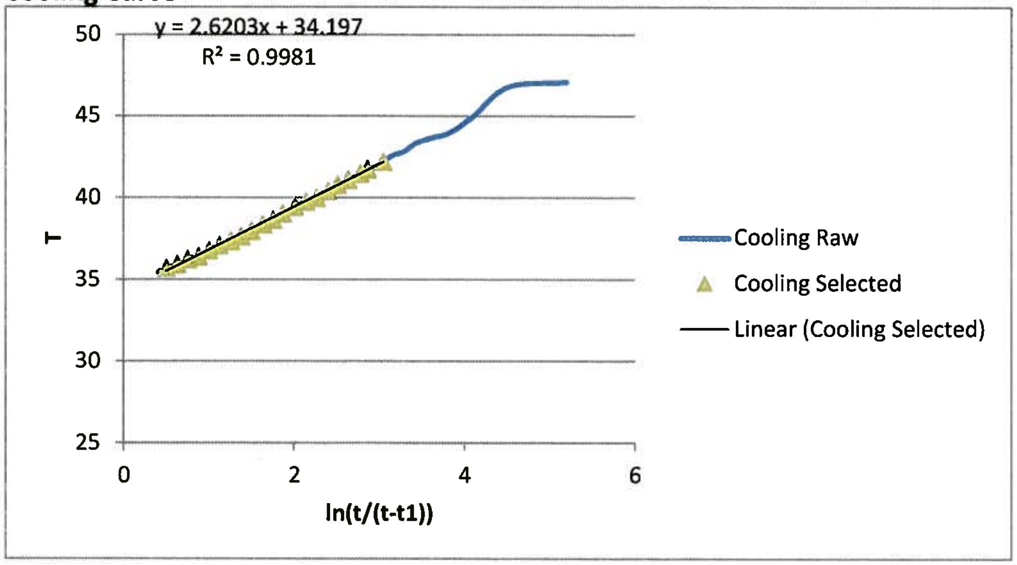
Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_90** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 90 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	35.7107
0.625	387	0.6257059	35.9505
0.75	341	0.7504781	36.2528
0.875	309	0.8735289	36.4318
1	285	0.9985288	36.813
1.125	267	1.1213405	37.1123
1.25	252	1.252763	37.3673
1.375	241	1.3739231	37.6596
1.5	232	1.4954937	37.9936
1.625	224	1.6274564	38.4086
1.75	218	1.7469089	38.6783
1.875	213	1.8647846	39.0718
2	208	2.0053336	39.4678
2.125	204	2.1400662	39.7834
2.25	201	2.2587825	40.0225
2.375	198	2.3978953	40.4573
2.5	196	2.5055259	40.821
2.625	194	2.6288008	41.1265
2.75	192	2.7725887	41.5045
2.875	191	2.8543782	41.7824
3	189	3.0445224	42.2427

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Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_90d** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 90 °C

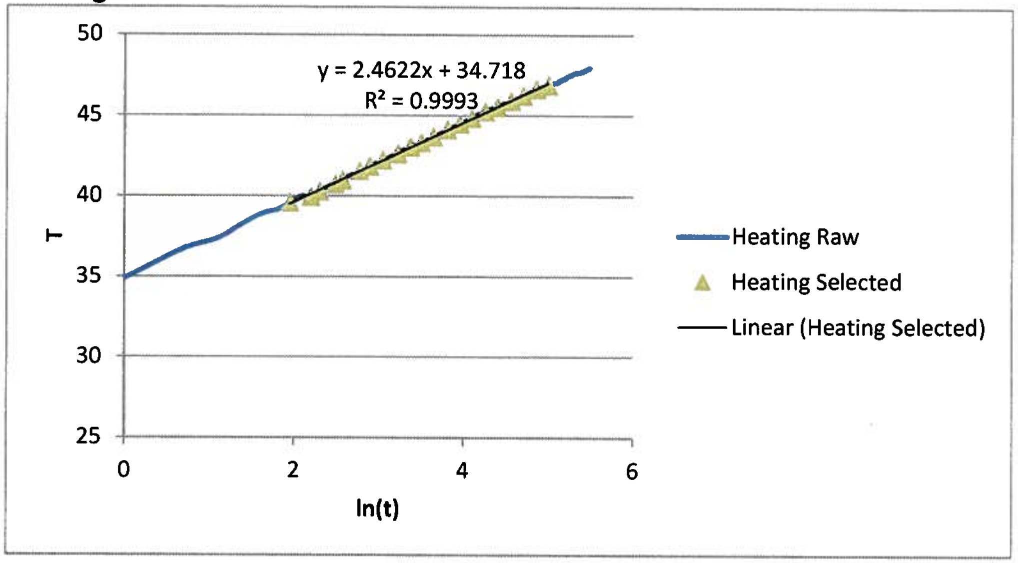
	Heating	Cooling	Average
Slope	2.4622	2.6639	2.56305
Therm Cond	0.1063	0.0983	0.1021

Current: 0.2 A
R: 89.73 ohms/m
Q: 3.5892 W/m
C: 0.91659 Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	300	240

Thermal Conductivity, W/(m·K) = 0.1021

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	Temp
2	7	1.9459101	39.61
2.15	9	2.1972246	39.9867
2.3	10	2.3025851	40.3269
2.45	12	2.4849066	40.7643
2.6	13	2.5649494	41.018
2.75	16	2.7725887	41.5841
2.9	18	2.8903718	41.8475
3.05	21	3.0445224	42.246
3.2	25	3.2188758	42.6417
3.35	29	3.3672958	43.0554
3.5	33	3.4965076	43.3265
3.65	38	3.6375862	43.6903
3.8	45	3.8066625	44.1504
3.95	52	3.9512437	44.5076
4.1	60	4.0943446	44.8402
4.25	70	4.2484952	45.2601
4.4	81	4.3944492	45.5314
4.55	95	4.5538769	45.908
4.7	110	4.7004804	46.2485
4.85	128	4.8520303	46.6634
5	148	4.9972123	46.9009

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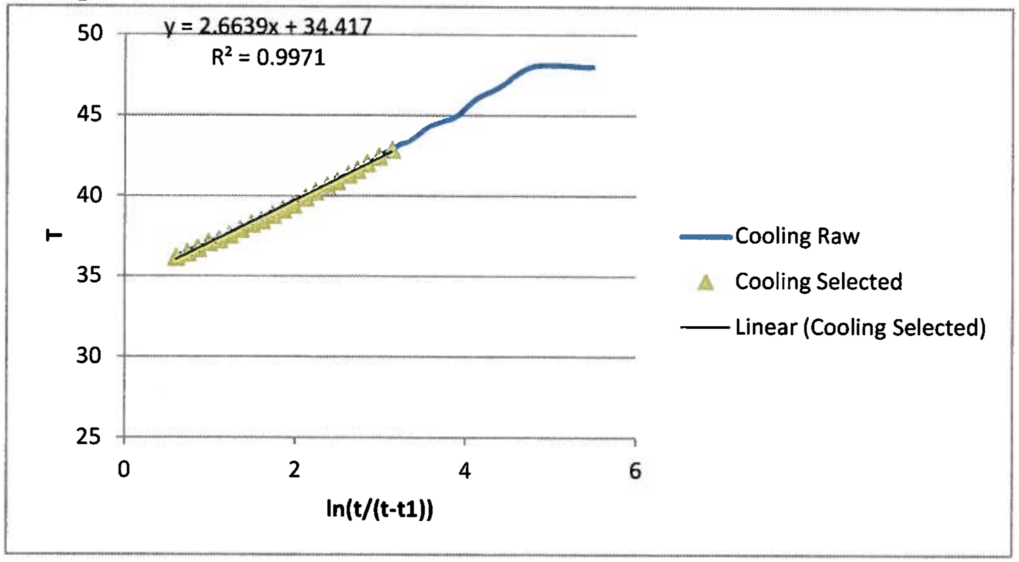
Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 90d** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 90 °C

Cooling Curve



	Beg	End	Delta
Range	0.6	3.1	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	Temp
0.6	532	0.5998897	36.206
0.725	465	0.725937	36.4817
0.85	419	0.8504851	36.758
0.975	385	0.9765096	37.1336
1.1	360	1.0986123	37.3172
1.225	340	1.2237754	37.62
1.35	324	1.3499267	37.9733
1.475	311	1.477113	38.3041
1.6	301	1.5962364	38.5295
1.725	292	1.7255101	38.8666
1.85	285	1.8458267	39.1907
1.975	279	1.9676501	39.493
2.1	273	2.1129642	39.9454
2.225	269	2.2274155	40.3195
2.35	265	2.360854	40.6907
2.475	262	2.4773021	40.9634
2.6	259	2.6123891	41.3915
2.725	257	2.7158627	41.655
2.85	255	2.8332133	42.0848
2.975	253	2.9684401	42.4835
3.1	251	3.1275577	42.9073

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Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163_90t** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 90 °C

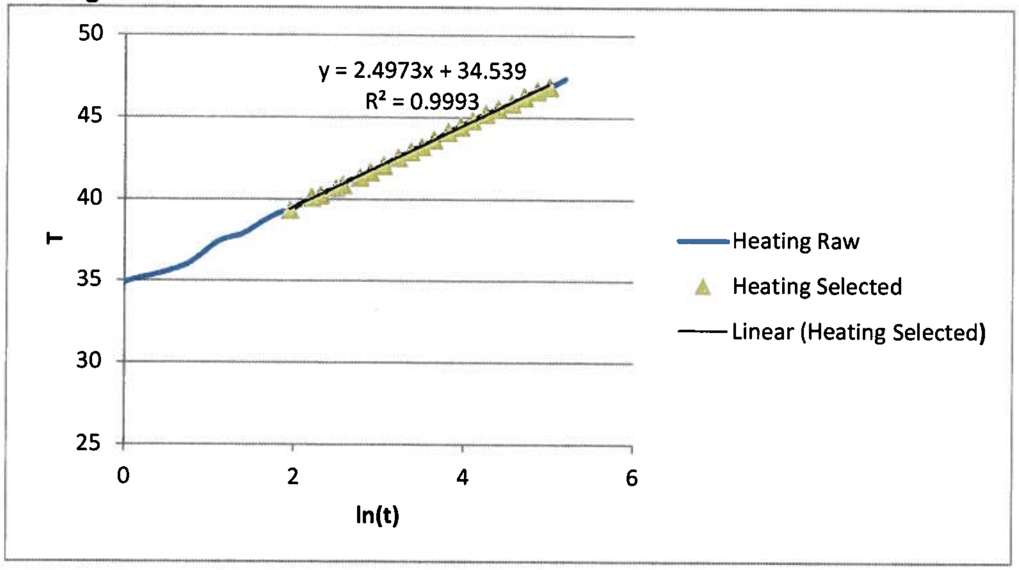
	Heating	Cooling	Average
Slope	2.4973	2.6191	2.5582
Therm Cond	0.1048	0.1000	0.1023

Current: 0.2 A
R: 89.73 ohms/m
Q: 3.5892 W/m
C: 0.91659 Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Thermal Conductivity, W/(m·K) = 0.1023

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	39.3838
2.15	9	2.1972246	40.1309
2.3	10	2.3025851	40.2836
2.45	12	2.4849066	40.7101
2.6	13	2.5649494	40.9291
2.75	16	2.7725887	41.3744
2.9	18	2.8903718	41.6915
3.05	21	3.0445224	42.0883
3.2	25	3.2188758	42.5941
3.35	29	3.3672958	42.936
3.5	33	3.4965076	43.2455
3.65	38	3.6375862	43.653
3.8	45	3.8066625	44.1458
3.95	52	3.9512437	44.4758
4.1	60	4.0943446	44.8457
4.25	70	4.2484952	45.2434
4.4	81	4.3944492	45.5347
4.55	95	4.5538769	45.8832
4.7	110	4.7004804	46.2591
4.85	128	4.8520303	46.6305
5	148	4.9972123	46.8933

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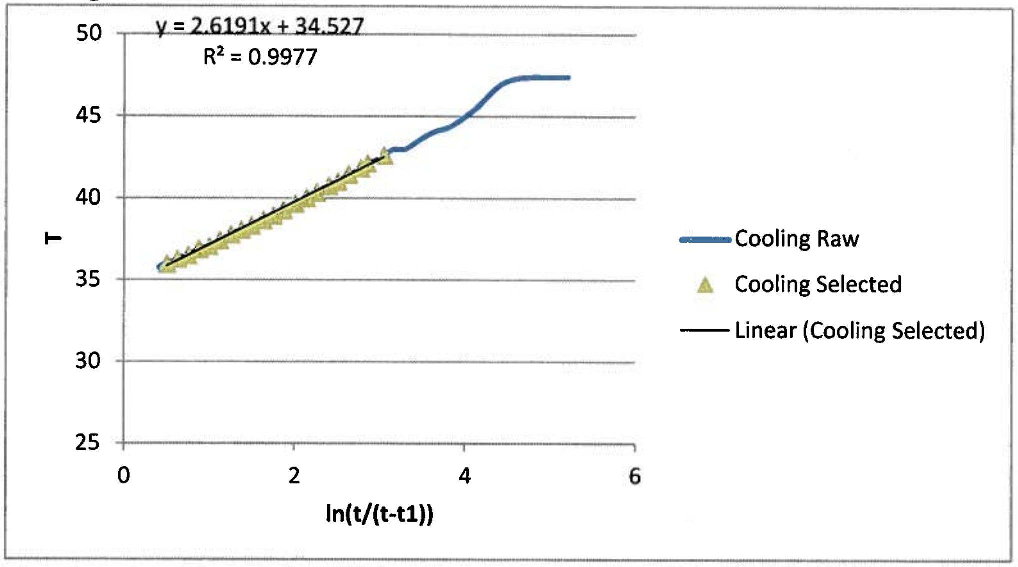
Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **486163 90t** Client ID: (2/15) Cs Loaded Resorcinol Resin Actual Test Temperature: 90 °C

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	35.9959
0.625	387	0.6257059	36.3156
0.75	341	0.7504781	36.5121
0.875	309	0.8735289	36.9053
1	285	0.9985288	37.0861
1.125	267	1.1213405	37.4441
1.25	252	1.252763	37.821
1.375	241	1.3739231	38.0886
1.5	232	1.4954937	38.3452
1.625	224	1.6274564	38.6849
1.75	218	1.7469089	38.9545
1.875	213	1.8647846	39.2823
2	208	2.0053336	39.6948
2.125	204	2.1400662	40.0461
2.25	201	2.2587825	40.3873
2.375	198	2.3978953	40.7803
2.5	196	2.5055259	41.0603
2.625	194	2.6288008	41.4838
2.75	192	2.7725887	41.8673
2.875	191	2.8543782	42.1581
3	189	3.0445224	42.6447

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Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

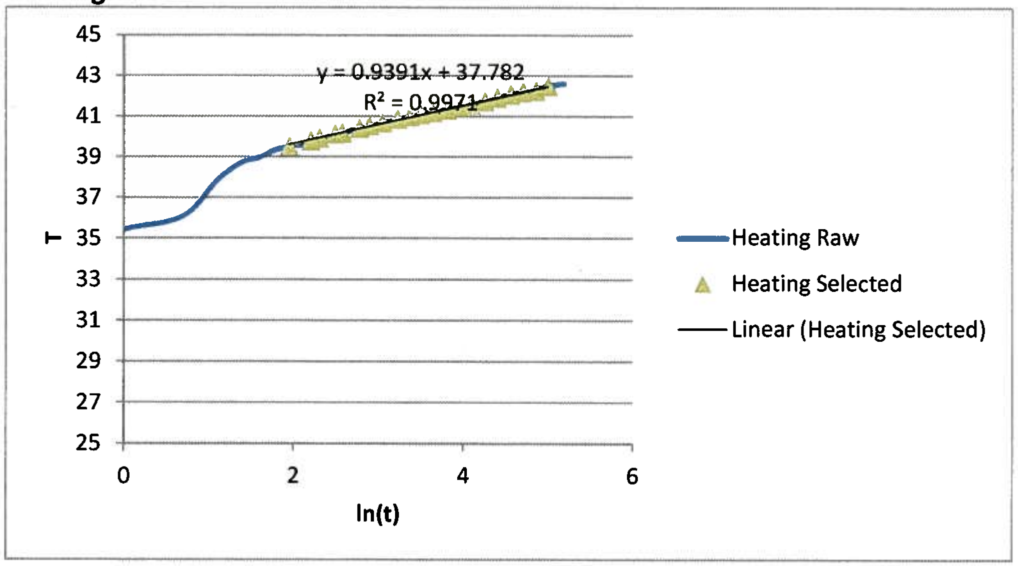
Sample ID: **Glycerine CCV 031712**

	Heating	Cooling	Average
Slope	0.9391	0.8567	0.8979
Therm Cond	0.279	0.306	**0.292**
TV	0.292	0.292	0.292
Recovery	95.5%	105%	100%

Current:	0.2	A
R:	89.73	ohms/m
Q:	3.5892	W/m
C:	0.91659	Cal Factor

Heating:	On (s)	Off (s)	Delta
	60	240	180

Heating Curve



	Beg	End	Delta
Range	2	5	0.15

Goal ln(t)	Time,s	ln(t)	T, °C
2	7	1.9459101	39.5172
2.15	9	2.1972246	39.7857
2.3	10	2.3025851	39.9414
2.45	12	2.4849066	40.1418
2.6	13	2.5649494	40.1828
2.75	16	2.7725887	40.4147
2.9	18	2.8903718	40.5348
3.05	21	3.0445224	40.6904
3.2	25	3.2188758	40.8789
3.35	29	3.3672958	40.9924
3.5	33	3.4965076	41.1098
3.65	38	3.6375862	41.2103
3.8	45	3.8066625	41.3484
3.95	52	3.9512437	41.4489
4.1	60	4.0943446	41.5478
4.25	70	4.2484952	41.7409
4.4	81	4.3944492	41.9201
4.55	95	4.5538769	42.0884
4.7	110	4.7004804	42.1953
4.85	128	4.8520303	42.2632
5	148	4.9972123	42.5018

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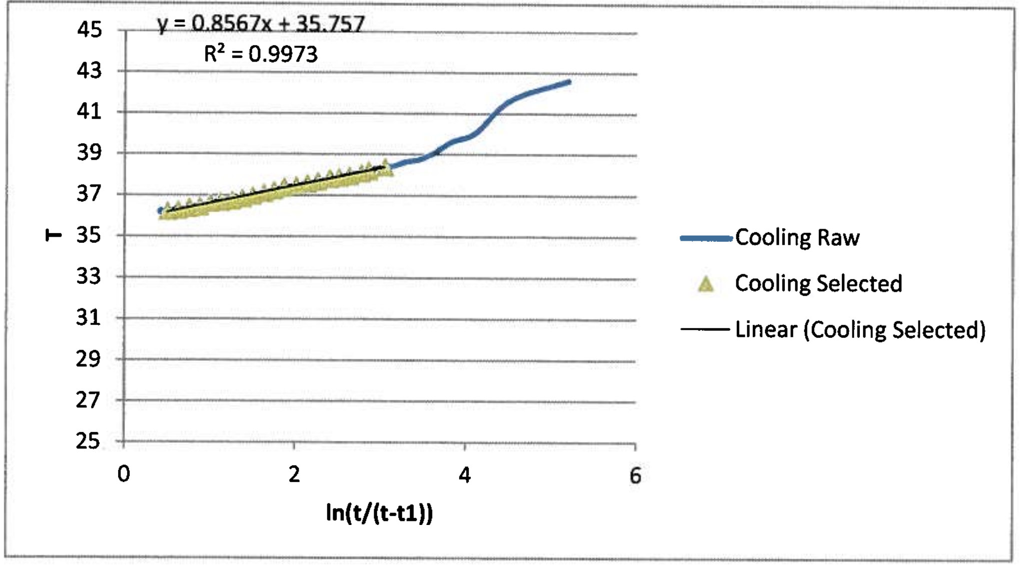
Southwest Research Institute

Thermal Conductivity - ASTM D5334/D5930

Sample ID: **Glycerine CCV 031712**

Cooling Curve



	Beg	End	Delta
Range	0.5	3	0.125

Goal ln(t/(t-t1))	Time,s	ln(t/(t-t1))	T, °C
0.5	457	0.5006659	36.25
0.625	387	0.6257059	36.3099
0.75	341	0.7504781	36.4167
0.875	309	0.8735289	36.4653
1	285	0.9985288	36.6429
1.125	267	1.1213405	36.713
1.25	252	1.252763	36.7726
1.375	241	1.3739231	36.8684
1.5	232	1.4954937	37.0073
1.625	224	1.6274564	37.1305
1.75	218	1.7469089	37.2739
1.875	213	1.8647846	37.4128
2	208	2.0053336	37.4699
2.125	204	2.1400662	37.5883
2.25	201	2.2587825	37.7077
2.375	198	2.3978953	37.8298
2.5	196	2.5055259	37.8924
2.625	194	2.6288008	37.9806
2.75	192	2.7725887	38.1092
2.875	191	2.8543782	38.2185
3	189	3.0445224	38.4053

020029

020030

SOUTHWEST RESEARCH INSTITUTE

CLIENT: Battelle Memorial Ins. PNNL

TASK ORDER#: 120319-5

SRR#: 46112

SDG#: 486163

VTSR: 120319-5

PROJECT #: 13295.12.008

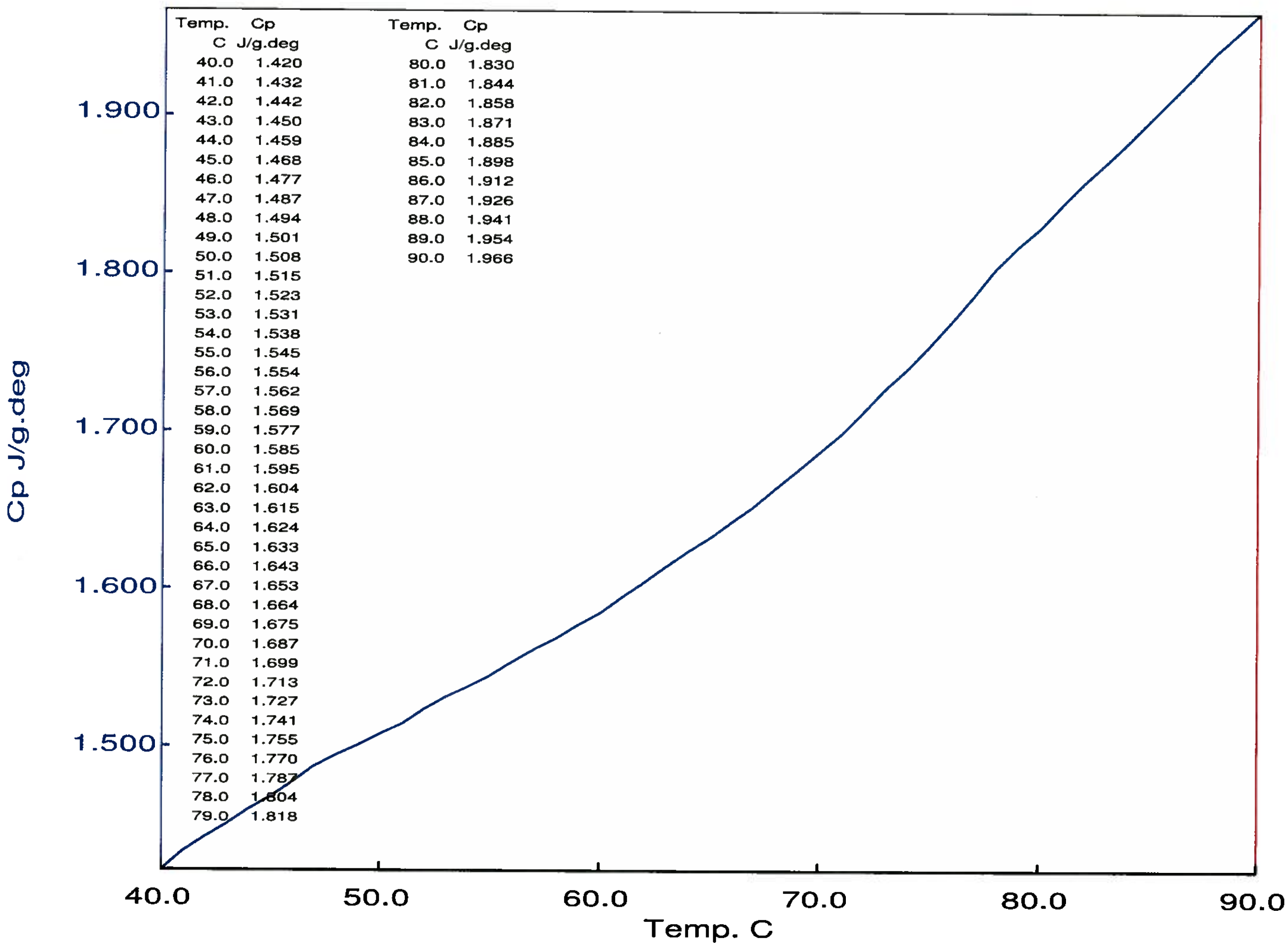
Specific Heat Capacity Data

<< DSC >>
Data Name:120316-486163-cp
Date: 12/ 3/16 17:55
Sample: 486163
9.68 mg
Reference:
0 mg

Temperature Program:

	[C]		[C/min]	[min]	[sec]
1*	25 -	25	10	4	0.2
2*	25 -	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



Temp. C	Cp J/g.deg	Temp. C	Cp J/g.deg
40.0	1.420	80.0	1.830
41.0	1.432	81.0	1.844
42.0	1.442	82.0	1.858
43.0	1.450	83.0	1.871
44.0	1.459	84.0	1.885
45.0	1.468	85.0	1.898
46.0	1.477	86.0	1.912
47.0	1.487	87.0	1.926
48.0	1.494	88.0	1.941
49.0	1.501	89.0	1.954
50.0	1.508	90.0	1.966
51.0	1.515		
52.0	1.523		
53.0	1.531		
54.0	1.538		
55.0	1.545		
56.0	1.554		
57.0	1.562		
58.0	1.569		
59.0	1.577		
60.0	1.585		
61.0	1.595		
62.0	1.604		
63.0	1.615		
64.0	1.624		
65.0	1.633		
66.0	1.643		
67.0	1.653		
68.0	1.664		
69.0	1.675		
70.0	1.687		
71.0	1.699		
72.0	1.713		
73.0	1.727		
74.0	1.741		
75.0	1.755		
76.0	1.770		
77.0	1.787		
78.0	1.804		
79.0	1.818		

SwRI

020031

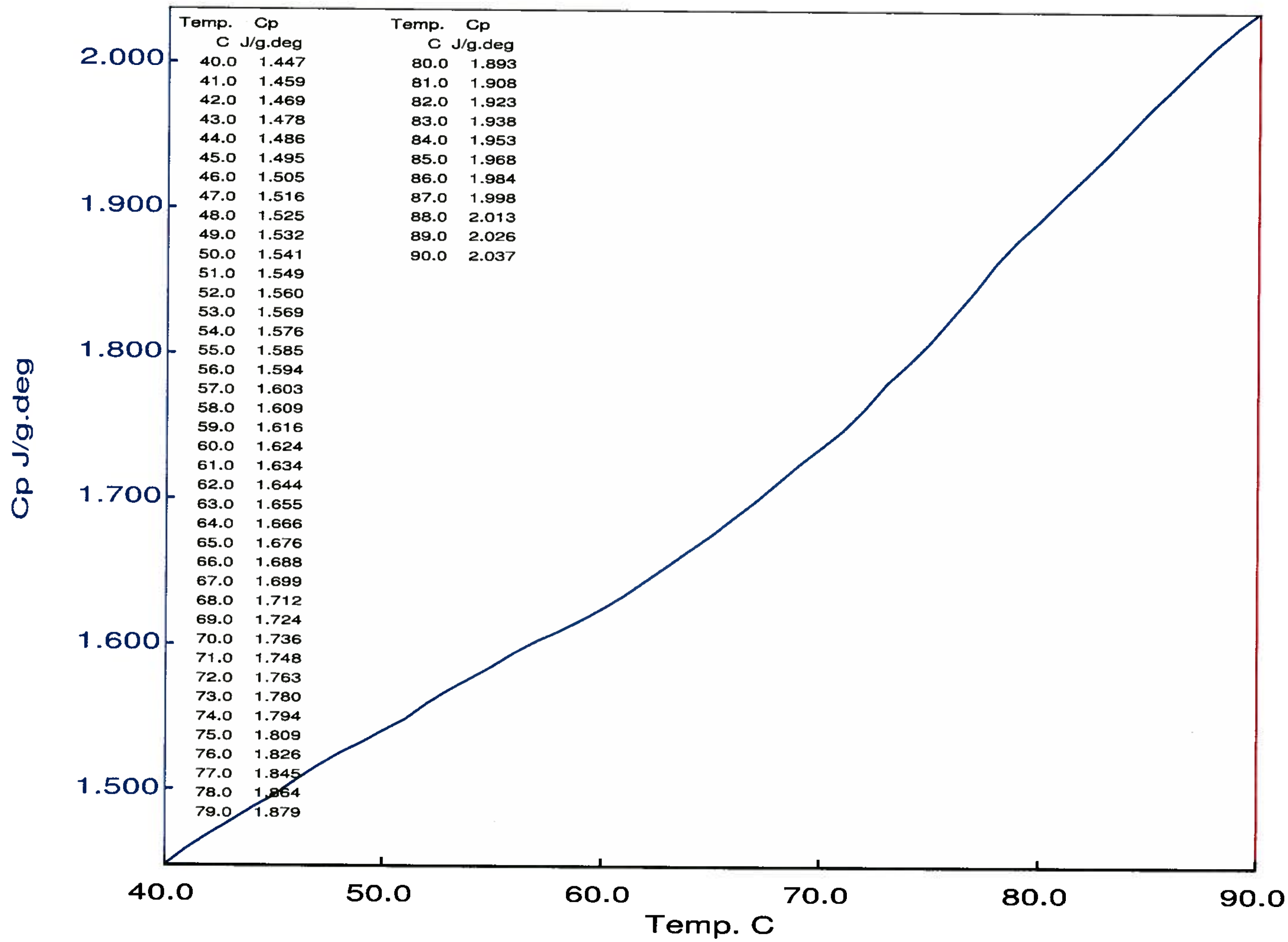
<< DSC >>

Data Name:120316-486163-2cp
Date: 12/ 3/16 18:22
Sample: 486163-2
8.67 mg
Reference:
0 mg

Temperature Program:

	[C]	[C/min]	[min]	[sec]
1*	25 - 25	10	4	0.2
2*	25 - 100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

020032

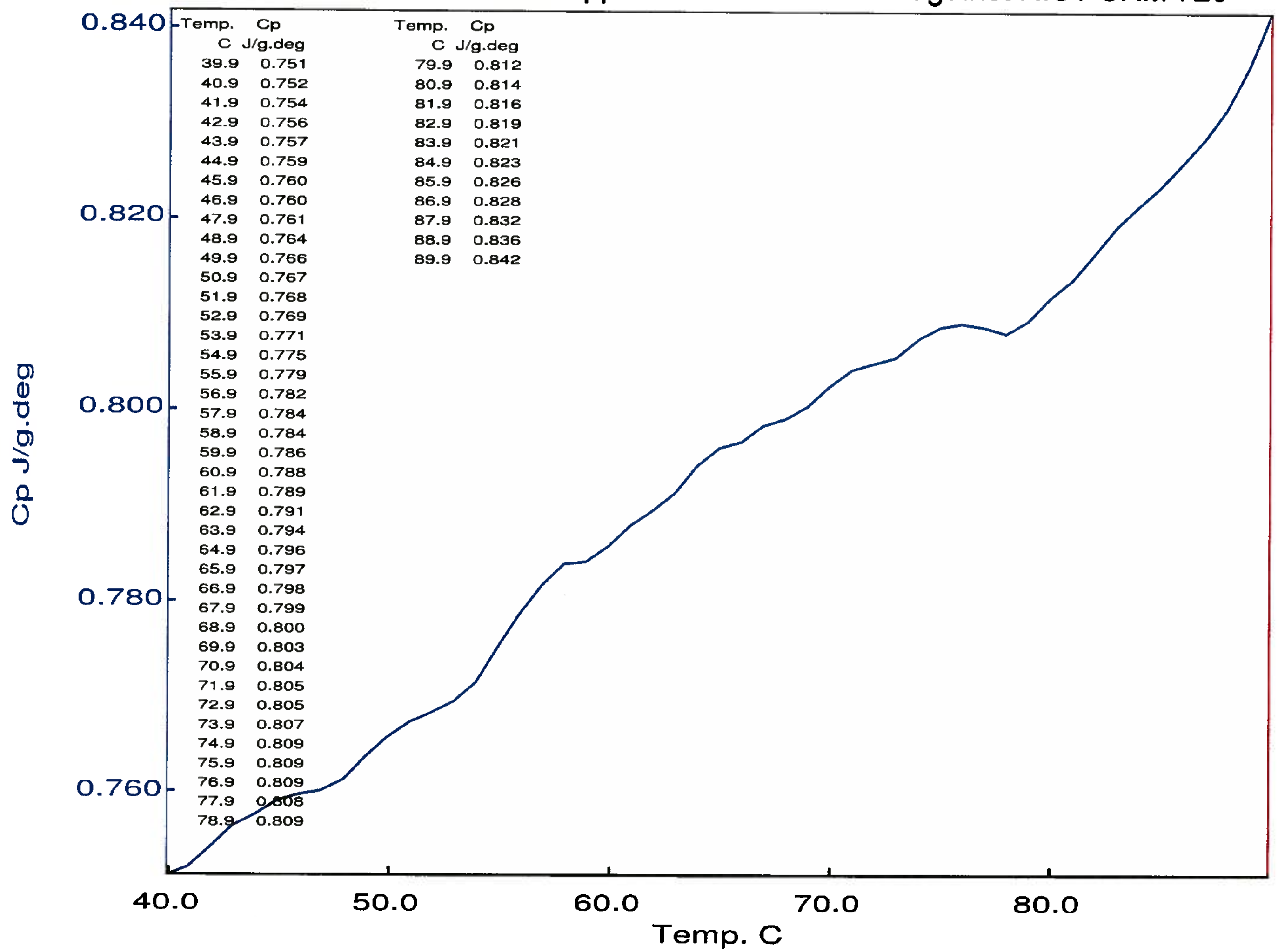
<< DSC >>
Data Name:120316-sap483-Cp v
Date: 12/ 3/16 16:33
Sample: PE sapphire 483
9.04 mg
Reference:
0 mg

Temperature Program:

		[C]	[C/min]	[min]	[sec]
1*	25 -	25	10	4	0.2
2*	25 -	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open

Perkin Elmer #0219 1483 Sapphire Disk Verification against NIST SRM 720



Temp. C	Cp J/g.deg	Temp. C	Cp J/g.deg
39.9	0.751	79.9	0.812
40.9	0.752	80.9	0.814
41.9	0.754	81.9	0.816
42.9	0.756	82.9	0.819
43.9	0.757	83.9	0.821
44.9	0.759	84.9	0.823
45.9	0.760	85.9	0.826
46.9	0.760	86.9	0.828
47.9	0.761	87.9	0.832
48.9	0.764	88.9	0.836
49.9	0.766	89.9	0.842
50.9	0.767		
51.9	0.768		
52.9	0.769		
53.9	0.771		
54.9	0.775		
55.9	0.779		
56.9	0.782		
57.9	0.784		
58.9	0.784		
59.9	0.786		
60.9	0.788		
61.9	0.789		
62.9	0.791		
63.9	0.794		
64.9	0.796		
65.9	0.797		
66.9	0.798		
67.9	0.799		
68.9	0.800		
69.9	0.803		
70.9	0.804		
71.9	0.805		
72.9	0.805		
73.9	0.807		
74.9	0.809		
75.9	0.809		
76.9	0.809		
77.9	0.808		
78.9	0.809		

SwRI

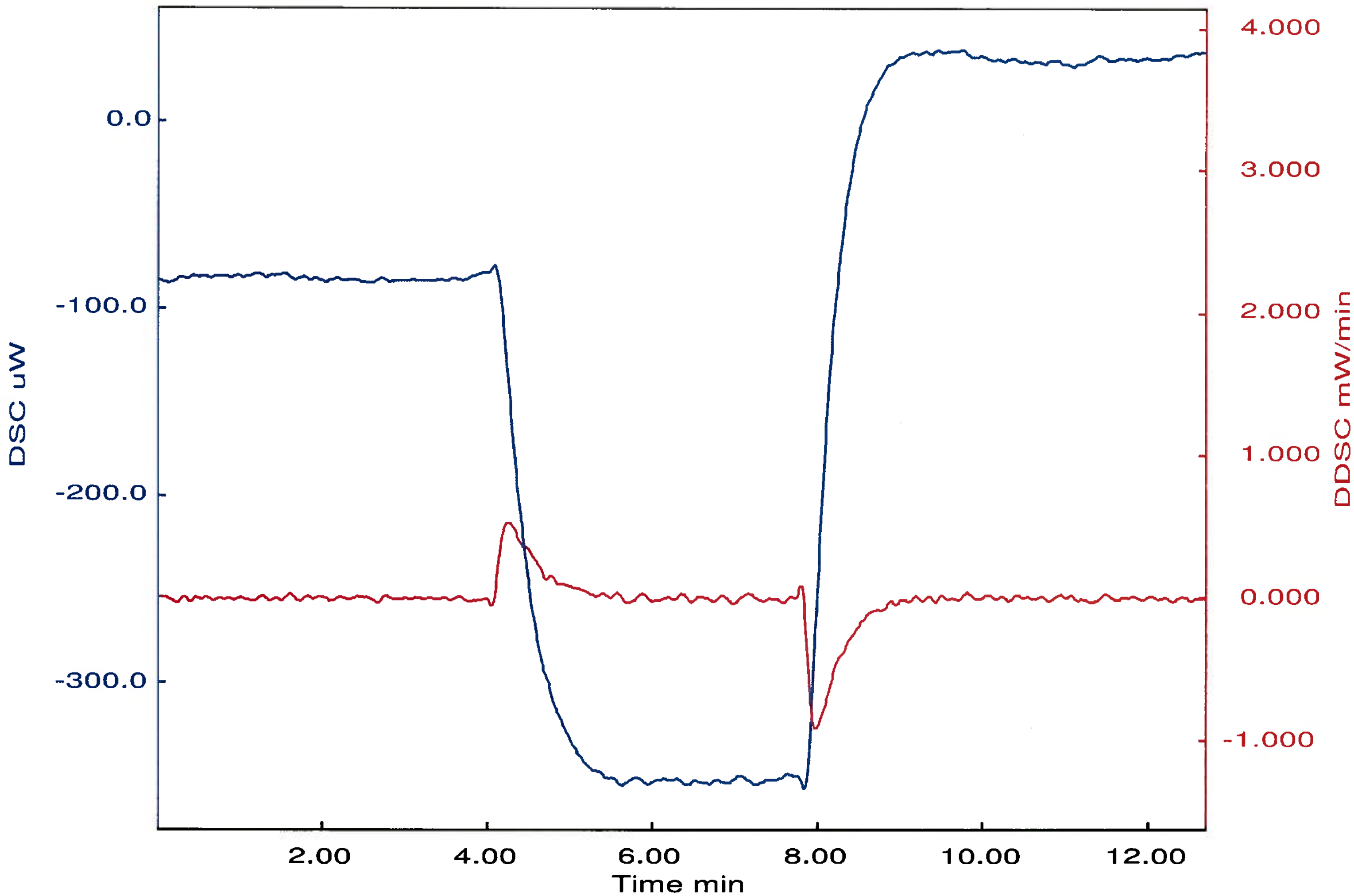
020033

<< DSC >>
Data Name:120316-blk2
Date: 12/ 3/16 15:57
Sample: blk pan
0 mg
Reference:
0 mg

Temperature Program:

			[C]	[C/min]	[min]	[sec]
1*	25 -		25	10	4	0.2
2*	25 -		100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

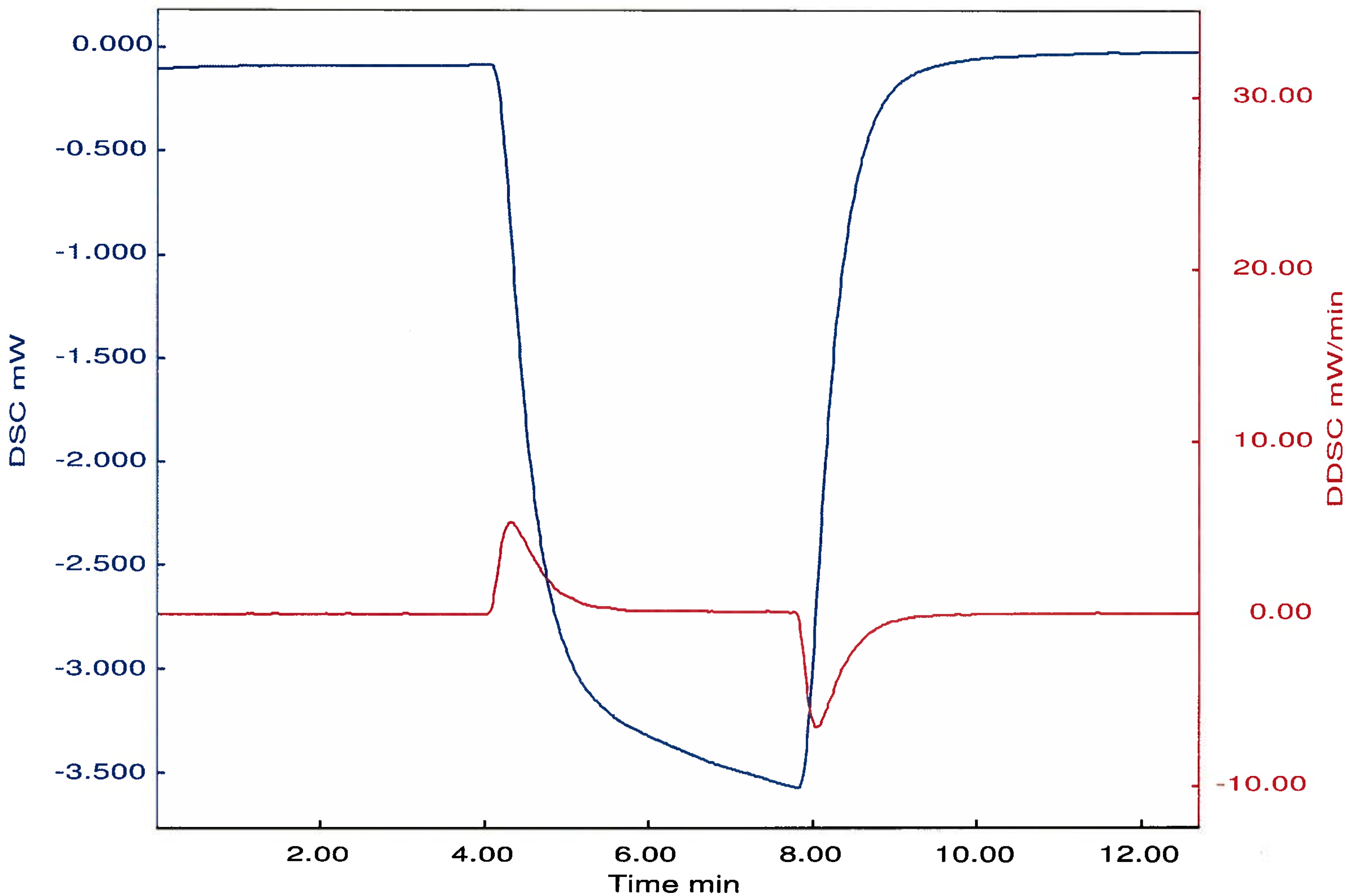
020034

<< DSC >>
Data Name:120316-nist-2
Date: 12/ 3/16 16:59
Sample: NIST sapphire
10.46 mg
Reference:
0 mg

Temperature Program:

	[C]		[C/min]	[min]	[sec]
1*	25 -	25	10	4	0.2
2*	25 -	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

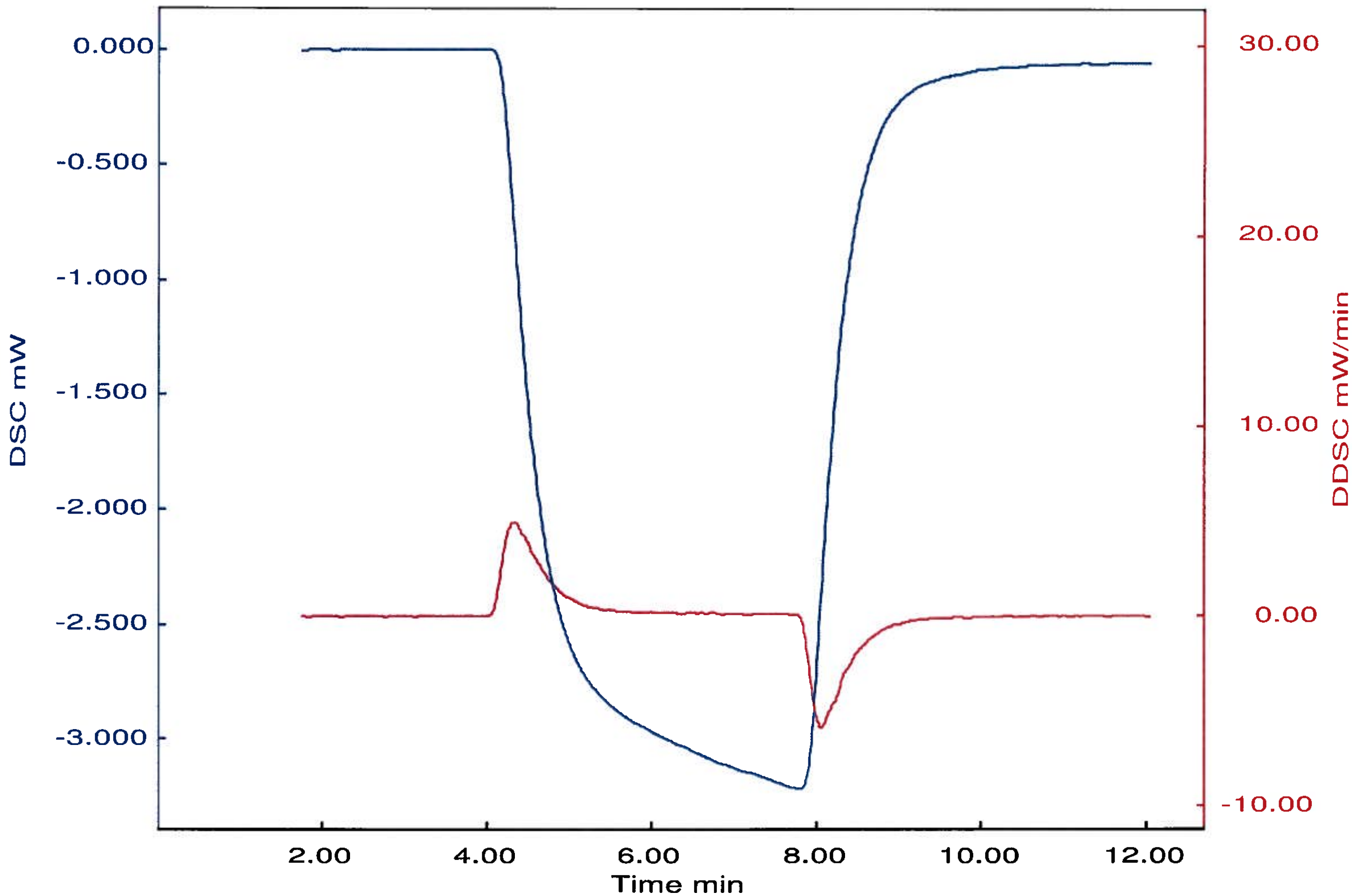
020035

<< DSC >>
Data Name:120316-nist-2sub
Date: 12/ 3/16 16:59
Sample: NIST sapphire
10.46 mg
Reference:
0 mg

Temperature Program:

		[C]	[C/min]	[min]	[sec]
1*	25 -	25	10	4	0.2
2*	25 -	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

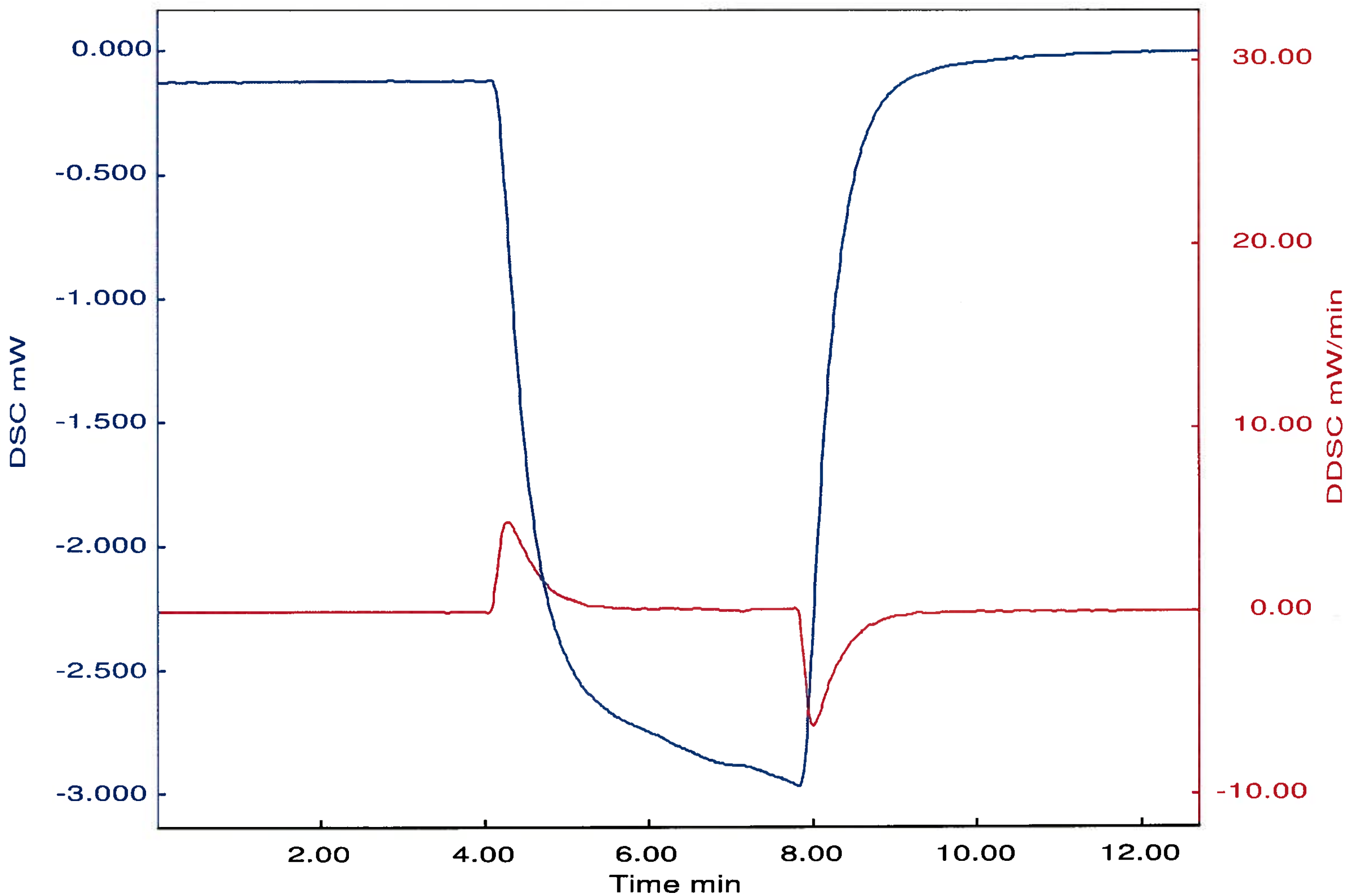
020036

<< DSC >>
Data Name:120316-sap483-2
Date: 12/ 3/16 16:33
Sample: PE sapphire 483
9.04 mg
Reference:
0 mg

Temperature Program:

			[C]	[C/min]	[min]	[sec]
1*	25 -		25	10	4	0.2
2*	25 -		100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

020037

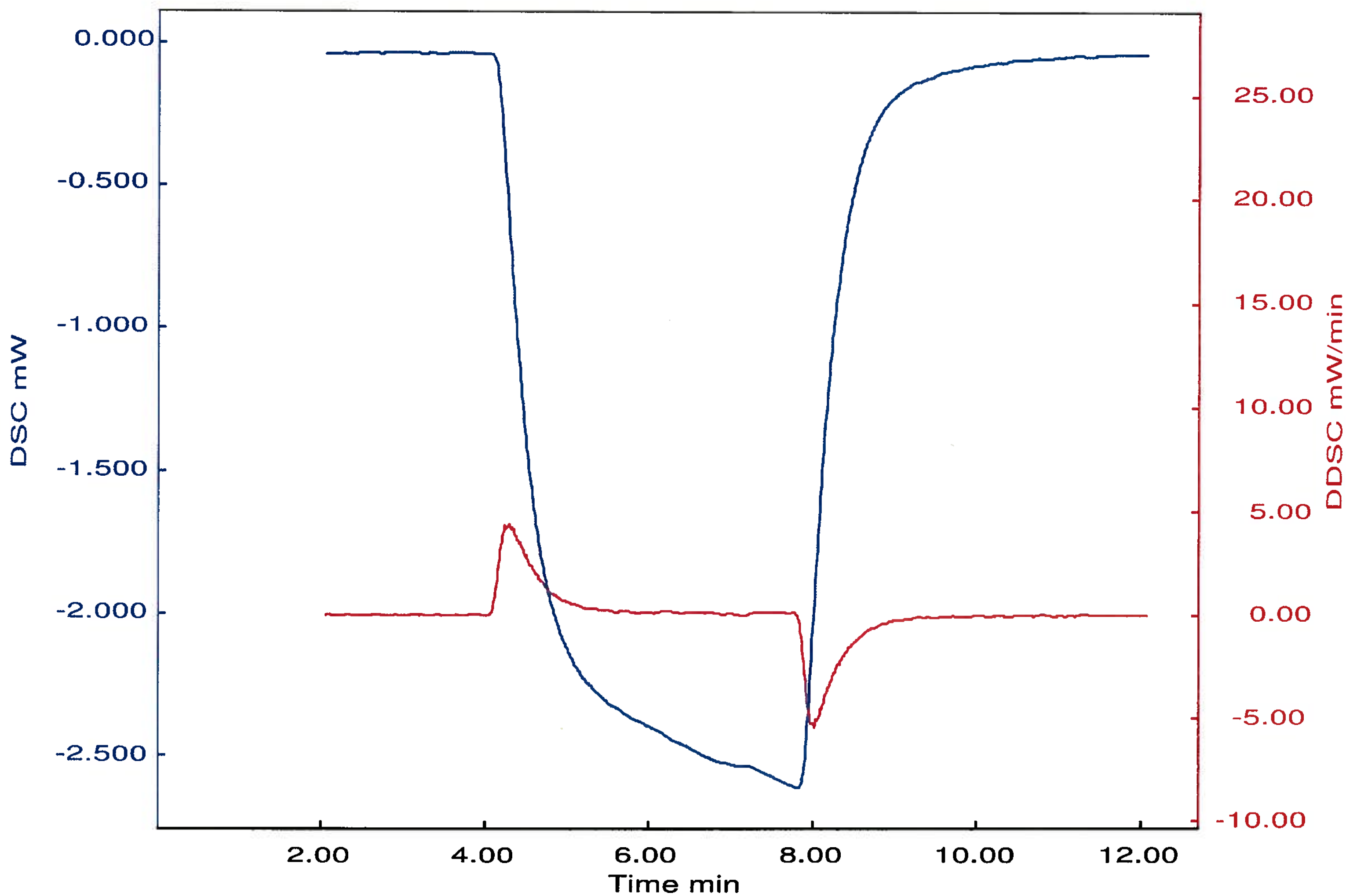
<< DSC >>

Data Name:120316-sap483-2sub
Date: 12/ 3/16 16:33
Sample: PE sapphire 483
9.04 mg
Reference:
0 mg

Temperature Program:

	[C]	[C/min]	[min]	[sec]
1* 25 -	25	10	4	0.2
2* 25 -	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

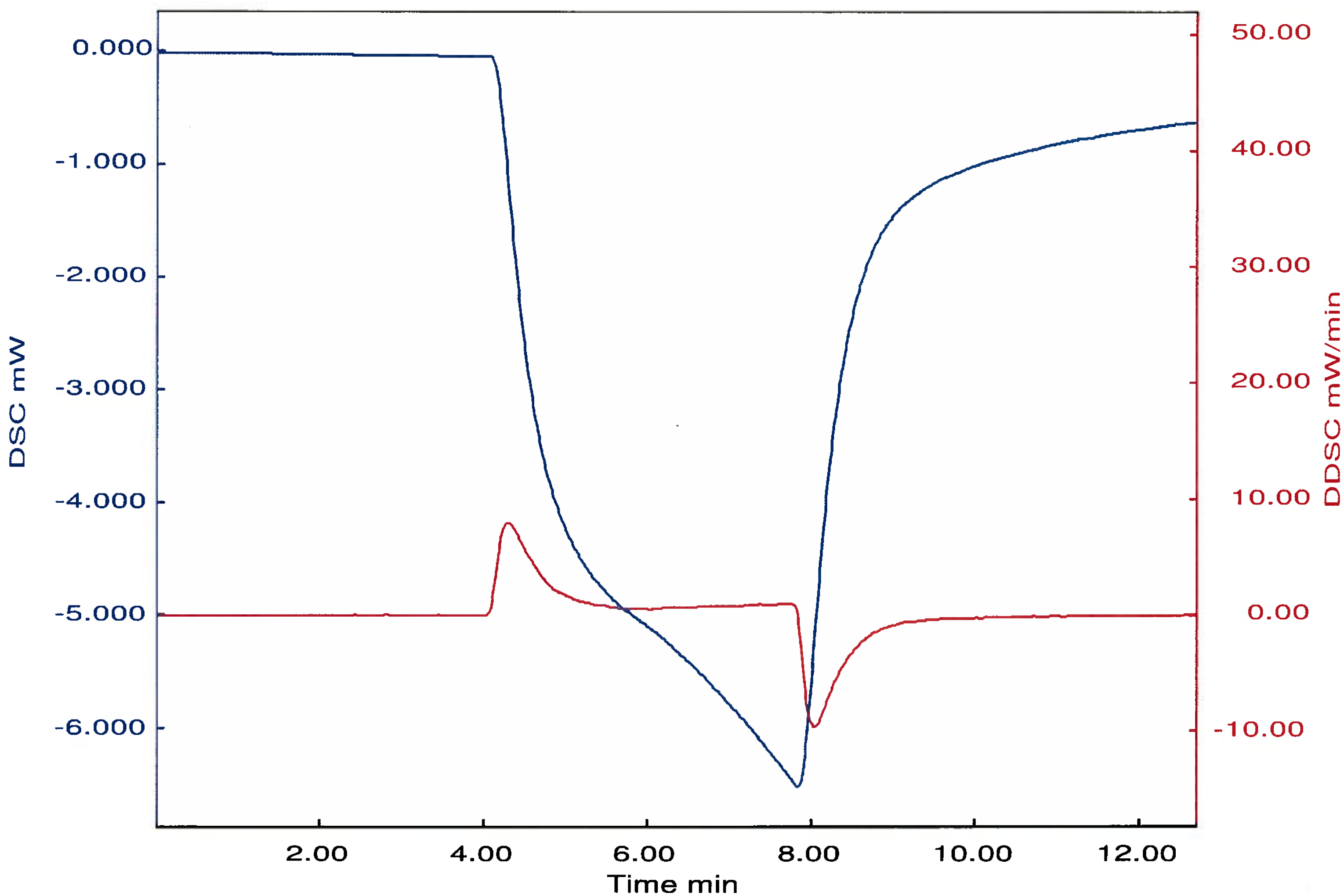
020038

<< DSC >>
Data Name:120316-486163
Date: 12/ 3/16 17:55
Sample: 486163
9.68 mg
Reference:
0 mg

Temperature Program:

			[C]	[C/min]	[min]	[sec]
1*	25 -	25		10	4	0.2
2*	25 -	100		20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

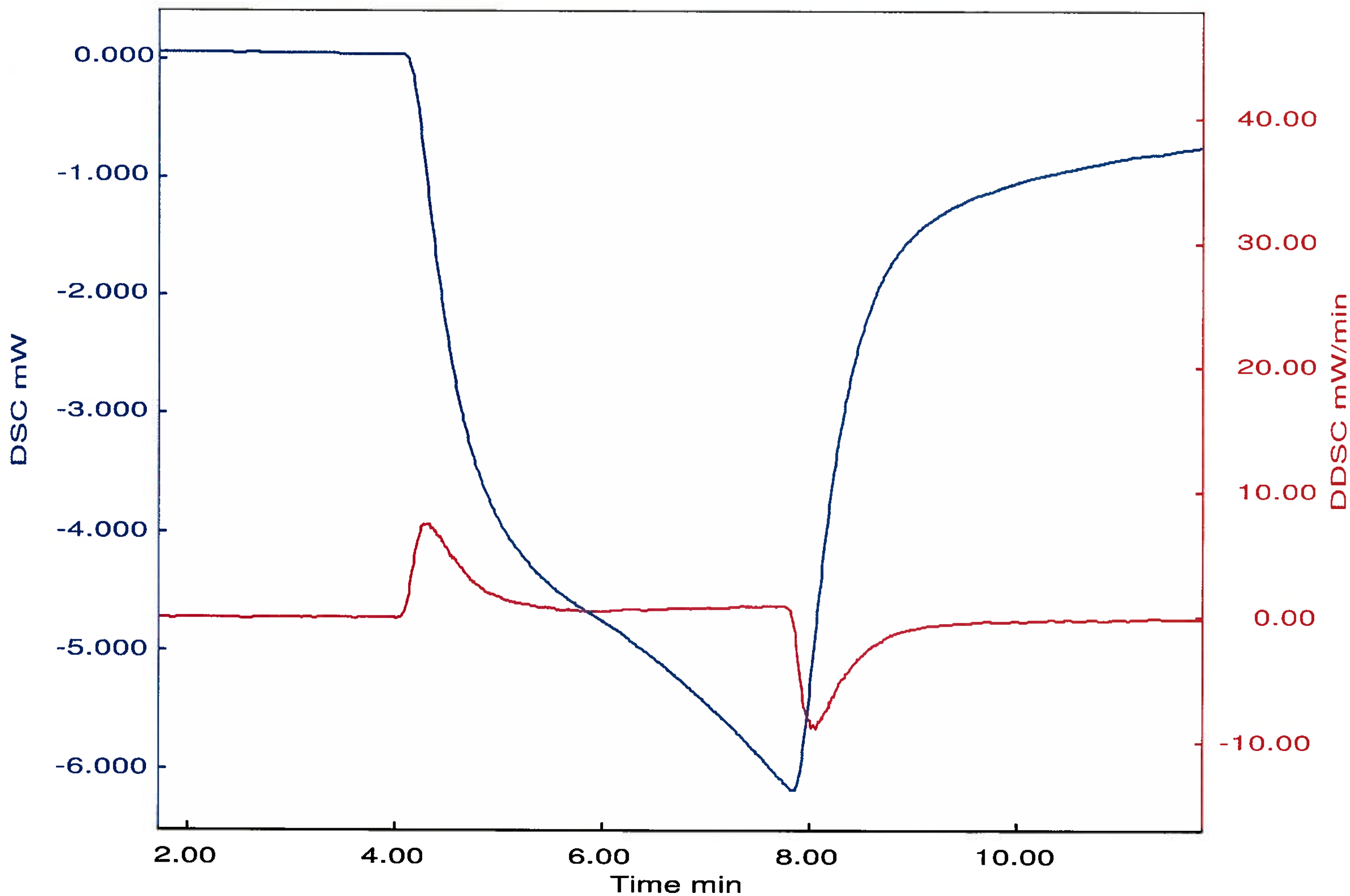
020039

<< DSC >>
Data Name:120316-486163-sub
Date: 12/ 3/16 17:55
Sample: 486163
9.68 mg
Reference:
0 mg

Temperature Program:

		[C]	[C/min]	[min]	[sec]
1*	25 -	25	10	4	0.2
2*	25 -	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

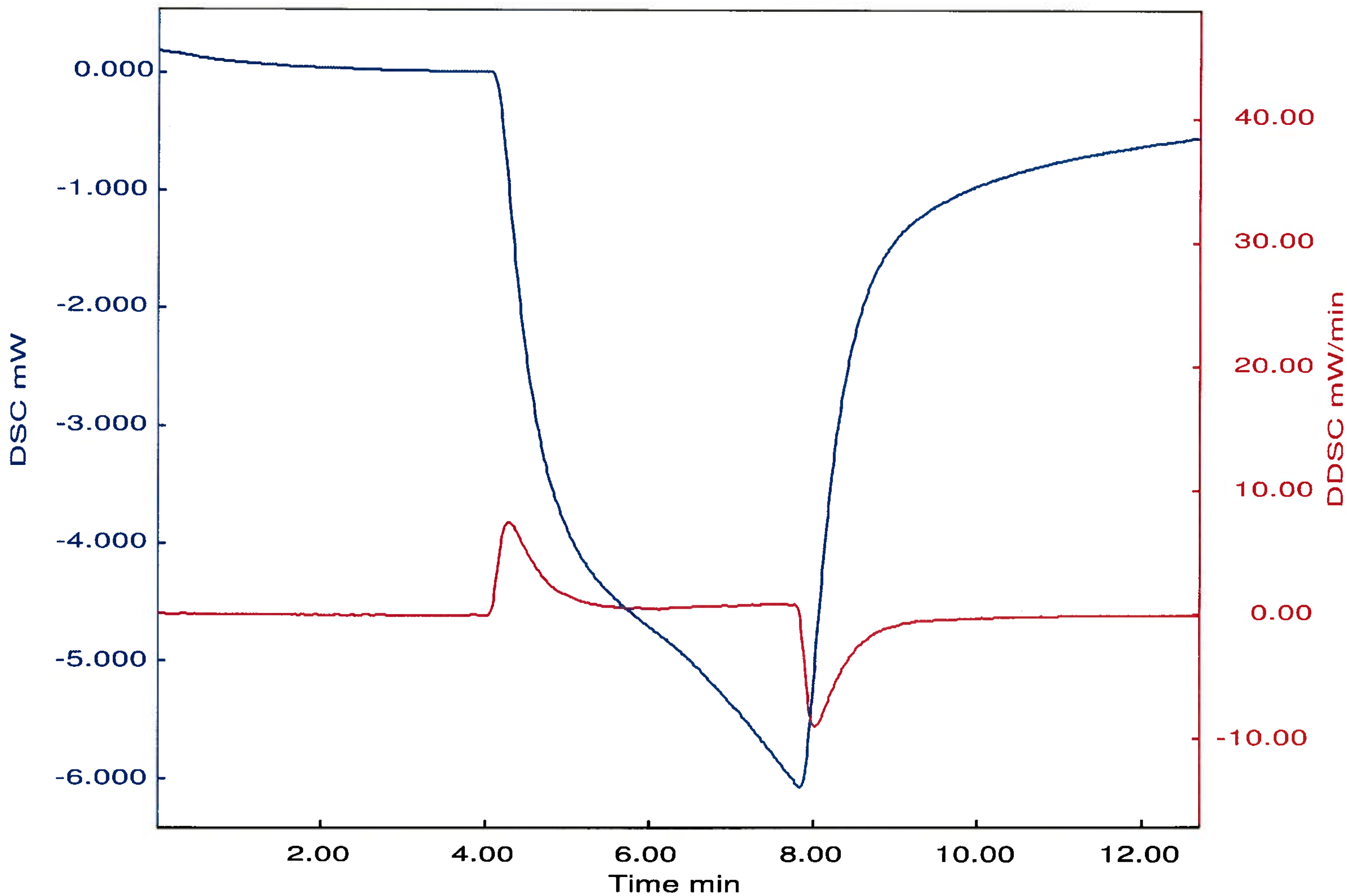
020040

<< DSC >>
Data Name:120316-486163-2
Date: 12/ 3/16 18:22
Sample: 486163-2
8.67 mg
Reference:
0 mg

Temperature Program:

			[C]	[C/min]	[min]	[sec]
1*	25	-	25	10	4	0.2
2*	25	-	100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

020041

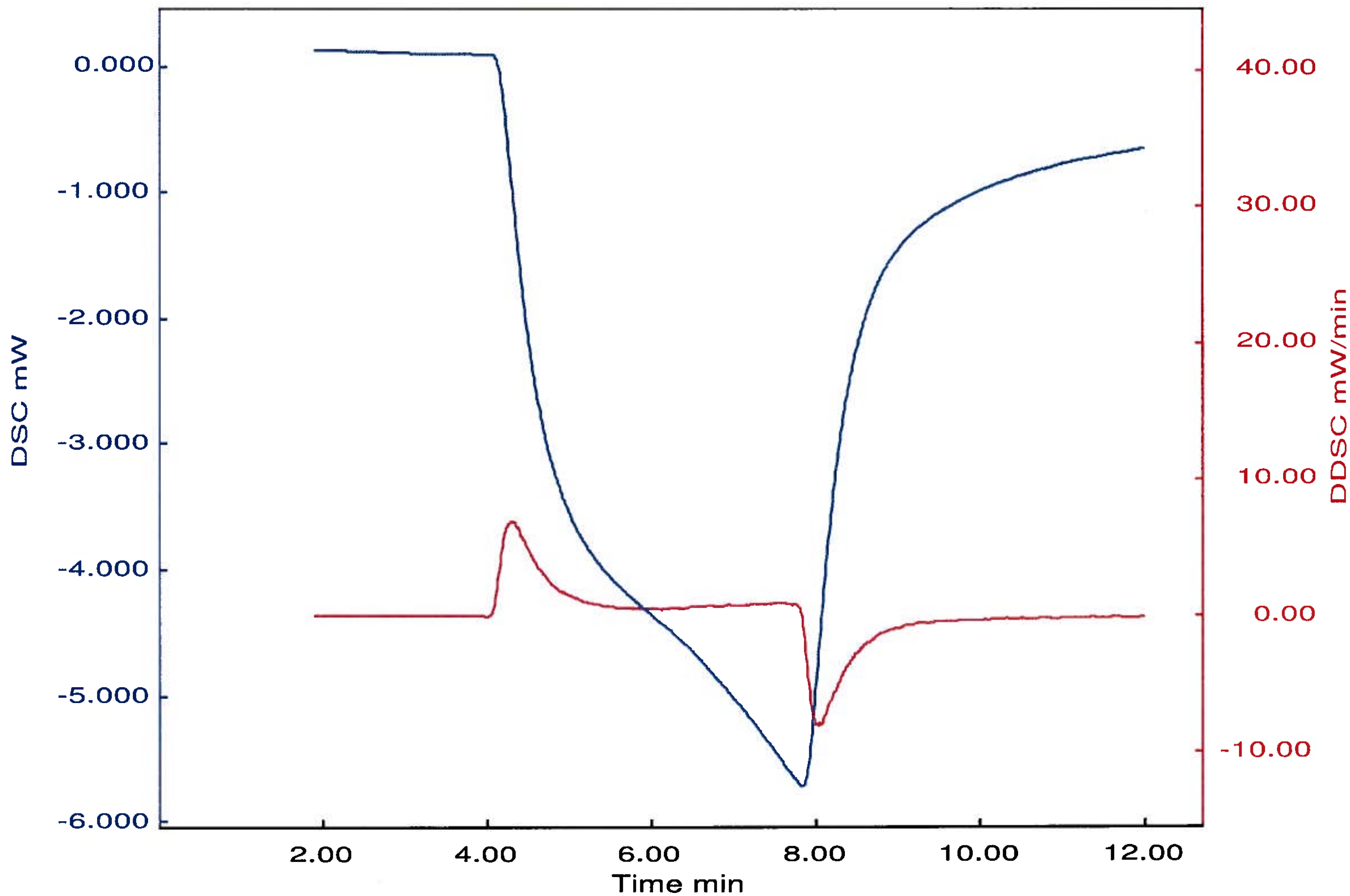
<< DSC >>

Data Name: 120316-486163-2sub
Date: 12/ 3/16 18:22
Sample: 486163-2
8.67 mg
Reference:
0 mg

Temperature Program:

	[C]	[C/min]	[min]	[sec]
1*	25 - 25	10	4	0.2
2*	25 - 100	20	5	0.2

Comments:
Operator rss
oval sensor, al open



SwRI

020042

SOUTHWEST RESEARCH INSTITUTE
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

TGA/DSC-FTIR Data

02 0044rev1
M 5.22.12

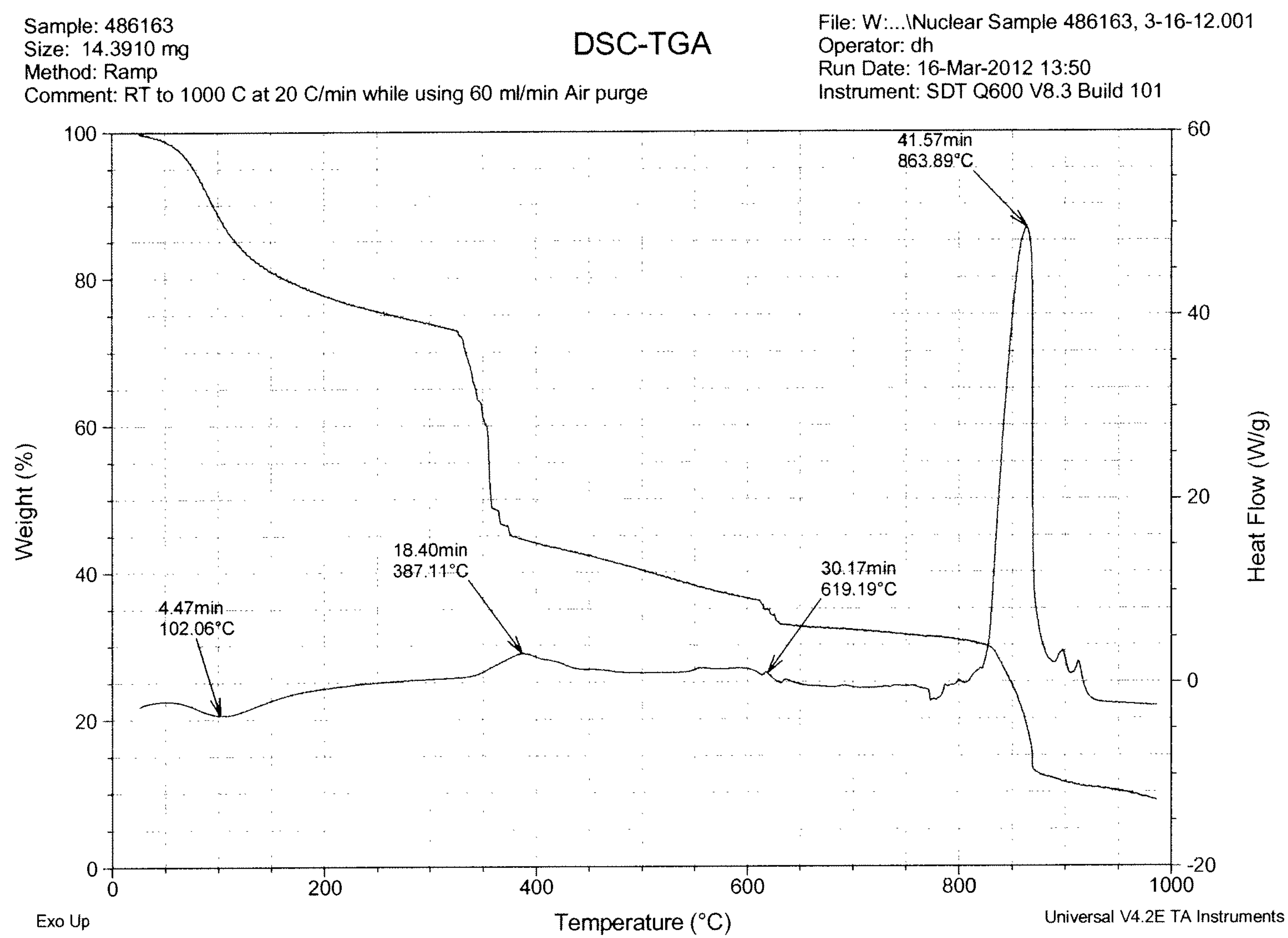


Figure 1. Weight loss curve with heat flow for Cs-loaded resorcinol resin.

020045rev1
M 5.22.12

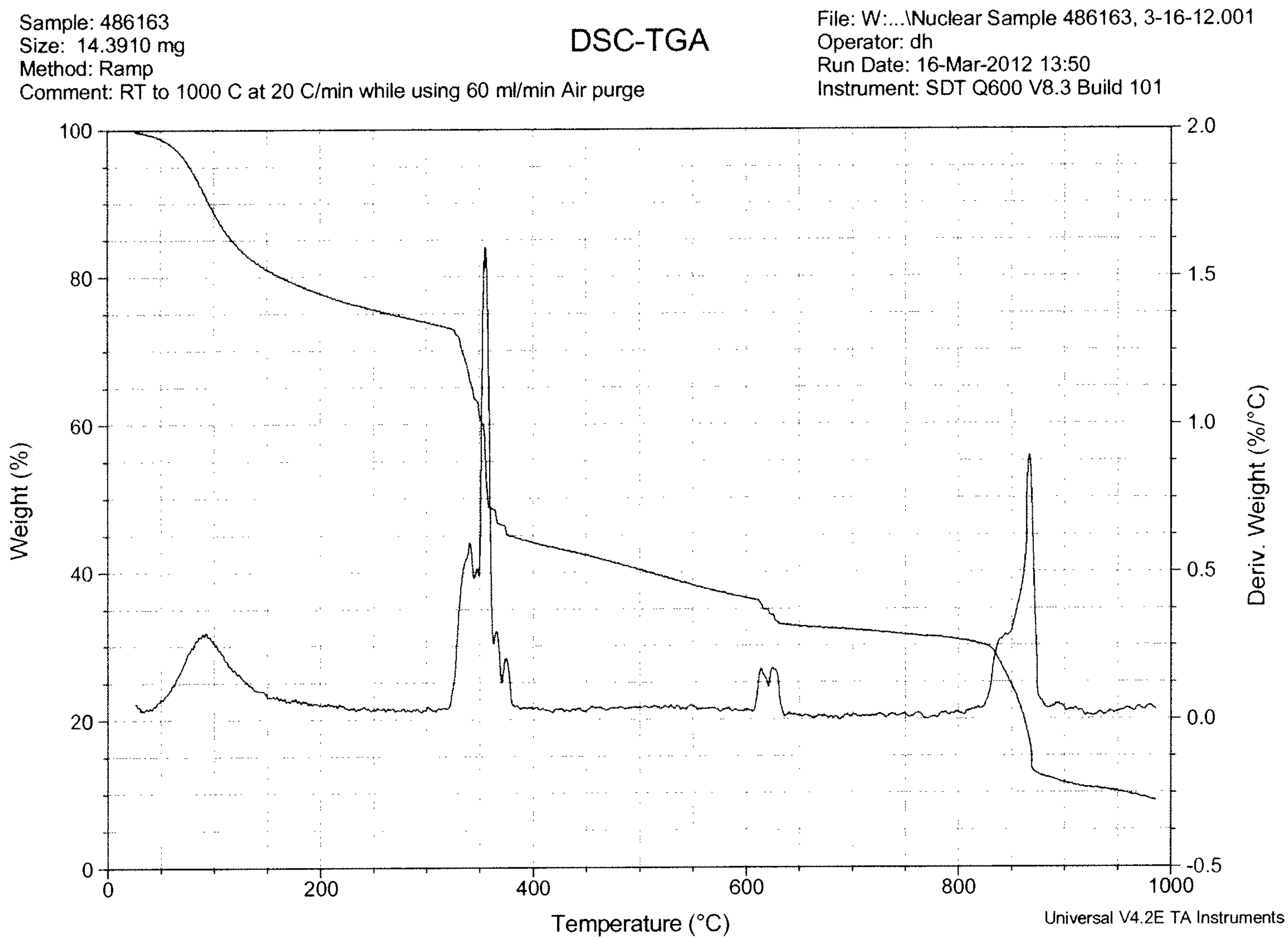


Figure 2. Weight loss curve and the derivative curve for Cs-loaded resorcinol resin.

020045A rev 1
MS 5.22.12

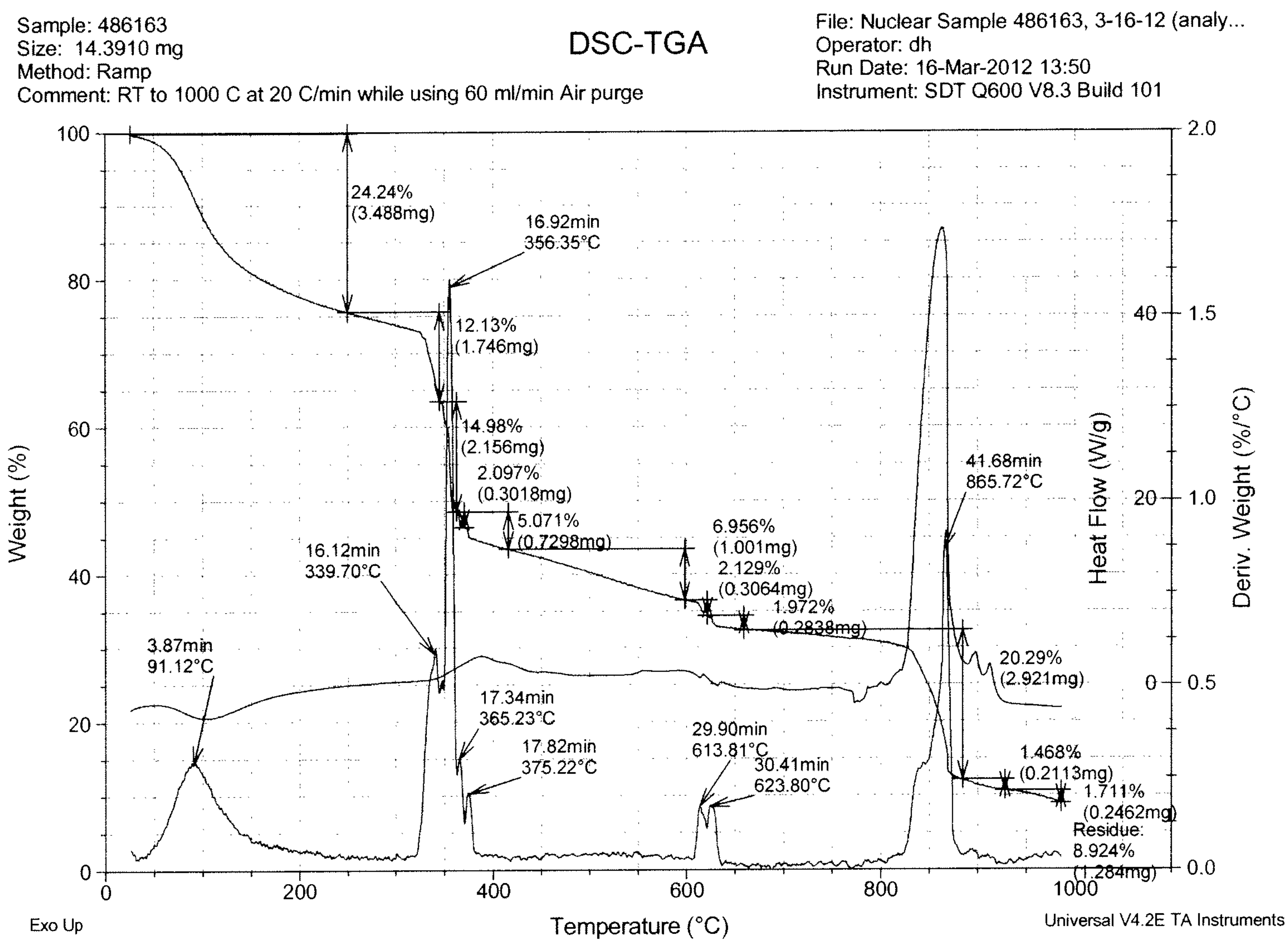


Figure 3. Weight loss curve with the derivative curve and heat flow curve included.

020045B rev1
M 5.22.12

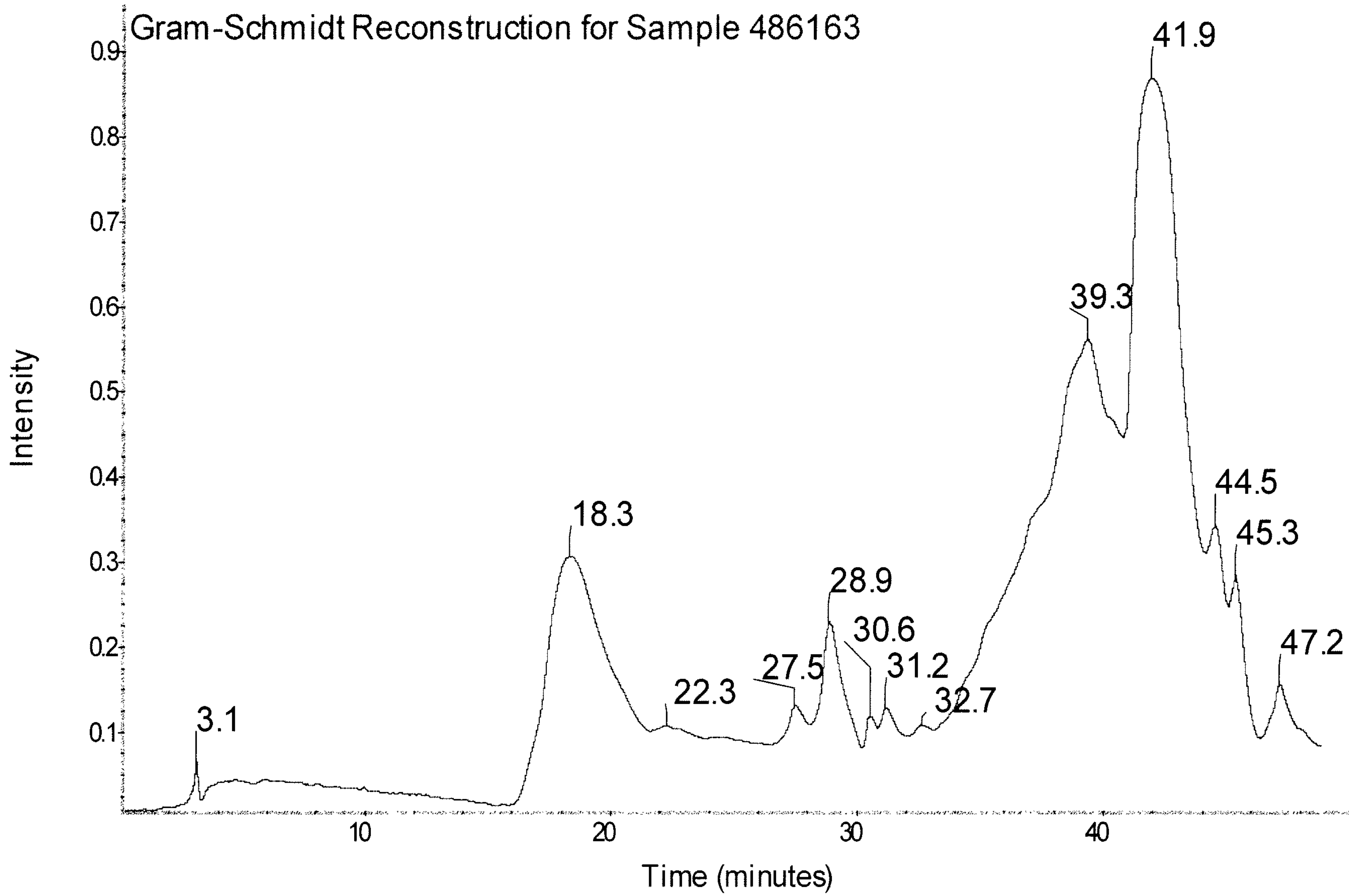


Figure 4. Gram-Schmidt Reconstruction for TGA/DSC analyis of the Cs-loaded resorcinol resin (see Figure 1)

020045C rev 1
MA 5.22.12

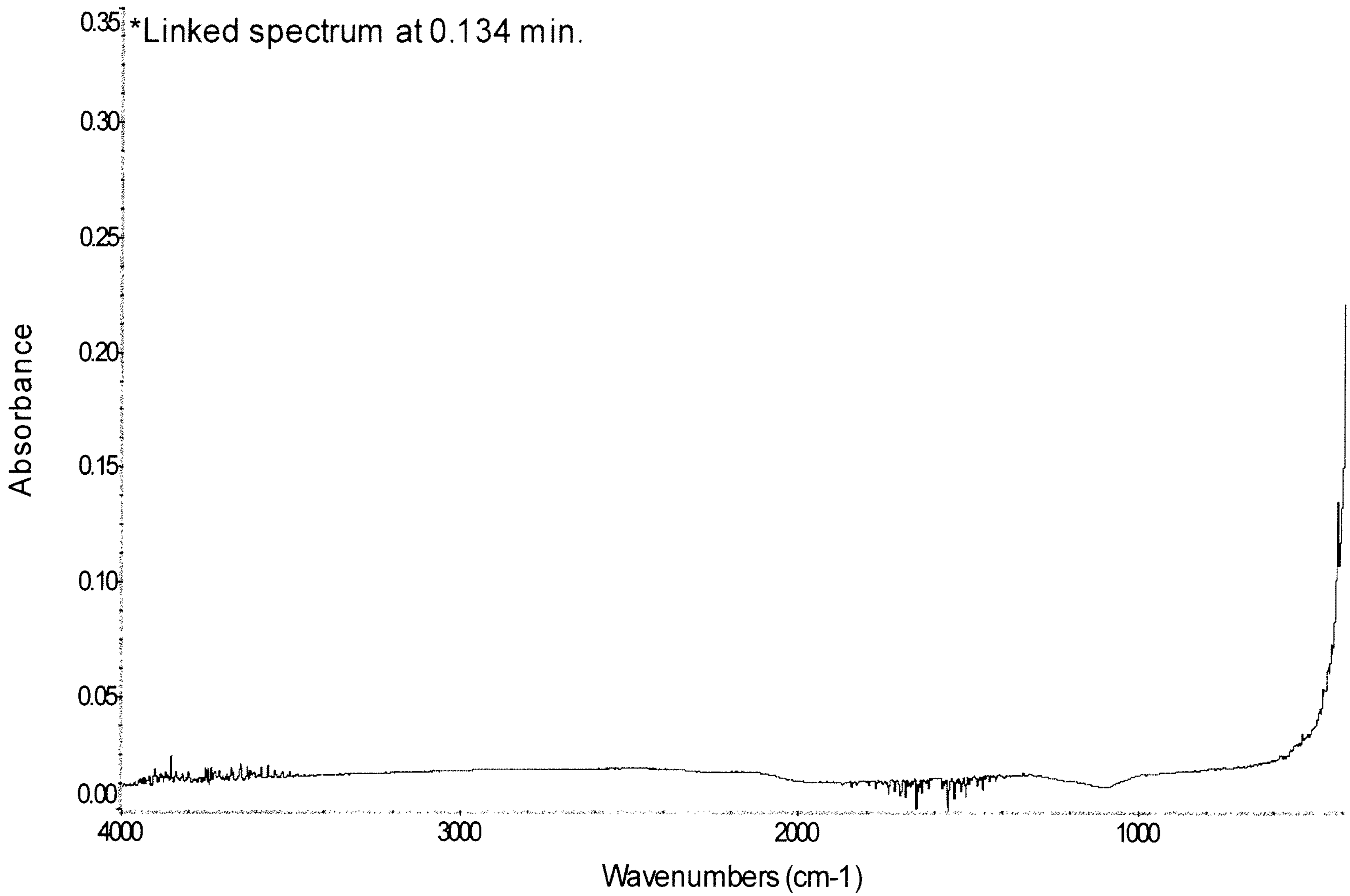


Figure 5. Initial FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin.

0200450 rev 1
M 5.22.12

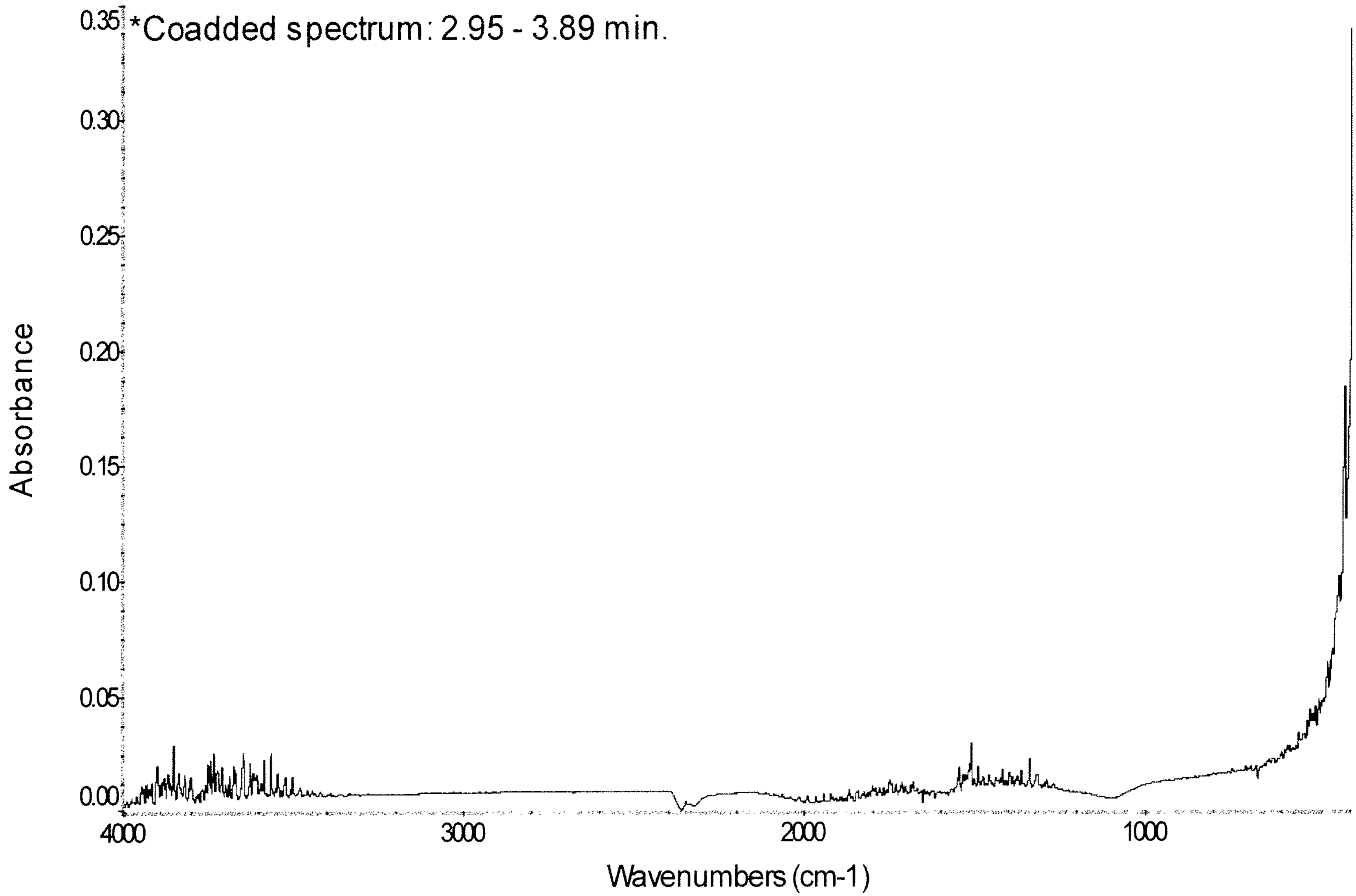


Figure 6. FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin at 2.95-3.89 min.

020045E rev 1
M 5.22.12

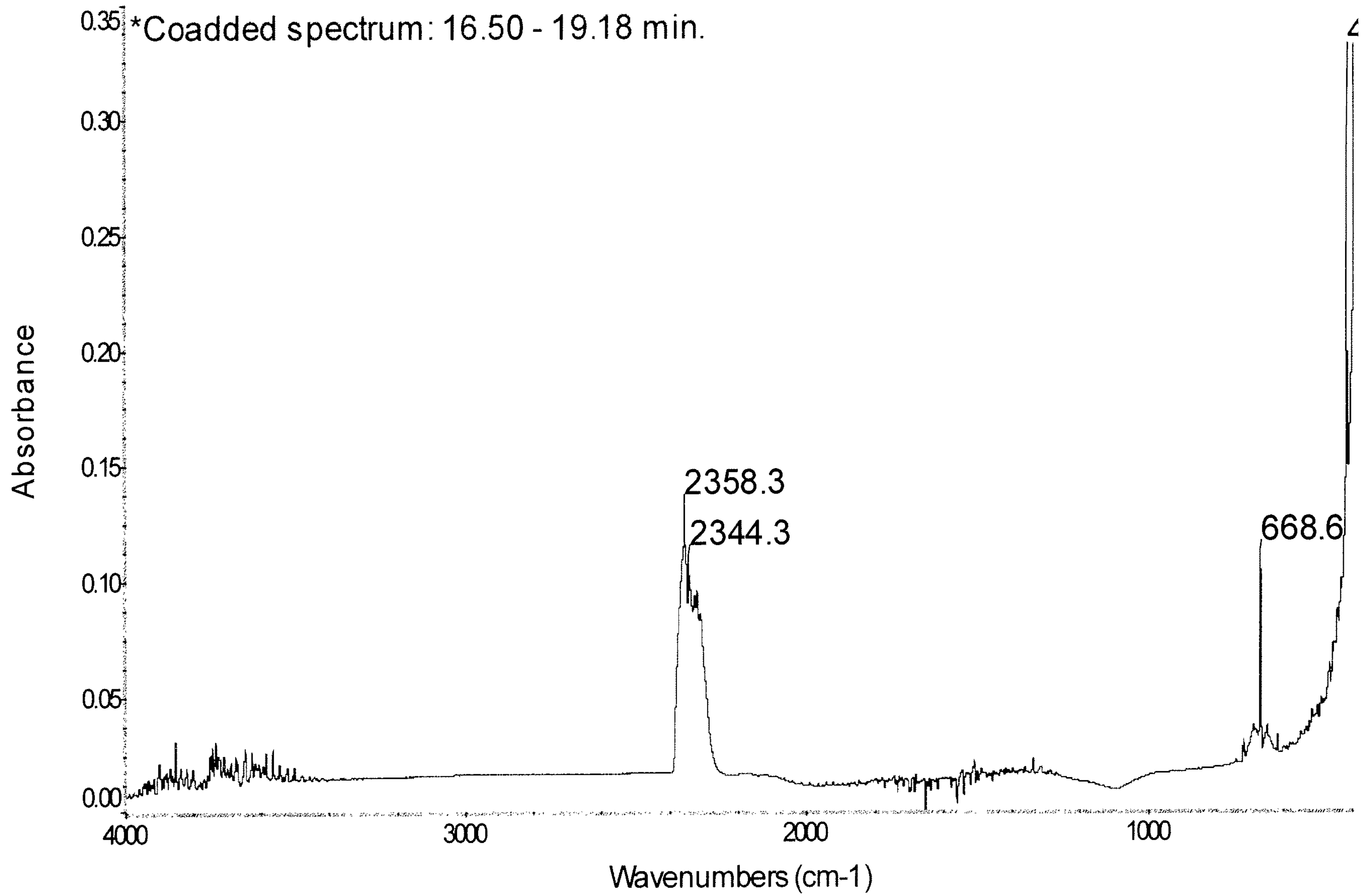


Figure 7. FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin at 16.50-19.80 min

020045F rev 1
AA 5.22.12

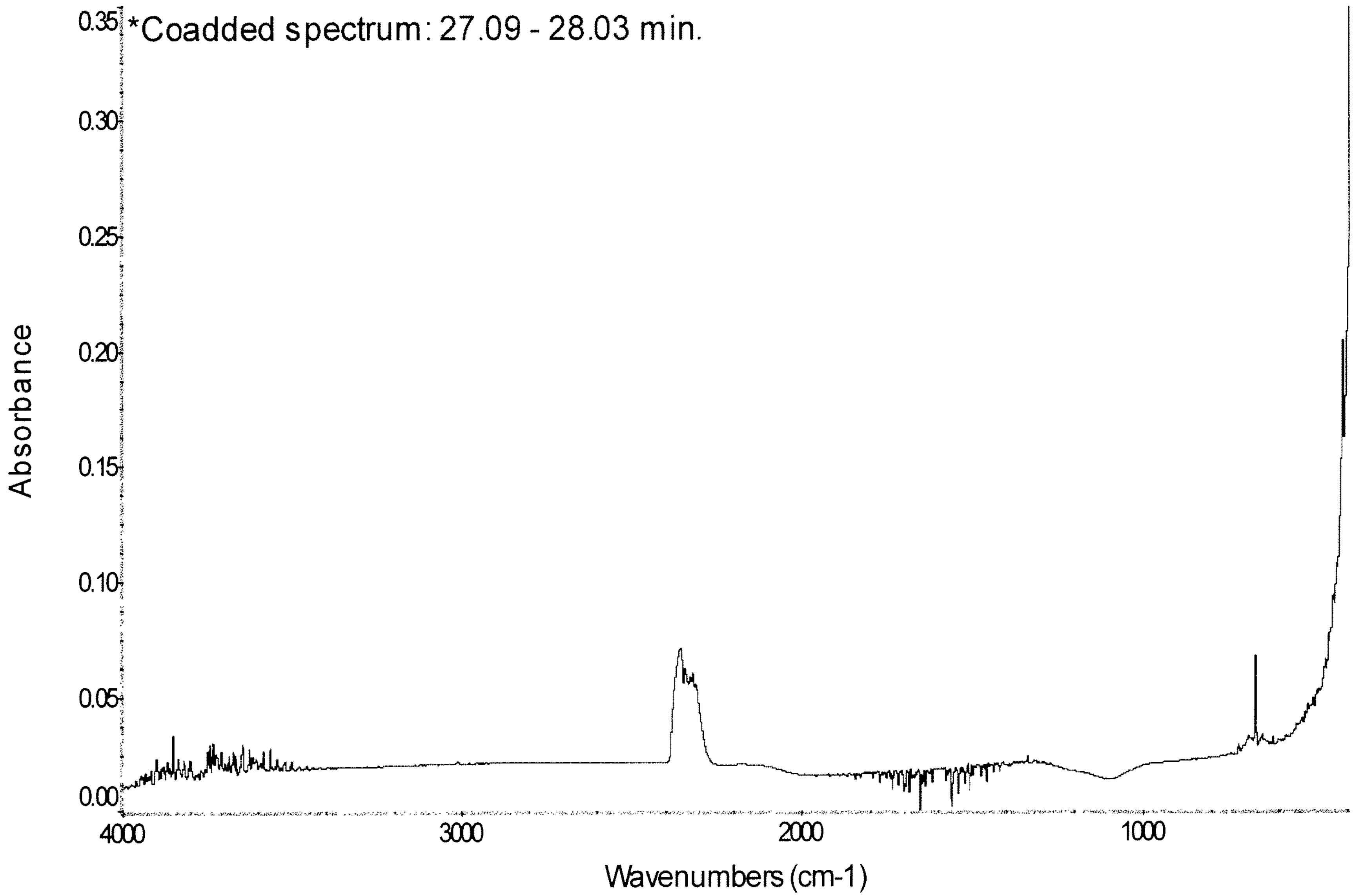


Figure 8. FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin at 27.09-28.03 min.

020045G rev1
AH 5.22.12

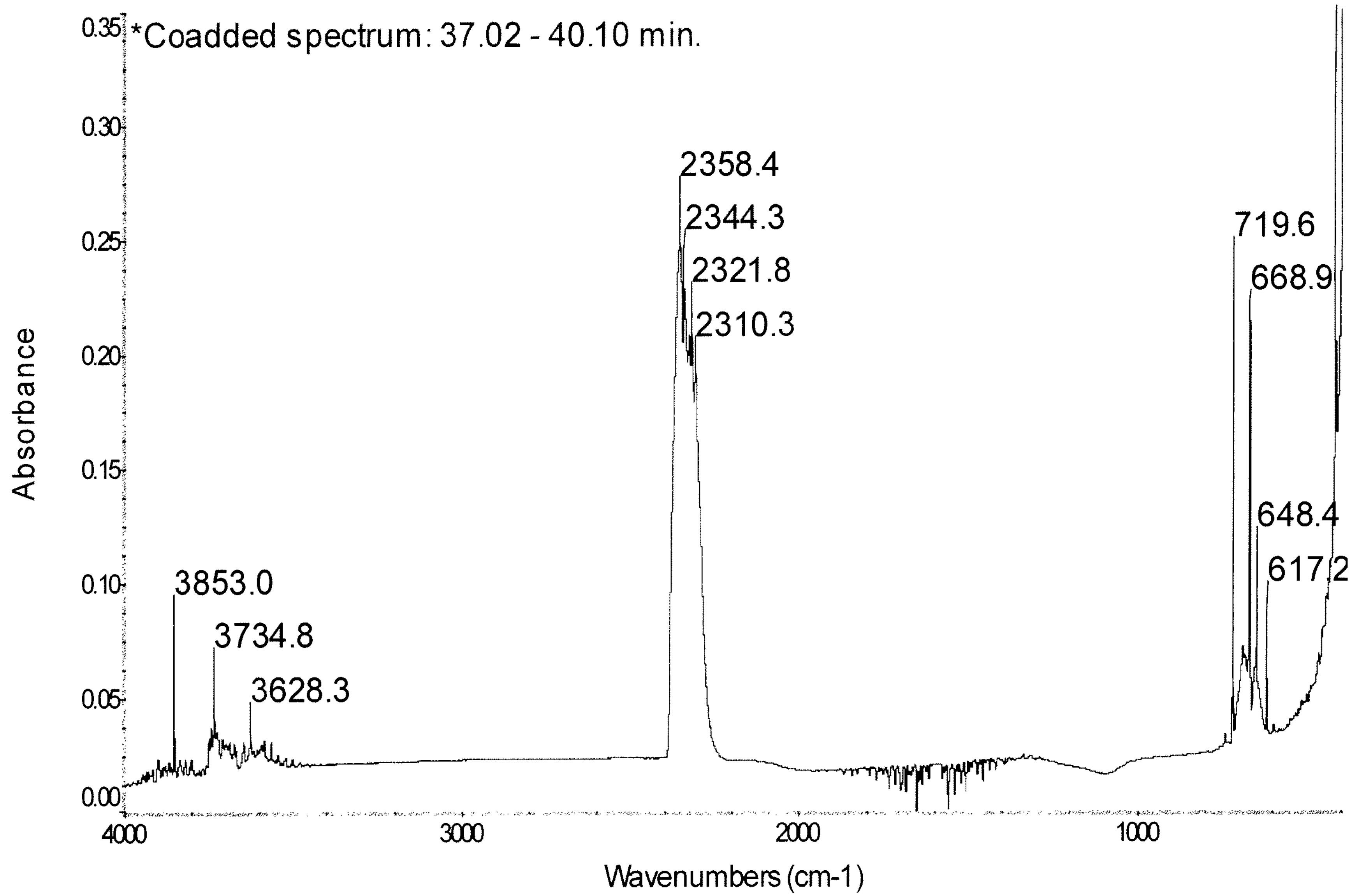


Figure 9. FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin at 37.02-40.10 min.

020045H rev 1
M 5.22.12

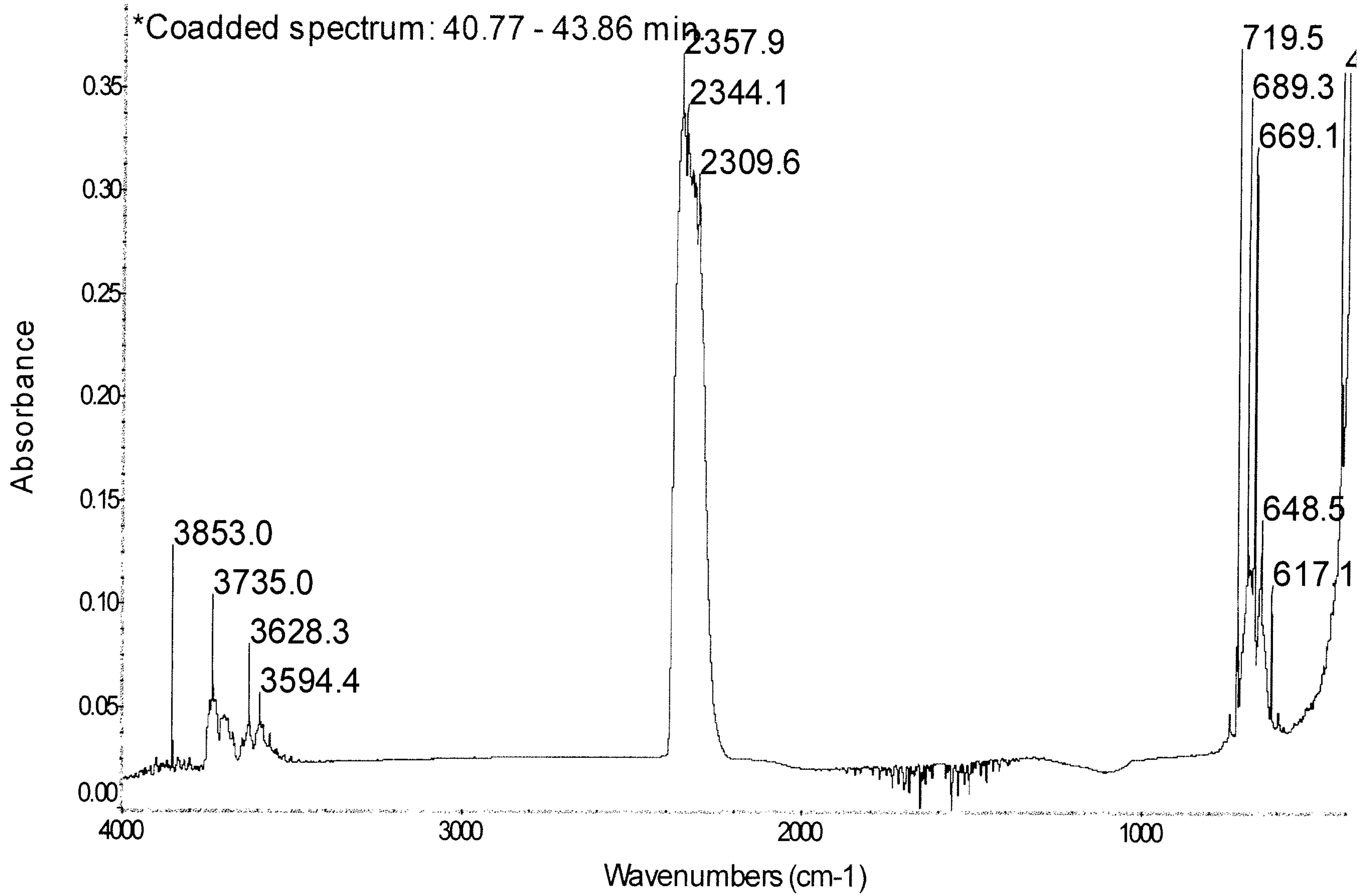


Figure 10. FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin at 40.77-43.86 min.

020045I rev 1
AA 5.22.12

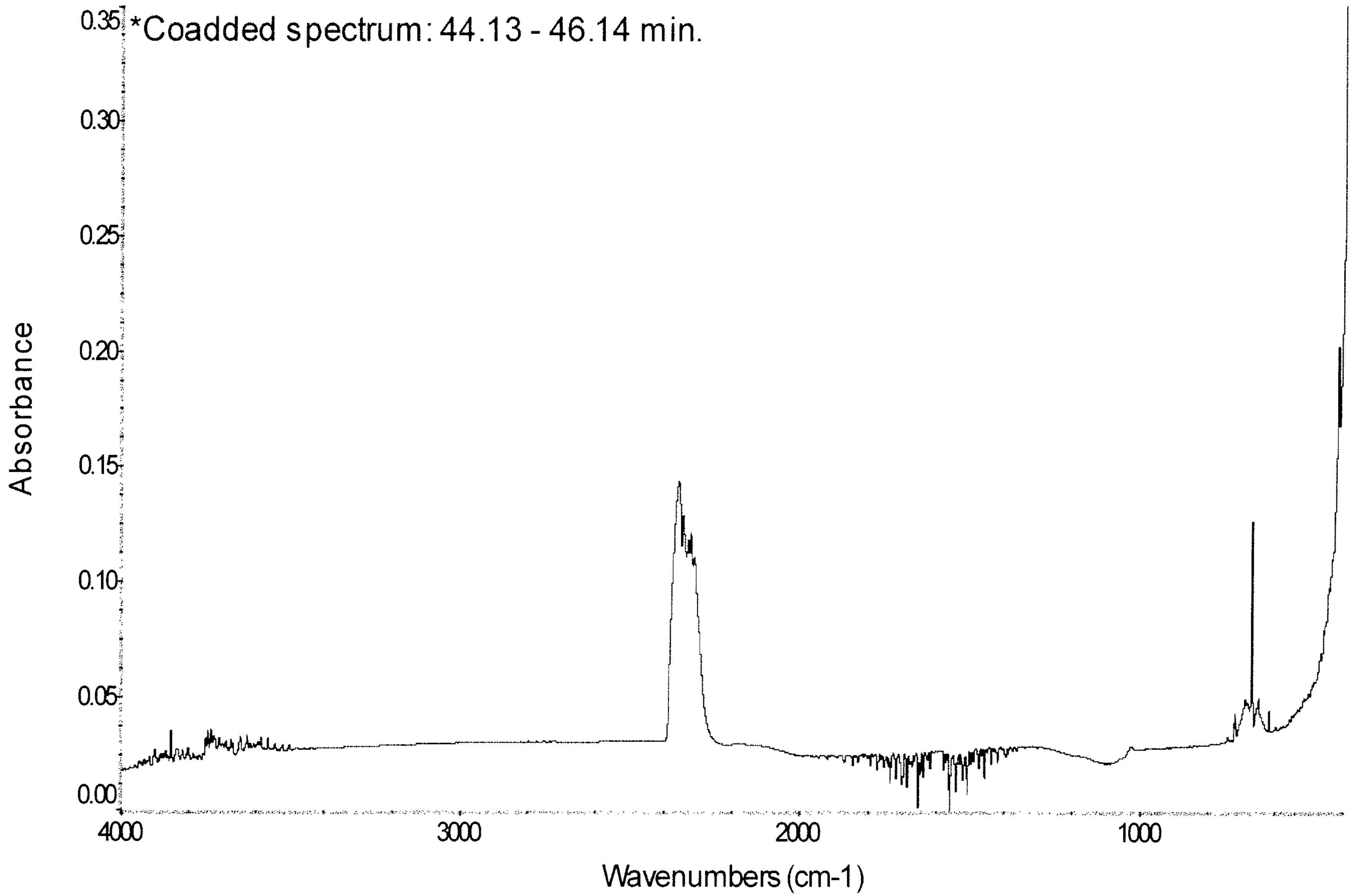


Figure 11. FTIR spectrum collected from TGA/DSC analyis of the Cs-loaded resorcinol resin at 44.13-46.14 min.

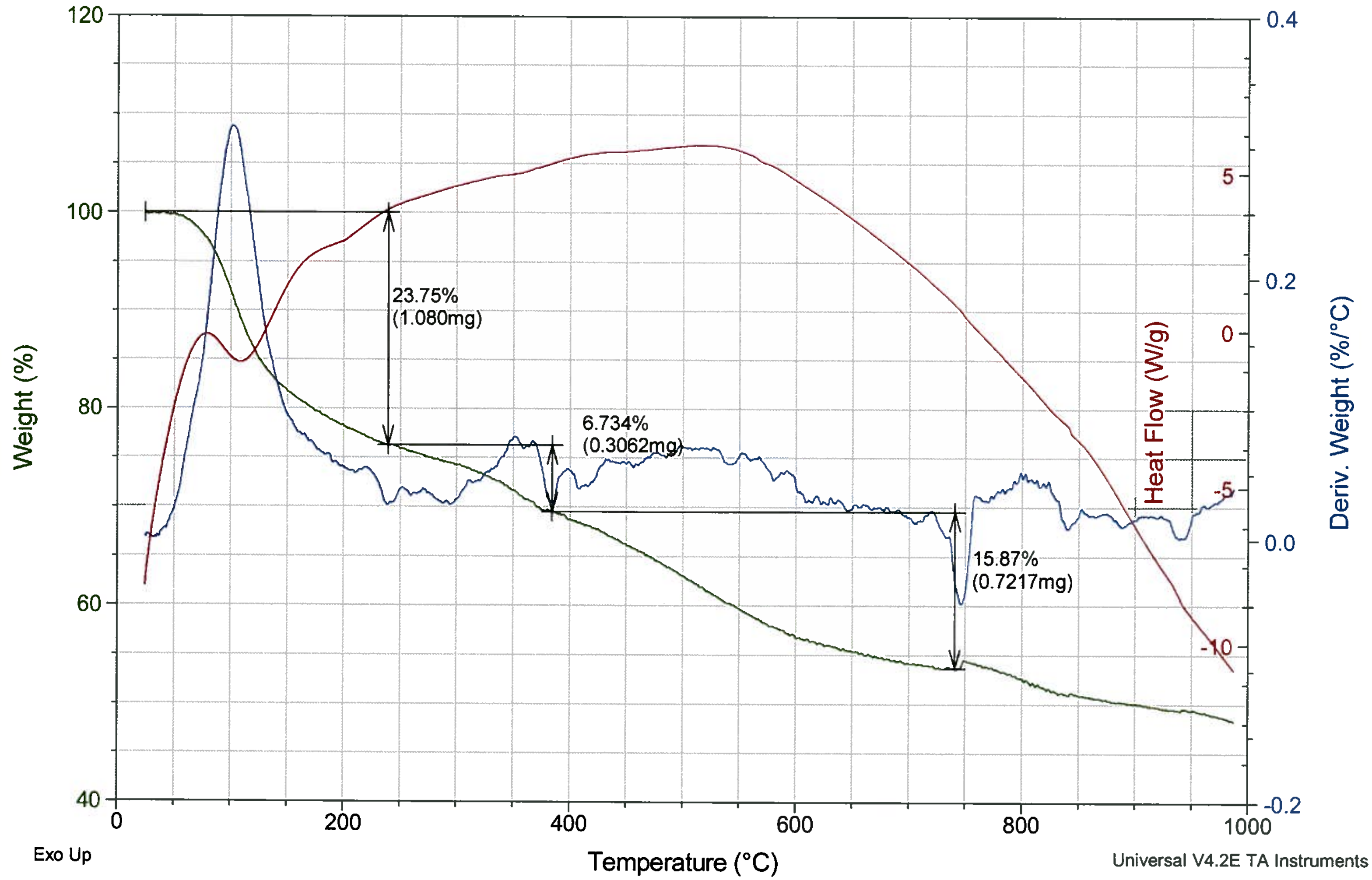
SOUTHWEST RESEARCH INSTITUTE
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

TGA/DSC-FTIR in N_2

Sample: 486163 (N2 using ceramic lids)
Size: 4.5470 mg
Method: Ramp
Comment: RT to 1000 C at 20 C/min using a 100 ml/min N2 purge

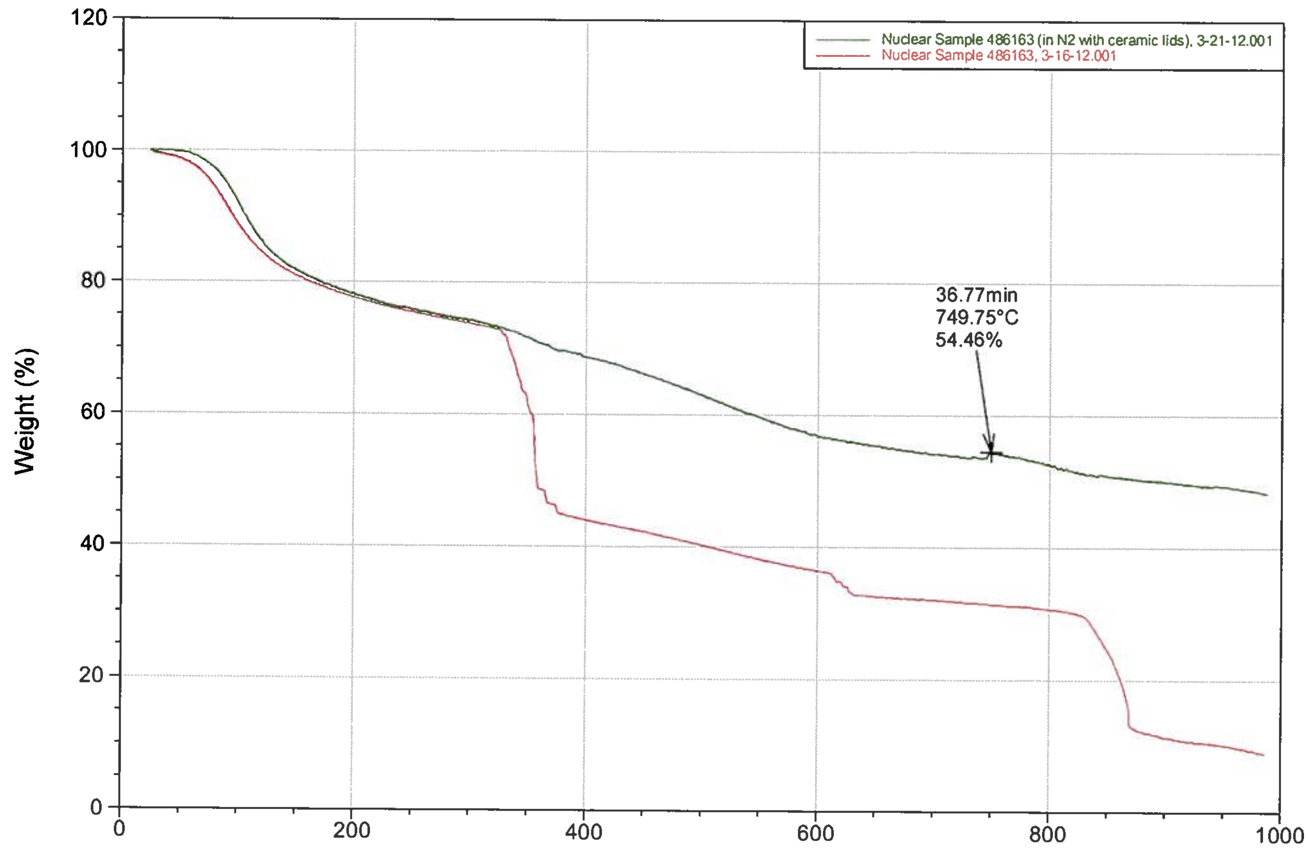
DSC-TGA

File: Nuclear Sample 486163 (in N2 with cer...
Operator: dh
Run Date: 21-Mar-2012 19:33
Instrument: SDT Q600 V8.3 Build 101



020047

Overlay of Sample 486163 run with air purge (TGA/FTIR) and run with nitrogen purge



020048

SOUTHWEST RESEARCH INSTITUTE
CLIENT: Battelle Memorial Ins. PNNL
TASK ORDER#: 120319-5
SRR#: 46112
SDG#: 486163
VTSR: 120319-5
PROJECT #: 13295.12.008

Logbook Copies

020050

91

TITLE BATTELLE RESORCINAL RESIN (CESIUM LOADING) SwRI® PROJECT NO. 13795.12.008

BOOK NO. 04-0406-036

Work continued from Page

REAGENTS: 1M NaOH - 40.0g OF NaOH ~~WAS ADDE~~ (INORG 8949) WAS ADDED TO 1.0L OF D.I. H_2O AND DISSOLVED. SOLUTION WAS ALLOWED TO COOL PRIOR TO USE. F.V. OF 1.0L OBTAINED. JM 02/13/12

1M NaOH - 40.0g OF NaOH (INORG 9603) WAS ADDED TO 1.0L OF D.I. H_2O AND DISSOLVED. SOLUTION WAS ALLOWED TO COOL PRIOR TO USE. A F.V. OF 1.0L OBTAINED. JM 02/13/12

0.5M HNO_3 - 32.0mL OF HNO_3 (INORG 9651) WAS ADDED TO 900mL OF D.I. H_2O AND BROUGHT TO A F.V. OF 1000mL WITH D.I. H_2O. JM 02/13/12

0.01M $CsNO_3$/1M NaOH - 1.95g $CsNO_3$ (INORG 1008) WAS ADDED TO 900mL OF 1M NaOH (40.0g NaOH [INORG 9651] PER 1.0L D.I. H_2O) AND DISSOLVED. BROUGHT TO A F.V. OF 1000mL USING SAME 1M NaOH JM 02/13/12

PROCEDURE: RESIN WAS ALIQUOTED INTO 4 EQUAL PARTS OF 250mL BED VOLUMES TO YIELD A TOTAL BED VOLUME OF 1000mL. EACH ALIQUOT WAS TREATED THE SAME AND USED THE SAME REAGENTS, AS DESCRIBED IN THE FOLLOWING METHOD (SEE ATTACHED): TABLE 1.1

Balance #19

RSS 3/13/12

Work continued to Page

SIGNATURE [signature] DATE 02/13/12

DISCLOSED TO AND UNDERSTOOD BY [signature] DATE WITNESS DATE 3/13/12

TITLE Specific Heat Capacity ASTM ~~D~~ E1269 (RSS RE 4/13/12)

PROJECT NO.

BOOK NO. 12-0406-013

SwRI®

Work continued from Page

Balance # 12

(1) Blk-2 file 120316 blk2 15:57 RE ~~20~~ 25°C – 100°C (RE 3/16/12)

(2) PE sapphire 483 (Perkin Elmer 0219 1483) Wt. 9.04 mg 16:33
file 120316-Sap 483-2 RE ~~20~~ 25°C → 100°C (RSS 3/16/12)

(3) NIST sapphire #720 (Inorg #9700) Wt. 10.46 mg 16:59
file: 120316 nist-2 25°C → 100°C

(4) 486163 - file 120316-486163 RE RSS 3/16/12 ~~20~~ 25 – 100°C
empty pan: 0.01452 g
+ sample: 0.02420 g
end 0.02419 g

(5) 486163 duplicate (↓ spilled some) file 120316-486163-2
sample + pan: 0.02319 g 17:55 25 → 100°C
0.02317 g

Used DSC sub Program
(2)-(1) = 120316-Sap 483-2sub
(3)-(1) = nist-2sub
(4)-(1) = 486163-sub
(5)-(1) = 486163-2sub

Used Cp Program

486163: Sample name 486163-sub
reference name ↓ sap 483-2sub
486163 dup: Sample name 486163-2sub
reference name ~~486163-2sub~~ Sap 483-2sub (RE RSS 3/16/12)
C.V. of sap 483: Sample name: 120316-sap 483-2sub
verification name: ~~sap 483-2sub~~ NIST-2sub (RE RSS 3/16/12)

Work continued to Page

SIGNATURE [signature] DATE 3/16/12

DISCLOSED TO AND UNDERSTOOD BY M. Mendoza DATE WITNESS DATE 4/20/12

Appendix B

Cesium Loading Procedure

Spherical Resorcinol Formaldehyde (SRF) Resin Preparations for Loading with Cesium

> Caution: It is important that the SRF resin not be allowed to dry. There is no potential for a safety hazard should this occur; however, the ion exchange performance of the SRF resin will be severely reduced and inaccurate data generated.

1. Determine the amount of SRF resin needed for experimental testing. Fill container, no more than $1/5^{th}$ full, with SRF resin. Use volume measurement for performing each step.

> Note: Given the relatively large volume of resin needed for testing, use a Büchner funnel to facilitate solution-resin contact.

2. The dry bed density of the SRF resin is approximately 0.456 g/mL. If the resin will be stored for a long period, bubble inert gas (e.g., Ar or N_2) over the resin for >30 min and seal the container.

3. Complete the "Bulk Pretreatment" portion of Table 1 (e.g., water rinses, resin expansion, and conversion) as follows:

   3.1 After adding the required solution volumes into the container, gently swirl by hand every 10 min during the specified duration. Alternatively, the analyst may choose to allow solution contact with the resin in a Büchner funnel.

   3.2 Decant the solutions to a waste container and retain the resin in the beaker. Alternatively, the analyst may choose to allow solution contact with the resin in a Büchner funnel, pulling the solution slowly through the resin with vacuum. Use care not to allow the resin to fully dry between solution contacts.

   3.3 Record each of the start/stop times, volumes, and solutions used in the space provided in Table 2.

   3.4 After completing the bulk pretreatment steps, transfer the resin to the testing vessel.

Table 1. Ion Exchanger Pretreatment and Process Steps

Process/Pretreatment Step	Solution	Volume	Time	Mixing	Flowrate
Bulk Pretreatment					
Water Rinse	DI Water	3 RV[(a)]	30 min	Swirl[(b)]	NA[(c)]
Resin Expansion – 1st	1 M NaOH	3 RV	30 min	Swirl	NA
Resin Expansion – 2nd	1 M NaOH	3 RV	>4 h	Soak	NA
Water Rinse – 1st	DI Water	3RV	30 min	Swirl	NA
Water Rinse – 2nd	DI Water	3RV	30 min	Swirl	NA
Water Rinse – 3rd	DI Water	3RV	30 min	Swirl	NA
Resin Conversion – 1st	0.5 M HNO_3	3 RV	30 min	Swirl	NA
Resin Conversion – 2nd	0.5 M HNO_3	3 RV	30 min	Swirl	NA
Resin Conversion – 3rd	0.5 M HNO_3	3 RV	30 min	Swirl	NA
Water Rinse – 4th	DI Water	3 RV	1 min	Swirl	NA
Resin Expansion	1 M NaOH	3 RV	1 h	Swirl	NA
Cesium Loading[(d)]	+0.01 M $CsNO_3$	3 RV	1 h	Swirl	NA
Water Rinse – 5th	DI Water	10 RV	1 min	Swirl	NA

(a) Resin volume (RV).
(b) Gently swirling by hand every 10 min.
(c) Not applicable (NA). Alternatively, the analyst may choose to use a Büchner funnel and flow the solutions slowly through the resin bed at less than 6 RV/hr.
(d) Cesium loading should occur using a solution of 1 M NaOH plus 0.01 M $CsNO_3$.

Note: Table 2 provides the analyst with the option of preconditioning up to five resin samples all at the same time or up to two different times. If additional preconditioning is needed, the analyst may photocopy the table for additional cycles or record the data directly into the LRB.

Table 2. Example Data Sheet for Bulk Ion Exchanger Pretreatment

Bulk Pretreatment	Solution Used	Samples Processed? 1st 2nd 3rd 4th 5th	Volume (mL)	Start Time	Stop Time	Step Duration
Water Rinse	DI Water					
Resin Expansion – 1st	1 M NaOH					
Resin Expansion – 2nd	1 M NaOH					
Water Rinse – 1st	DI Water					
Water Rinse – 2nd	DI Water					
Water Rinse – 3rd	DI Water					
Resin Conversion – 1st	0.5 M HNO_3					
Resin Conversion – 2nd	0.5 M HNO_3					
Resin Conversion – 3rd	0.5 M HNO_3					
Water Rinse – 4th	DI Water					
Resin Expansion	1 M NaOH					
Cesium Loading	+0.01 M $CsNO_3$					
Water Rinse – 5th	DI Water					
Bulk Pretreatment	**Solution Used**	**Samples Processed? 1st 2nd 3rd 4th 5th**	**Volume (mL)**	**Start Time**	**Stop Time**	**Step Duration**
Water Rinse	DI Water					
Resin Expansion – 1st	1 M NaOH					
Resin Expansion – 2nd	1 M NaOH					
Water Rinse – 1st	DI Water					
Water Rinse – 2nd	DI Water					
Water Rinse – 3rd	DI Water					
Resin Conversion – 1st	0.5 M HNO_3					
Resin Conversion – 2nd	0.5 M HNO_3					
Resin Conversion – 3rd	0.5 M HNO_3					
Water Rinse – 4th	DI Water					
Resin Expansion	1 M NaOH					
Cesium Loading	+0.01 M $CsNO_3$					
Water Rinse – 5th	DI Water					

Observations:

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