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New Jersey 08405

# Characterization of the Flammability and Thermal Decomposition Properties of Aircraft Skin Composite Materials and Combustible Surrogates

March 2014

Final Report

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16. Abstract


Currently, the aircraft industry is shifting toward the use of new aircraft skin materials. In place of aluminum, aircraft are now being constructed from composite materials, which typically include combustible components. The objective of this test series was to quantify the small-scale burn characteristics of two new aircraft skin composite materials and a candidate wood surrogate. If testing was successful, a wood surrogate could be used as a readily available, cost-effective in future large-scale flammability and suppression tests.

A series of small-scale fire tests and analytical test methods were conducted to characterize the flammability and thermal decomposition properties of the materials. These tests were designed to develop a data set that could be used to validate intermediate scale tests and as input in the development of flame spread and thermal decomposition models for these materials. The composite materials evaluated were a carbon fiber-reinforced polymer (CFRP) and a glass laminate aluminum reinforced epoxy (GLARE), and the wood surrogate evaluated was an oriented strand board (OSB). The small-scale fire tests conducted in this research included cone calorimetry testing, lateral ignition and flame spread testing, and thermal decomposition testing. The analytical work conducted included thermogravimetric analysis, differential scanning calorimetry, and pyrolysis gas chromatograph/mass spectroscopy. The materials' flammability and thermal decomposition properties were derived from these tests.

Tests suggest the average heat capacity during decomposition and the average thermal conductivities of the two materials is similar. This indicates the OSB is a reasonable surrogate for the CFRP over the course of the decomposition process when the heat capacity and thermal conductivity parameters strongly influence the results. The overall average apparent heat capacity is comparable, which is consistent with the heat capacity results of the thermal decomposition apparatus. This suggests the OSB is a reasonable overall surrogate material for the CFRP when the heat capacity is a significant parameter, but there may be significant differences in thermal absorption rates on smaller time scales.


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## LIST OF ACRONYMS

| | |
|---|---|
| CFRP | Carbon fiber-reinforced polymer |
| DSC | Differential scanning calorimetry |
| GLARE | Glass laminate aluminum reinforced epoxy |
| LIFT | Lateral ignition and flame spread test |
| OSB | Oriented strand board |
| PY-GC/MS | Pyrolysis gas chromatograph/mass spectroscopy |
| TDA | Thermal decomposition apparatus |
| TGA | Thermogravimetric analysis |

# EXECUTIVE SUMMARY

Currently, the aircraft industry is trending toward the use of new aircraft skin materials. In place of aluminum, aircraft are now being constructed from composite materials, which include combustible components. The objective of the test series described in this report was to quantify the small-scale burn characteristics of two new aircraft skin composite materials and a candidate wood surrogate. If supported by the analysis, a wood surrogate could be used as a readily available, cost-effective material in future large-scale flammability and suppression tests. The small-scale tests support an overall objective to determine if additional firefighting agent is required should the composite skin material become involved in a fire.

A series of small-scale fire tests and analytical test methods were conducted to characterize the flammability and thermal decomposition properties of the materials. These tests were designed to develop a data set that could be used as validation for intermediate scale tests and as input in the development of flame spread and thermal decomposition models for these materials. The composite materials evaluated were a carbon fiber-reinforced polymer (CFRP) laminate and a glass laminate aluminum reinforced epoxy (GLARE), and the wood surrogate evaluated was an oriented strand board (OSB).

The small-scale fire tests conducted in this research included cone calorimetry, lateral ignition and flame spread testing (LIFT), and thermal decomposition testing using a unique thermal decomposition apparatus (TDA). The flammability properties of all three materials were developed using the cone calorimeter and LIFT apparatus. However, thermal decomposition properties were only developed for the OSB and CFRP materials during this period of the study. In addition, the results of the LIFT on the CFRP and GLARE materials was inconclusive due to intermittent flame spread and bowing at the joints of the material samples. It is expected that if the LIFT procedure was conducted at higher heat fluxes, it would yield flame spread parameters that could be used to assess the potential of the OSB to function as a surrogate test material. The TDA results were used to determine thermal decomposition properties for the CFRP and GLARE materials. These thermal properties were derived using inverse heat transfer analysis and decomposition modeling.

The analytical test work included thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and pyrolysis gas chromatograph/mass spectroscopy (PY-GC/MS) to derive the materials' flammability and thermal decomposition properties. The TGA results were used to characterize the changes that occur in material density as a function of temperature. The DSC results were used to approximate the apparent specific heat capacities and heat of decomposition for the OSB and CFRP materials. The PY-GC/MS test results were intended to be used to approximate the gas enthalpies for the combustion gases that escape the material during decomposition; however, this analysis is planned during the continuation of this work.

Overall, the test results reported for the cone calorimeter, LIFT, and thermal decomposition testing of the OSB samples were comparable to published data for similar products. A comparison of the OSB cone calorimeter test results to the CFRP and GLARE cone calorimeter tests revealed that the OSB and CFRP have similar ignition characteristics, but the GLARE material has a significantly higher ignition temperature and critical heat flux. This suggests that OSB is not well suited as a surrogate test material for the GLARE, when the critical heat flux

and ignition temperature have a strong influence on the test results. This is not the case for the OSB and CFRP materials, although it is noted that the OSB has a somewhat lower critical heat flux and ignition temperature and, thus, is easier to ignite than the CFRP.

The thermal decomposition tests of the composites were only conducted for the CFRP material given the cone calorimeter test results and the complexity associated in working with the GLARE material. A comparison of the OSB and CFRP TDA and TGA data indicates some differences in the decomposition and thermal properties of the two materials. First, the OSB begins to decompose at a temperature that is 50°C lower than the CFRP. This is consistent with the slightly lower ignition temperature and critical ignition heat flux for the OSB material as compared to the CFRP material observed in the cone calorimeter tests. A second difference revealed in the TDA tests is that the OSB material decreases in thickness during decomposition whereas the CFRP material increases in thickness. It is interesting that the final thickness of the two materials is within 2 mm. The TDA tests also revealed that the heat capacity of the OSB decreases during decomposition but the heat capacity of the CFRP increases during decomposition. The average heat capacity during decomposition and the average thermal conductivities of the two materials is similar. This indicates the OSB is a reasonable surrogate for the CFRP over the course of the decomposition process where the heat capacity and thermal conductivity parameters strongly influence the results.

The DSC revealed that there are significant differences in the apparent specific heat of the OSB and CFRP by as much as a factor of four. These differences are based on a correction for the actual mass using mass loss data determined in the TGA at the same heating rate. The overall average apparent heat capacity is comparable, which is consistent with the TDA heat capacity results. This suggests the OSB is a reasonable overall surrogate material for the CFRP when the heat capacity is a significant parameter, but there may be significant differences in thermal absorption rates on smaller time scales.

## 1. INTRODUCTION.

Currently, the aircraft industry is shifting toward the use of new aircraft skin materials. In place of aluminum, aircraft are now being constructed from composite materials, which include combustible components. The objective of this test series was to quantify the small-scale burn characteristics of two new aircraft skin composite materials and a candidate wood surrogate. If testing proved successful, a wood surrogate could be used as a readily available, cost-effective material in future large-scale flammability and suppression tests. The small-scale tests support an overall objective to determine if additional firefighting agent is required should the composite material be exposed to an external fire.

This report describes the methodology and results of a study undertaken for the Federal Aviation Administration (FAA) to determine the flammability and thermal decomposition properties of the materials. The composites evaluated were a carbon fiber-reinforced polymer (CFRP) laminate and a glass laminate aluminum reinforced epoxy (GLARE), and the wood surrogate evaluated was oriented strand board (OSB). The flammability and thermal decomposition properties provided in this report were determined using standardized bench-scale fire test methods and established data analysis routines. The following small-scale tests were performed: cone calorimeter testing [1], lateral ignition flame spread testing (LIFT) [2], and thermal decomposition apparatus (TDA) testing. A summary of the test method, test procedures, and bench-scale results are presented in the following sections.

Concurrent to this small-scale experimental work, a series of intermediate-scale fire tests were conducted to evaluate the flammability and suppressibility of the CFRP and wood surrogate materials. Those findings will be provided in a separate report. Both of these experimental efforts contributed to flame-spread and fire growth modeling efforts designed to assess the applicability of the intermediate-scale results to full-scale fire scenarios.

## 2. OBJECTIVES.

The primary objective of the small-scale testing was to determine the flammability and thermal decomposition properties for each of the three materials being evaluated. The characterization of these properties was required to appropriately exercise both the fire and thermal decomposition models. In general, the flammability properties required for fire model input are direct outputs from the results of the various bench-scale fire tests. Within the flame-spread model, these outputs are used to dictate the rate of heat release and burning duration of the material under varying incident heat flux exposures as well as the rate of flame spread over the surface of the material. To develop the thermal properties required for decomposition modeling input, the empirical data collected required inverse heat transfer analysis of both the thermal and gravimetric response of small samples when exposed to heat. These data included thermal conductivity, specific heat capacity, and density as a function of temperature as well as heat of decomposition and Arrhenius kinetic decomposition constants. These inputs, when incorporated into the thermal decomposition model, are used to predict thermal penetration into the material as well as predict the physical changes occurring within the material as it is heated. These properties were developed for OSB, CFRP, and GLARE. It should be noted that due to the complexity of the GLARE composite and the limited time frame for experimental testing, the thermal decomposition properties of this material were not determined. Only flammability data

for this material is presented in this work. The thermal decomposition product's raw data is available from Hughes Associates, Inc, and documented for future analysis.

## 3. SMALL-SCALE TESTS.

The flammability properties were determined using standardized fire test methods, and the thermal decomposition properties were characterized using a combination of analytical tests (DSC and PY-GC/MS) and a unique TDA.

### 3.1 MATERIAL DESCRIPTION.

Three different samples were evaluated in the small-scale tests described in this report: OSB, CFRP, and GLARE. The construction specification and/or general material descriptions of each material are provided in sections 3.1.1, 3.1.2, and 3.1.3.

#### 3.1.1 Oriented Strand Board.

The oriented strand board (OSB) was chosen as a small-scale test material because it is a wood material that is known to sustain combustion when exposed to a flaming fire source. Fire is sustained on the wood surface through flame propagation, more commonly called flame spread. Since wood is a standard building material, it has been assessed using various regulatory flame spread methods for more than half a century. The OSB has a smooth side, referred to as the finished side, and a rougher, unfinished side. In all tests performed for this analysis, the finished side of the material was exposed to the heat source. The OSB used was a Georgia Pacific Blue Ribbon® product with a nominal thickness of 14.7 mm (0.578 inch).

#### 3.1.2 Carbon Fiber-Reinforced Polymer.

The CFRP material evaluated was constructed using T-800 carbon fiber. The resin used in the construction of this composite was aerospace grade, 177°C (350°F) cured, toughened epoxy resin. The samples were constructed from unidirectional tape pre-preg in a quasi-isotropic, symmetric lay-up $[0, -45, 45, 90]S_2$ consisting of 16 plies with a total thickness of 3.2 mm (0.126 inch). The finished composite had a fiber content of approximately 60% and a resin content of approximately 40%.

#### 3.1.3 Glass Laminate Aluminum Reinforced Epoxy.

The GLARE material was fabricated according to standard protocols of fiber, aluminum, and resin as for aerospace use. The aluminum used in the material construction was 2024 T3. The pre-preg process used FM94-27%-$S_2$ glass fiber with 120°C (248°F) curing. The GLARE samples had a total nominal thickness of 2.5 mm. Each sample consisted of five layers of aluminum ($\tau$ = 0.3 mm each) and four layers of fiber at 0.127 mm per ply with total fiber per layer thickness of 0.254 mm with orientations at 0/90. The standard nomenclature for this particular GLARE configuration is GLARE3-5/4 0.3.

3.2 THE ASTM E1354 CONE CALORIMETER TEST.

The ASTM E1354, "Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products using an Oxygen Consumption Calorimeter," [1] provides a small-scale test procedure to measure the ignitability, heat release rate, mass loss rate, and combustion product generation rate of a material exposed to a specified irradiance level. Before the test, the sides of a square sample, 100 mm (4 inch) on each side, is wrapped in aluminum foil and placed in a steel sample holder. The holder with the sample is placed beneath the cone-shaped heater that provides a uniform irradiance on the sample surface, see figure 1. The sample mass is constantly monitored using a load cell, and the effluent from the sample is collected in the exhaust hood above the heater. In the duct downstream of the hood, the flow rate, smoke obscuration, and $O_2$, $CO_2$, and CO concentrations are measured continuously.

Figure 1. The ASTM E1354 Cone Calorimeter Test Apparatus

A spark igniter 12.5 mm (0.5 inch) above the sample surface is used to initiate the burning of any combustible gas mixture produced by the sample. Once the sample ignites, the burning of the sample causes a reduction in the oxygen concentration within the effluent collected by the hood. This reduction in oxygen concentration has been shown to correlate with the heat release rate of the material, 13.1 MJ per kg of $O_2$ consumed. This is known as the oxygen consumption principle. Using this principle, the heat release rate per unit area of the sample is determined with time using measurements made in the duct.

All test samples were approximately 100 mm (4 inches) square and were tested in the horizontal configuration using exposure heat fluxes of 25, 50, 75, and 100 kW/m$^2$. All samples were tested in duplicate. In addition to these calorimetry tests, the critical heat flux of each sample was measured. Figure 2 shows the cone calorimeter samples before testing.

For all tests, the samples were placed into the standard ASTM E1354 steel holder; however, the method of mounting each sample within the holder differed due to various physical and thermo-mechanical properties of the material. Both the OSB and CFRP samples were cut to size and placed into the standard holder with the unexposed side insulated using a 96 kg/m$^3$ ceramic insulation. However, due to the thermal expansion and deformation of the GLARE, as shown in figure 3, it was necessary to place the GLARE samples in the edge frame holder described in ASTM E1354-10, section 6.6.4.1 [1] during the test.

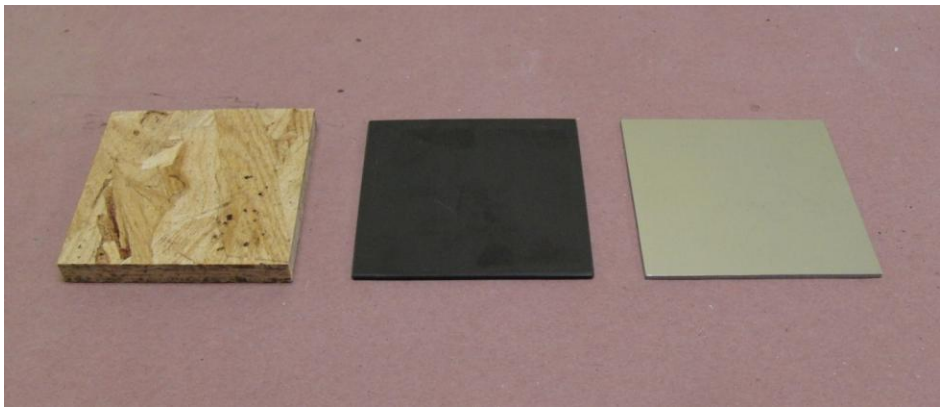


Figure 2. The ASTM E1354 Cone Calorimeter Test Samples: OSB (Left), CFRP (Center), and GLARE (Right)

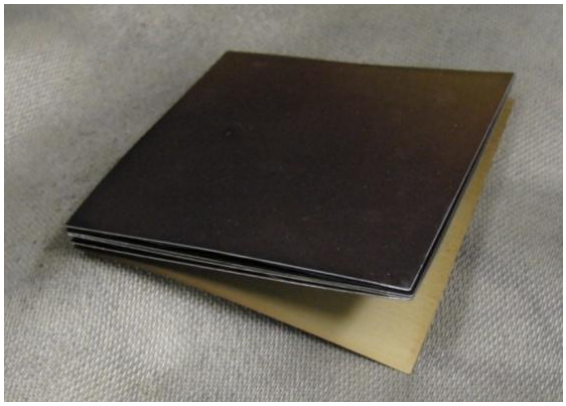


Figure 3. Thermal Deformation Observed for Unrestrained GLARE Sample Exposed to 50 kW/m$^2$ for Approximately 60 Seconds

Prior to each series of tests, the heat flux at the surface of the sample was measured using a Medtherm model GTW-7-32-485A Schmidt-Boelter-type total heat flux gauge. Once the desired heat flux was achieved, the gauge was removed, and background data was collected. After background data was collected for 2 minutes, the sample was exposed, and the test was started. All samples were burned to completion, and data was collected for an additional minute after self-extinguishment. Samples were photographed before, during, and after testing. All samples were evaluated in general accordance with the procedures outlined in ASTM E1354-10 [1].

3.3 THE ASTM E1321 LATERAL IGNITION AND FLAME SPREAD TEST.

The ASTM E1321-09 "Standard Test Method for Determining Material Ignition and Flame Spread" [2], provides a small-scale test procedure to determine the properties of a material related to the lateral spread of flame on a vertical surface due to an externally applied radiant flux. A photograph of the LIFT apparatus during calibration is provided in figure 4.

During a standard test, a 0.16-m (6.1-inch) by 0.8-m (31.5-inch) sample is exposed to a graduated heat flux. The cone calorimeter determined that this flux is 5 kW/m$^2$ higher at the hot end than the minimum heat flux necessary for ignition of the material. A plot of the graduated heat flux profile used to expose LIFT samples is provided in figure 5. The flux distribution shown in figure 5 is normalized with respect to the critical heat flux of the material being evaluated plus 5 kW/m$^2$. Once this heat flux profile is achieved, the sample is exposed, in the presence of a pilot flame along the top edge of the material, until the material ignites. Once ignited, the pilot is secured, and the rate of horizontal flame spread across the vertical face of the exposed sample is visually monitored and recorded. The test is conducted until the flame front ceases to spread, at which point the test is ended and the sample is removed. All samples in this study were evaluated in general accordance with the procedures outlined in ASTM E1321-09 [2].

Figure 4. The ASTM E1321 LIFT Apparatus

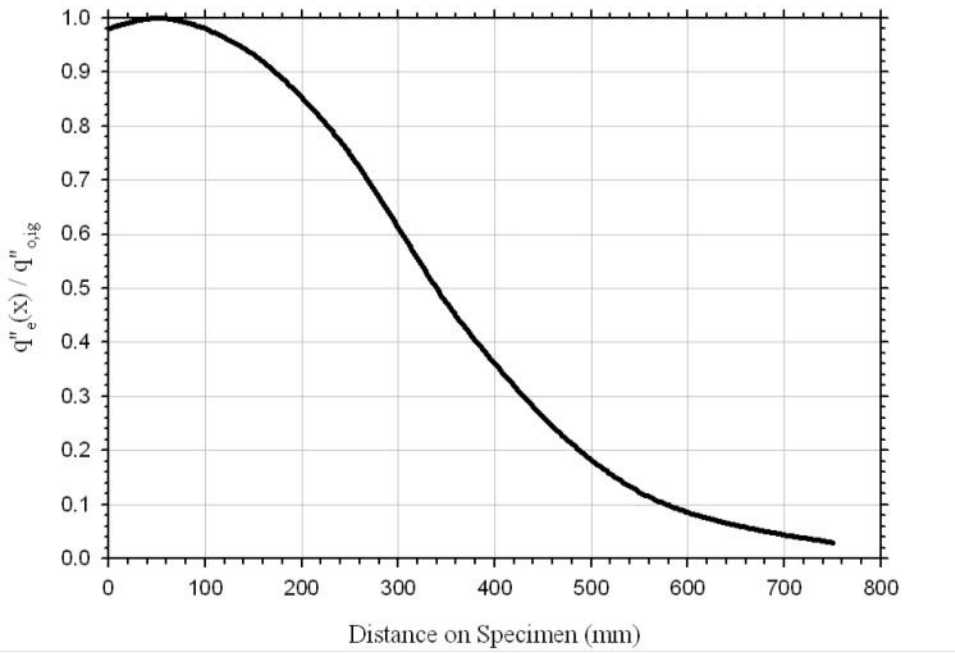
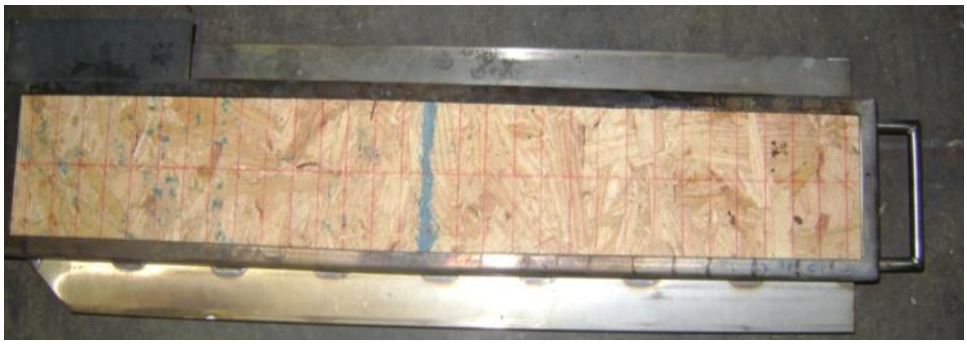
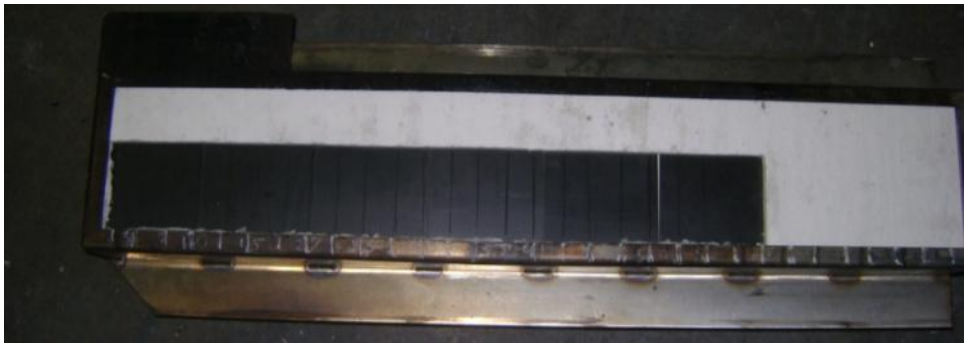



Figure 5. Normalized Heat Flux Distribution Over Sample Surface

It should be noted that, due to the sizes of the composite material samples received (i.e., 0.1 m (4 inch) square), the geometry of the CFRP and GLARE LIFT samples were smaller than that prescribed by the test standard. The CFRP composite sample evaluated was 0.1 m (4 inches) by 0.6 m (24 inches), and the GLARE composite sample evaluated was 0.1 m (4 inches) by 0.4 m (16 inches). A smaller sample size was used for the GLARE material based upon the flammability property data obtained for the material during previous testing. To accommodate these smaller samples, a modified sample holder was constructed from a 25.4-mm (1-inch) Unifrax Fiberfrax® Duraboard® LD ceramic insulation board. The composite samples were not constructed from one continuous section of the material; instead, they were created by butting several individual pieces together (figure 6).



(a) OSB



(b) CFRP

(c) GLARE

Figure 6. The ASTM E1321 LIFT Samples

## 3.4 THERMAL DECOMPOSITION TESTS.

The thermal decomposition properties of the composite and wood materials were determined using small-scale heating tests and analytical tests. All tests were designed to characterize the thermal conductivity and specific heat capacity of the composite and surrogate materials as a function of temperature. Analysis was conducted using thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and pyrolysis-gas chromatography-mass spectroscopy (PY-GCMS). These methods were used to obtain a detailed characterization of the change in

density with temperature, the heat of decomposition, and the enthalpy of combustion gases, respectively. Only the OSB and CFRP materials were evaluated in these tests. Testing of GLARE is planned during the continuation of this study.

3.4.1 Thermal Decomposition Apparatus.

The thermal properties for the OSB and CFRP materials in virgin and decomposed states were determined using the TDA. A test summary is provided in table 1 that lists the temperature set points selected for each material state. These set points were selected to provide bounding data points and midpoint values for each state.

Table 1. Decomposition Testing Conducted in TDA

| Test No. | Material | Set Point (°C) | Sample State | Purpose of Testing |
|---|---|---|---|---|
| 1 | OSB | 100 | Virgin | Virgin Properties |
| 2 | | 150 | | |
| 3 | | 200 | | |
| 4 | | 300 | | |
| 5 | | Ramp at 3°C/min | Virgin to Char | Decompose to Char |
| 6 | | 100 | Char | Char Properties |
| 7 | | 350 | | |
| 8 | | 500 | | |
| 9 | CFRP | 100 | Virgin | Virgin Properties |
| 10 | | 200 | | |
| 11 | | 300 | | |
| 12 | | Ramp at 3°C/min | Virgin to Fiber | Decompose to Fiber |
| 13 | | 200 | Fiber | Fiber Properties |
| 14 | | 400 | | |
| 15 | | 600 | | |

The TDA, shown in figure 7, was designed to be used in conjunction with the heater/exhaust hood assembly of the ASTM E1354 cone calorimeter. The TDA consisted of a 0.18-m (7-inch) cubed inerting chamber constructed from a 3.2-mm (0.125-inch) steel plate. A 0.13-m (5-inch) square opening was cut into the top of the inerting chamber permitting the exposure of samples as large as 0.10-m (4-inches) square. During testing, the inerting chamber was positioned beneath the conical heating element prescribed by the ASTM E1354 test method [1]. Test samples were placed onto a pedestal within the inerting chamber such that the exposed sample surface was 25.4 mm (1 inch) below the opening in the top of the chamber. The sample pedestal was attached to a load cell (Sartorius® FBG16EDE) with a 12-kg capacity and 0.1-g resolution. The environment inside the inerting chamber was controlled using a nitrogen purge system designed to flood the inerting chamber with nitrogen and allow both nitrogen and sample effluent to exhaust naturally through the opening in the top of the chamber. To achieve and maintain an inert environment within the sample chamber, a nitrogen ($N_2$) flow rate of 94 standard liters per

minute (200 standard cubic feet per hour) was used. The TDA was located beneath the ASTM E1354 exhaust hood during all tests, removing any exhaust products from the laboratory.

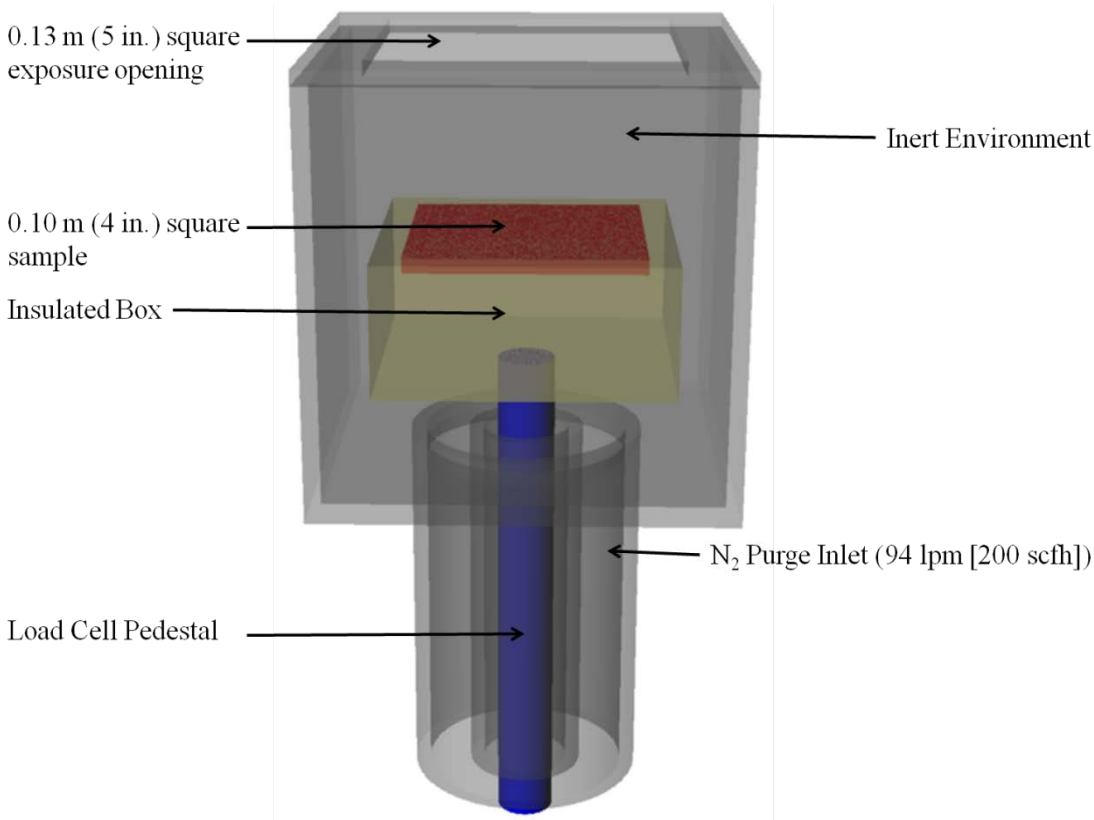



Figure 7. Rendering of the Thermal Decomposition Apparatus

The thermal environment on the exposed side of the sample was characterized using a water-cooled total heat flux gauge and a Wahl Model HSM 400 optical pyrometer. The water-cooled total heat flux gauge was a Medtherm Schmidt-Boelter-type gauge with a 0–100 kW/m$^2$ range and 3% accuracy. Prior to testing, the thermal exposure was characterized by placing the surface of the heat flux gauge at the same elevation as the sample.

The samples used in this testing were 0.1-m (4-inches) square. All samples were oven-dried at a temperature of 120°C (248°F) for a minimum of 24 hours prior to testing. The emissivity of the sample's exposed surface was controlled by coating the surface with a high-emissivity ($\varepsilon = 0.94$) black paint. The thermal response of each material tested was characterized using the Wahl Model HSM 400 optical pyrometer to measure exposed surface temperatures. A 0.25-mm (0.01-inch) diameter, bare-bead, Type K thermocouple was used to measure the unexposed side surface temperature.

The samples were installed within a unique sample holder designed to provide known boundary conditions on all sides of the sample. This is required for the conditions at the sample edges to be as close to adiabatic as possible. The sample holder was constructed of Unifrax Fiberfrax® Duraboard® LD ceramic fiber board, which has a low (0.063 W/(m-K) at 293 K) thermal

conductivity. Behind the sample, a single layer of 1.59-mm-thick ceramic felt was used to improve contact between the back of the sample and the sample holder. This provided a better measurement of the unexposed side temperature.

Prior to testing, the initial mass of the sample was measured. The sample was then wrapped in aluminum foil and placed into the sample holder. The sample holder was placed into the inerting chamber, and the $N_2$ purge system was activated. Approximately 1 minute later, the sample was exposed to the incident heat flux under evaluation, and the test was started. Tests were conducted until a steady-state condition on both the exposed and unexposed surface was reached. In general, this required exposure durations of 20 to 40 minutes. Once steady-state was reached, the sample was removed from beneath the incident heat flux with the purge system still active. The purge system was left active for an additional 2 minutes after removal to prevent thermal oxidation of the sample. After each test, the sample's final mass was measured. In general, the initial and final masses were comparable for all set point tests, which demonstrated that an inert environment was maintained throughout the exposure.

These test results were processed using the transient heat transfer code HEATING, V7.3 [3]. The heat transfer coefficient used in this analysis was determined to be 12 W/m-K based upon experimental testing using a sample with well-characterized thermal properties. During a test with a constant heat flux, the transient temperatures are governed by the specific heat capacity and surface emissivity, while the quasi-steady state temperatures are primarily a function of thermal conductivity and the heat transfer coefficient.

3.4.2 Thermogravimetric Analysis.

The OSB and CFRP mass loss at elevated temperatures was measured using a TA Instruments® Thermogravimetric Analyzer (TGA-2050). Tests were performed at heating rates of 2°, 20°, and 50°C/min in 100% Argon (inert) environment. All samples were taken from ambient conditions up to a maximum temperature of 700°C (1292°F). These temperature profiles and maximum temperatures were selected to provide bounding thermal decomposition parameters for each material. All samples weighed 20 mg ±1 mg. Fragment samples were cut from the wood and composite laminate samples.

3.4.3 Differential Scanning Calorimetry.

The specific heat capacity and heat of decomposition of the wood and composite materials were also measured using a TA Instruments® Differential Scanning Calorimeter (DSC-2010). The DSC tests were conducted at a heating rate of 20°C/min in a 100% nitrogen environment. As prescribed in ASTM E1269 [4], the heat flow due to the aluminum pan and lid was measured prior to testing. The lid contained holes to allow the pyrolysis gases to flow out of the pan. The DSC was calibrated with a sapphire sample weighing approximately 25 mg. The wood and composite materials were tested in fragment form with sample masses of 14 mg ±1 mg.

3.4.4 Pyrolysis–Gas Chromatograph/Mass Spectroscopy.

The characterization of the gases released during the OSB and CFRP thermal decomposition was done using a pyrolysis furnace coupled to a gas chromatograph/mass spectrometer. A Frontier

Laboratories PY-2020iD furnace was used to decompose the OSB and CFRP at set temperature thresholds. The gases released were then measured using an Agilent 6890N gas chromatograph coupled with an Agilent® 5973 mass spectrometer. In this test, samples were decomposed in the furnace for 25 seconds at temperature set points of 350°C (662°F) and 600°C (1112°F). The decomposition gases were transferred to and retained within a cryogenic trap at -188°C (-306°F) until analysis could be performed. The pyrolysate was injected in splitless mode with the injector at 230°C. Separation by chromatography was done using a 25-m nonpolar capillary column (BP5 SGE) with an inner diameter of 0.25 mm and phase thickness of 25 mm scavenged by helium at 1 ml/min. The chromatograph oven was programmed for analysis as follows: -40°C isothermal for 20 s, -40 to 150°C at a rate of 5°C/min, 150° to 250°C at a rate of 10°C/min, and 250° to 300°C at a rate of 20°C/min. The pyrolysis products were identified by mass spectrometry detection in positive electronic impact ionization with an ionization energy of 70 eV and a pressure of 0.1 Pa in the source. The mass range scavenged was between 40 and 600 daltons. This approach was adopted from previous work done to analyze the gases released during the decomposition of glass fiber-vinyl ester composite [5].

## 4. TEST RESULTS.

A total of 51 small-scale fire tests and subsequent analyses were conducted. This characterization was accomplished using six different test methods. The results of these tests and modeling input data generated from them are presented in sections 4.1 through 4.3.

### 4.1 THE ASTM E1354 CONE CALORIMETER RESULTS.

Prior to cone calorimeter testing, a series of critical heat flux tests were conducted on each sample to determine the minimum radiant flux required for piloted ignition of the samples. In these tests, the surface temperature of the samples was monitored to provide an approximate ignition temperature. The density of each sample was also measured prior to testing. A summary of these results is provided in table 2.

Table 2. Ignition Characteristics and Material Densities

| Sample Identification | Density ($kg/m^3$) | Critical Heat Flux ($kW/m^2$) | Ignition Temperature (K) |
|---|---|---|---|
| OSB | 696 | 12 | 610 |
| CFRP | 1598 | 16 | 658 |
| GLARE | 2320 | 25 | 848 |

The OSB material had the lowest density and critical heat flux. The critical heat flux for the CFRP material was only slightly higher (4 $kW/m^2$) than that measured for the OSB material. The GLARE material had a significantly higher critical heat flux than the other two materials evaluated.

A summary of the average results from the OSB, CFRP, and GLARE materials is provided in table 3. Plots of the average heat release rates at each exposure heat flux for each of these materials are also provided in figures 8 through 10.

Table 3. The ASTM E1354 Cone Calorimeter Results

| Material ID | Heat Flux ($kW/m^2$) | Fraction Burned (%) | Time to Ignition (s) | Burn Duration (s) | Test Avg. Eff. HOC (MJ/kg) | Total Heat Released ($MJ/m^2$) | Test Avg. HRR ($kW/m^2$) | Peak HRR ($kW/m^2$) | Time of Peak (s) | Test Avg. SEA ($m^2/kg$) |
|---|---|---|---|---|---|---|---|---|---|---|
| OSB | 25 | 75.5 | 158 | 847 | 11.8 | 70.8 | 83 | 182 | 615 | 61 |
| | 50 | 77.9 | 32 | 596 | 12.2 | 76.6 | 128 | 259 | 472 | 118 |
| | 75 | 80.1 | 14 | 525 | 14.1 | 94.0 | 179 | 336 | 402 | 187 |
| | 100 | 82.5 | 8 | 490 | 12.9 | 84.6 | 172 | 331 | 341 | 190 |
| CFRP | 25 | 22.2 | 166 | 189 | 18.5 | 18.5 | 96 | 213 | 73 | 946 |
| | 50 | 23.8 | 68 | 168 | 18.4 | 20.1 | 118 | 315 | 47 | 1069 |
| | 75 | 25.6 | 40 | 142 | 18.4 | 21.9 | 152 | 394 | 38 | 1069 |
| | 100 | 23.8 | 29 | 113 | 16 | 16.6 | 153 | 328 | 37 | 1133 |
| GLARE | 25 | 4.3 | 847 | 500 | 16.7 | 1.1 | 2.5 | 9 | 227 | 377 |
| | 50 | 8.9 | 239 | 234 | 19.2 | 9.8 | 41.5 | 128 | 58 | 1092 |
| | 75 | 9.4 | 98.5 | 164 | 17.9 | 9.6 | 57.0 | 167.5 | 66 | 1282 |
| | 100 | 9.05 | 82.5 | 129 | 16.85 | 8.75 | 66.5 | 167.5 | 33.5 | 1323 |

HOC = Heat of combustion
HRR = Heat release rate
SEA = Specific extinction coefficient

In general, the results from the OSB samples were consistent and repeatable. As the incident heat flux increased, the time to ignition decreased, and the peak heat release rates generally increased.

Similar trends were observed for the CFRP samples except for the HRR of 100 $kW/m^2$ samples. The peak heat release rates increased from the 25 to 30 and 75 $kW/m^2$ samples but decreased for the 100 $kW/m^2$ samples. For the CFRP samples, the time to ignition decreased as expected, but the peak heat release rate did not increase; thus, the results for the 100 $kW/^2$ exposure are questionable. Results obtained from the testing conducted at heat fluxes of 25–75 $kW/m^2$ were relatively consistent with the results reported in the literature.

Finally, the results from the GLARE samples for exposures of 50 $kW/m^2$ and greater were generally good and consistent with the behavior expected (i.e., decreasing time to ignition/increasing peak heat release rate as exposure heat flux increases). However, due to the fact that 25 $kW/m^2$ was identified as the critical heat flux for this material, the heat release rate results produced at this exposure were minimal, as shown in figure 10.

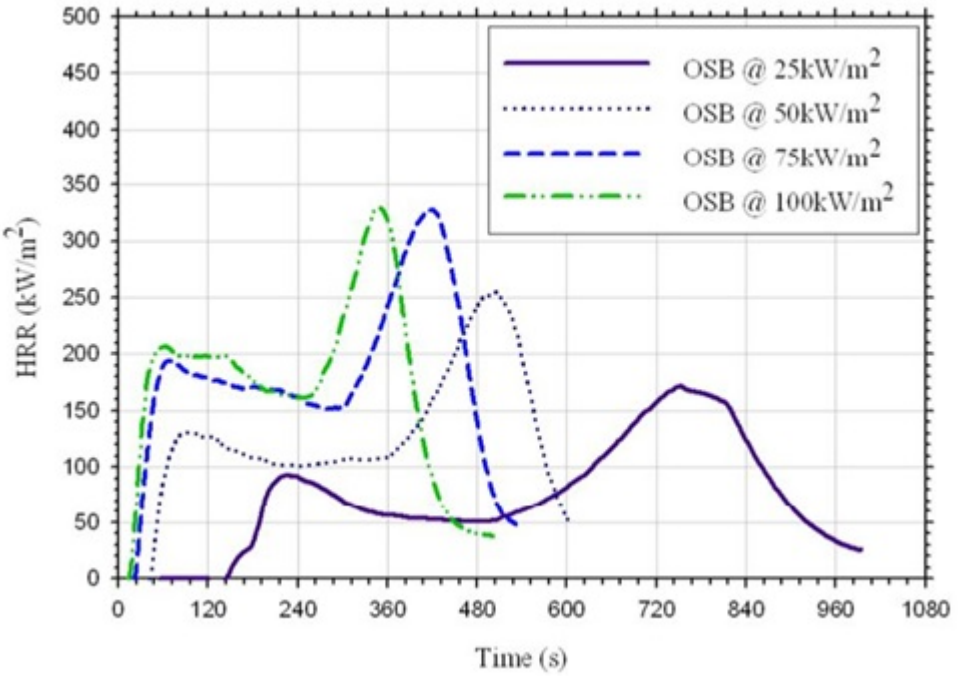



Figure 8. Average OSB Heat Release Rates Measured in ASTM E1354 Cone Calorimeter Testing

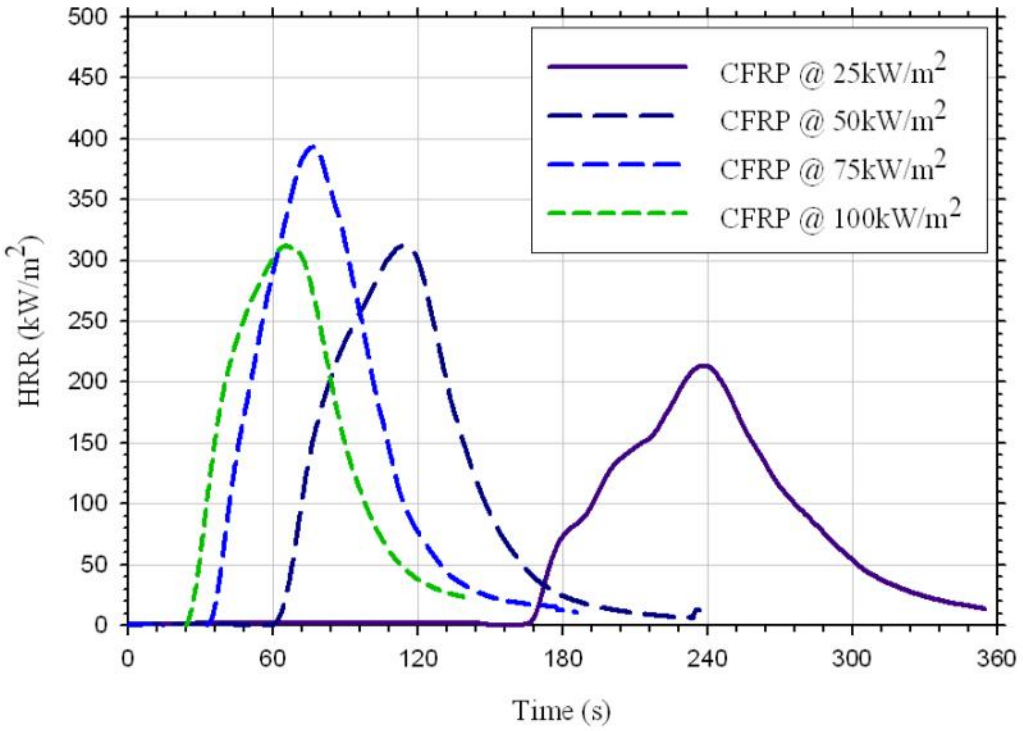



Figure 9. Average CFRP Heat Release Rates Measured in ASTM E1354 Cone Calorimeter Testing

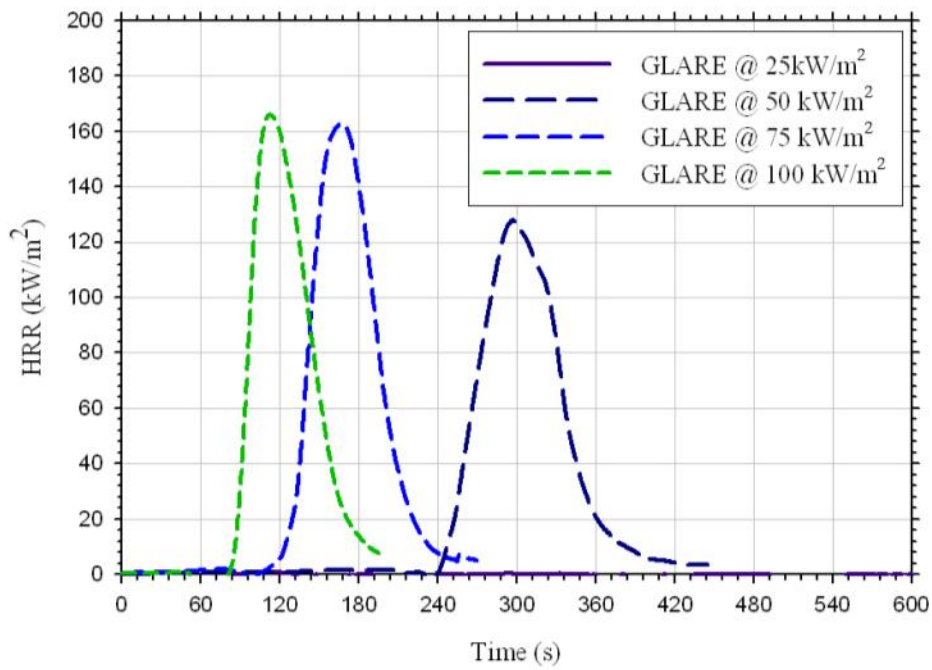



Figure 10. Average GLARE Heat Release Rates Measured in ASTM E1354 Cone Calorimeter Testing

4.2 THE ASTM E1321 LATERAL IGNITION AND FLAME SPREAD TEST RESULTS.

Flame spread testing with the LIFT apparatus was only conducted once for each composite material. Additional tests are planned in the future. Triplicate testing was conducted for the OSB wood surrogate. Photographs of the lateral flame front progressing down each sample material are provided in figure 11.

In general, it was found that OSB was the only material on which quantifiable lateral flame spread was observed. On this material, lateral flame spread was observed as far as 0.45–0.55 m (1.5–1.8 ft) over the course of 8 to 10 minutes. The OSB lateral flame spread results are provided in figure 12. Both composite materials exhibited very little lateral flame spread under the exposures specified by the standard. The CFRP material ignited at a joint location (i.e., where two samples were butted together), and the flame did not spread more than 0.05 m (2 inches) away from this location for the duration of the exposure. The flame burned only in this location before self-extinguishing in the presence of the external radiant flux. Similar behavior was observed for the GLARE material. During the evaluation of the GLARE material, the samples deflected toward the radiant exposure thus exposing more of the seam. In turn, this exposed material ignited and burned. As shown in figure 11(c), the burning occurred primarily at each exposed seam with no involvement of the exposed material face and no lateral flame spread.

The limited degree of flame spread observed for both composite materials prevented any analysis of lateral flame-spread parameters. An analysis was done for the OSB material in general

accordance with the methods recommended in ASTM E1321 [2]. The results of this analysis are provided in table 4.

(a) OSB

(b) CFRP

(c) GLARE

Figure 11. Lateral Flame Spread/Flame Attachment on Exposed Surface of (a) OSB, (b) CFRP, and (c) GLARE Materials When Evaluated in ASTM E1321 LIFT Apparatus

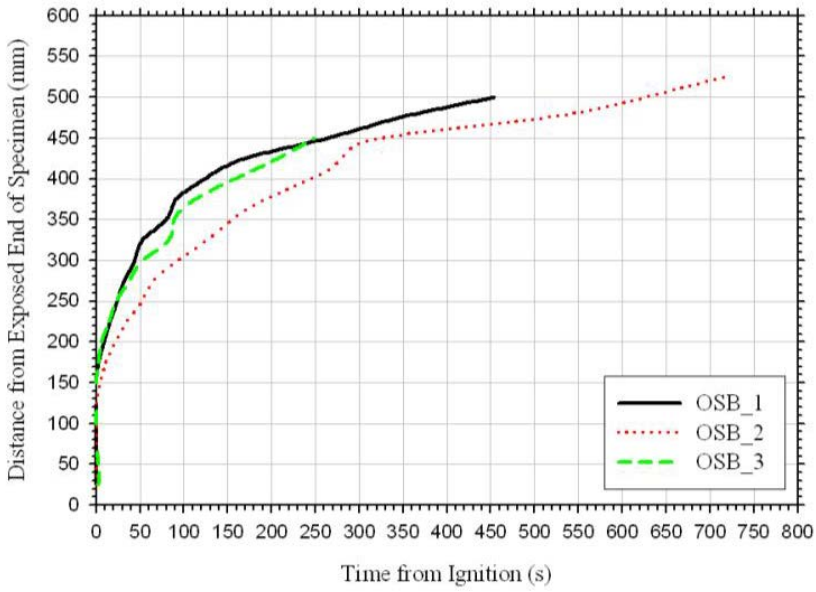



Figure 12. The ASTM E1321 LIFT Results for OSB

Table 4. Flammability Parameters Derived from LIFT

| Material | $q''_{o,ig.}$ ($kW/m^2$) | $T_{ig}$ (K) | $K\rho c$ $(kW/m^2\text{-}K)^2\text{-}s$ | $\Phi$ ($kW^2/m^3$) | $T_{s,min}$ (K) |
|---|---|---|---|---|---|
| OSB | 12 | 610 | 0.283 | 4.3 | 471 |
| CFRP | 16 | 658 | - - | - - | - - |
| GLARE | 25 | 848 | - - | - - | - - |

The flame-spread data presented in figure 12 combined with critical heat flux and time-to-ignition data obtained for the OSB material were used to derive material flammability properties in accordance with ASTM E1321 procedures [2]. The main parameters derived from this test method are summarized in table 4 and include: the material thermal-heating property ($k\rho c$); the flame-heating parameter ($\Phi$); and the minimum temperature for lateral flame spread ($T_{s,min}$). A complete description of the derivations used to develop these material properties can be found in ASTM E1321 [2]. Due to the limited and inconsistent flame-spread behavior of the CFRP and GLARE materials, the flammability parameters listed in table 4 could not be calculated. It is expected that additional testing at higher-incident heat fluxes would be required to properly assess the lateral flame-spread behavior of these materials.

## 4.3 THERMAL DECOMPOSITION RESULTS.

### 4.3.1 Thermal Decomposition Apparatus Results.

As indicated earlier, the thermal properties of both the virgin and decomposed states of the OSB and CFRP materials were characterized using the TDA. For these tests, the virgin state is defined as the condition in which the material was received after being exposed to ambient conditions. The decomposed state is defined as the condition of the material after being heated

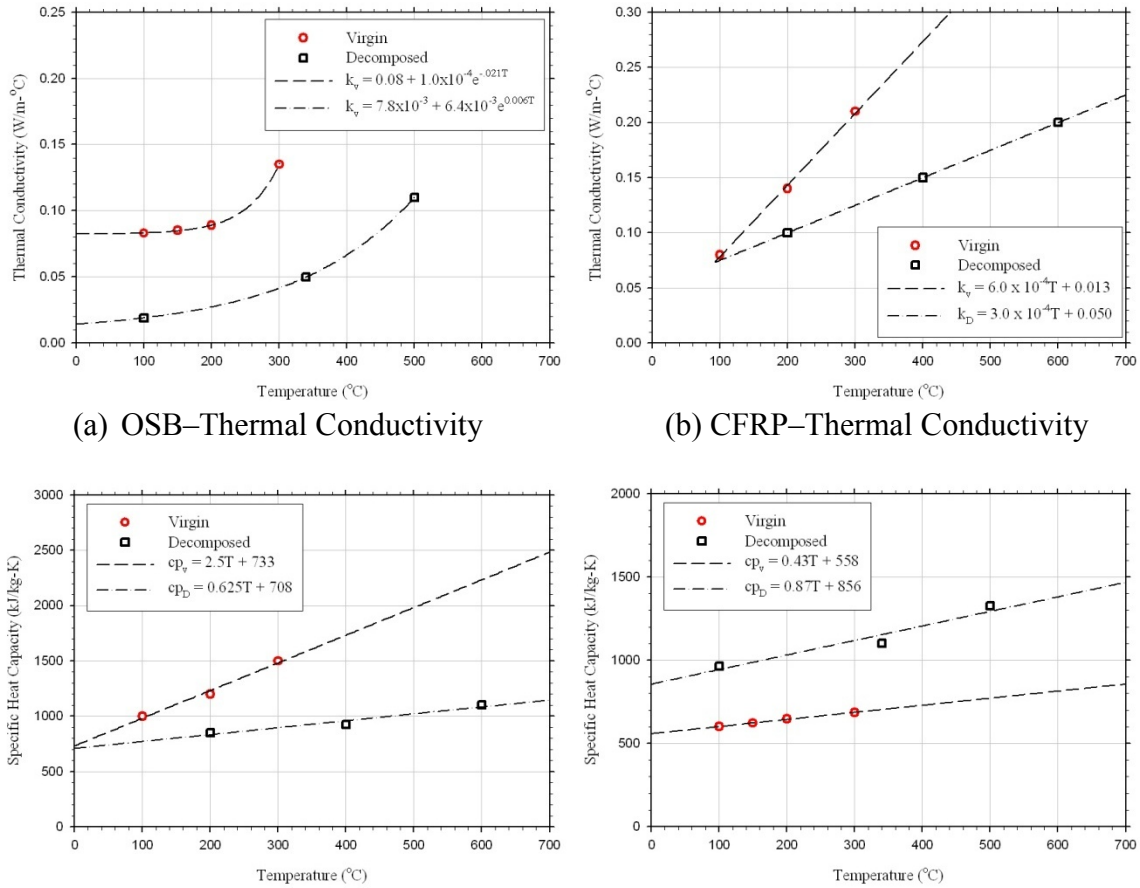
to 600°C (1112°F). Once decomposed, OSB and CFRP sample geometry and corresponding densities changed. It was found that the CFRP samples had an average decomposed thickness of 6.7 mm and a density of 1176 k/m$^3$. The OSB samples had an average decomposed thickness of 8.8 mm and a density of 464 k/m$^3$. The CFRP sample nearly doubled in thickness with a 25% decrease in density, while the OSB sample decreased in thickness by approximately 26% with a density change of 66%. A summary of these changes is provided in table 5.

Table 5. Sample Thickness and Density in Both the Virgin and Decomposed Material States

| Material | Initial Thickness (mm) | Decomposed Thickness (mm) | Initial Density (kg/m$^3$) | Decomposed Density (kg/m$^3$) |
|---|---|---|---|---|
| OSB | 14.7 | 8.8 | 696 | 464 |
| CFRP | 3.2 | 6.7 | 1598 | 1176 |

Using the temperature data collected in the TDA, the thermal conductivity and specific heat capacity of virgin and decomposed states of both materials were developed at each set point identified in table 1. A summary of these derived thermal properties is provided in figure 13.

In general, the virgin material properties determined in this work were relatively consistent with those reported in the literature. Literature data for the CFRP material suggests that the thermal conductivities of the material at 25°C (77°F) and 300°C (572°F) could range from 0.10 to 0.13 W/m-K and 0.26 to 0.32 W/m-K, respectively. As shown in figure 13, the thermal conductivities derived in this work were 15%–20% lower than the ranges provided in the literature, which could be an artifact of slight material differences or measurement errors in the empirical data sets. Similar comparisons were made for the virgin material specific heat values, and the values in this work were found to be within 15%–25% of the data reported in the literature. Temperature-dependent thermal-property data for the composite decomposed material and the wood surrogate material were not available in the literature; thus, comparisons could not be made.



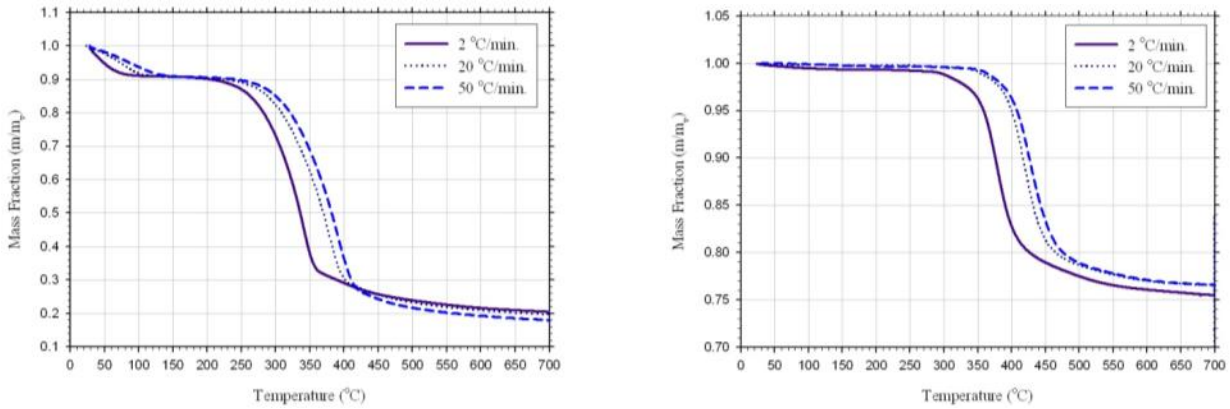

(a) OSB–Thermal Conductivity (b) CFRP–Thermal Conductivity

(c) OSB–Specific Heat Capacity (d) CFRP–Specific Heat Capacity

Figure 13. Thermal Conductivity and Specific Heat Capacity at Different Material States

4.3.2 Thermogravimetric Analysis Results.

Three fragment samples of OSB and CFRP were tested in the TGA. These samples were tested at heating rates of 2°, 20°, and 50°C/min in a 100% Argon (inert) environment. Plots of the decomposed fraction at each heating rate are provided in figure 14. It should be noted that the temperature in the plot corresponds to the gas temperatures proximate to the sample, not the actual sample temperatures. However, due to the relatively small size of the samples, these temperatures are assumed to be equal. Both materials were found to have a relatively consistent fraction of mass remaining with values of 0.19 ±0.10 and 0.76 ±0.06 for the wood and composite materials, respectively.




(a) OSB Results (b) CFRP Results

Figure 14. Decomposition of OSB and CFRP Samples in TGA at Various Heating Rates

Thermogravimetric analysis of the OSB material shows that the thermal decomposition of the material could be divided into two separate reactions with the first reaction corresponding to the removal of moisture from the sample, occurring at temperatures less than l00°C (212°F). The second reaction corresponded to the decomposition of the wood material. The sample moisture content was on average 8.8% of the total sample mass. In general, the secondary reaction of the OSB material occurred within the temperature range of 225°–425°C (437°–797°F). Reactions associated with the thermal decomposition of the CFRP composite occurred in a single phase generally between the temperature range of 300°–475°C (572°–887°F). Table 6 contains the Arrhenius kinetic parameters developed using the TGA data presented in figure 14. The parameter that provide the best fit at each heating rate are provided, as well as the parameters that provide the best fit for all heating rates. These parameters were determined using a thermal decomposition model.

Table 6. Arrhenius Decomposition Kinetic Parameters for OSB and CFRP Samples

| Material | Reaction Stage | Heating Rate (°C/min) | A [Hz] | E [J/mol] | N [- -] |
|---|---|---|---|---|---|
| OSB | 1—Water | 2 | -- | -- | -- |
| | | 20 | 500 | 2.75E+4 | 2.0 |
| | | 50 | 500 | 2.65E+4 | 2.0 |
| | | Best Fit | 500 | 2.70E+4 | 2.0 |
| | 2—Wood | 2 | 2.5E+5 | 7.5E+4 | 1.8 |
| | | 20 | 4.5E+5 | 7.2E+4 | 1.5 |
| | | 50 | 5.0E+5 | 7.0E+4 | 1.2 |
| | | Best Fit | 4.0E+4 | 7.2E+4 | 1.5 |
| Composite | N/A | 2 | 2.20E+36 | 4.52E+5 | 6.5 |
| | | 20 | 9.80E+36 | 4.68E+5 | 6.5 |
| | | 50 | 9.00E+36 | 4.69E+5 | 6.5 |
| | | Best Fit | 7.00E+36 | 4.63E+5 | 6.5 |

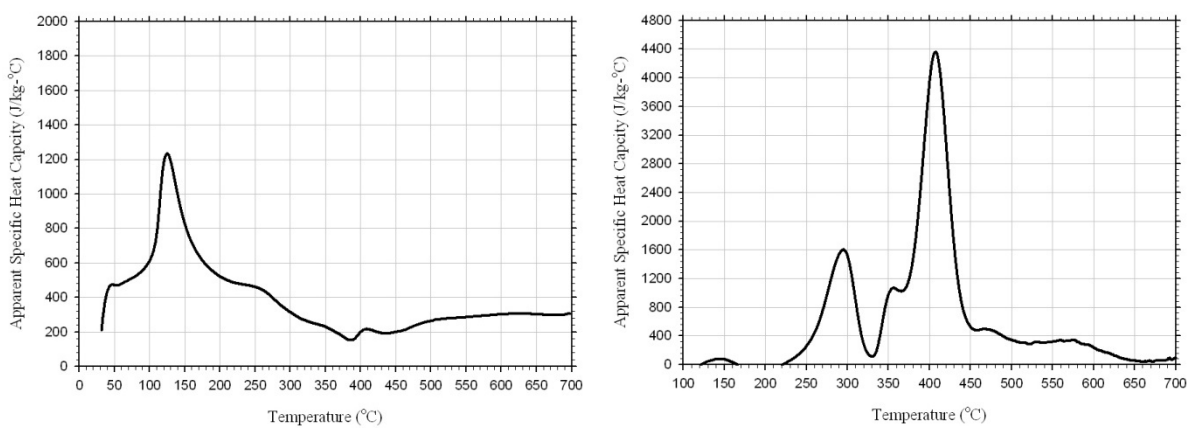
4.3.3 Differential Scanning Calorimetry Results.

Tests were conducted on fragment samples of both OSB and CFRP in a DSC to determine the apparent specific heat capacity of the material as a function of temperature. These tests were conducted at a heating rate of 20°C/min in an inert environment. The apparent specific heat capacity measured using the DSC is based on the initial mass, which is assumed to be constant during the test. However, the specific heat capacity based on the actual mass is required to determine the heat of decomposition. Consequently, the specific heat capacity was corrected for the actual mass, using the mass loss data from the TGA at the same heating rate (i.e., 20°C/min).

Using the methodology outlined by Lattimer [6] the heat of decomposition of both the OSB and CFRP materials was calculated (table 7). The value obtained for the composite material was found to be slightly higher than the values determined by Quintiere [7] for a similar composite. However, the methods used to determine these values differed. The values reported in the literature are based on a per-unit, original-mass basis, while the values reported in this work are corrected for mass loss as a function of temperature. Taking these variables into account, the heat of decomposition calculated from this work was found to be generally similar to that obtained in the previous study. The apparent specific heat capacities for both materials are presented in figure 15.

Table 7. Heat of Decomposition for OSB and CFRP Materials

| Sample ID | Initial Mass (g) | Decomposed Mass (g) | Heat of Decomposition (kJ/kg) |
|---|---|---|---|
| OSB | 15.7 | 3.1 | -260 |
| CFRP | 14.0 | 10.7 | -350 |




(a) – OSB (b) - CFRP

Figure 15. Apparent Specific Heat Capacities

4.3.4 Pyrolysis-Gas Chromatograph/Mass Spectroscopy Results.

It was intended that the PY-GCMS tests would be used to characterize the specific heat capacity of the gases released during the decomposition of the materials. This can be done by identifying

the various gases and concentrations of these gases as they are released at various stages of decomposition. The analysis required to determine these gas enthalpies remains to be done. The raw data collected for this analysis is presented in appendix A.

## 5. SUMMARY.

A total of 51 small-scale fire tests and analyses were conducted to develop a data set of model input parameters for flame spread and thermal decomposition model development. These tests were designed to characterize the flammability and thermal decomposition properties of three different materials; OSB, CFRP, and GLARE. This characterization was done using six different test methods. The results of the LIFT on the CFRP and GLARE materials were inconclusive due to intermittent flame spread and bowing in the material samples at the joints. It is expected that LIFT conducted at higher heat fluxes would yield flame spread parameters that could be used to assess the potential of the OSB to function as a surrogate test material. Also, the analysis of the PY-GCMS test results was not completed as part of this effort, although the raw data is provided in appendix A.

Overall, the results reported for the cone calorimeter, LIFT, and thermal decomposition testing of the OSB samples are comparable to published data for similar products. A comparison of the OSB cone calorimeter test results to the CFRP and GLARE cone calorimeter tests results reveals that the OSB and CFRP have similar ignition characteristics, but the GLARE material has a significantly higher ignition temperature and critical heat flux. This suggests that the OSB material is not well suited as a surrogate test material for the GLARE, when the critical heat flux and ignition temperature will have a strong influence on the test results. This is not the case for the OSB and CFRP materials, although it is noted that the OSB has a somewhat lower critical heat flux and ignition temperature and is therefore easier to ignite than the CFRP.

The thermal decomposition tests of the composites were only conducted for the CFRP material given the results of the cone calorimeter testing and the complexity associated in working with the GLARE material. A comparison of the OSB and CFRP TDA and TGA data indicates some differences in the decomposition and thermal properties of the two materials. First, the OSB begins to decompose at a temperature that is 50°C lower than the CFRP. This is consistent with the slightly lower ignition temperature and critical ignition heat flux for the OSB material compared to those for the CFRP material observed in the cone calorimeter tests. A second difference shown in the TDA testing is that the OSB material decreases in thickness during decomposition, whereas the CFRP material increases in thickness. It is interesting that the final thickness of the two materials is within 2 mm. The TDA testing also showed that the heat capacity of the OSB decreases during decomposition, but the heat capacity of the CFRP increases during decomposition. The average heat capacity during decomposition and the average thermal conductivities of the two materials is similar, which indicates the OSB is a reasonable surrogate for the CFRP over the course of the decomposition process, where the heat capacity and thermal conductivity parameters strongly influence the results.

Finally, it was observed through the DSC testing that there were significant differences in the apparent specific heat of the OSB and CFRP, by as much as a factor of four. These differences are based on a correction for the actual mass using mass loss data determined in the TGA at the same heating rate. The overall average apparent heat capacity is comparable, which is consistent with the TDA heat capacity results. This suggests the OSB is a reasonable overall surrogate material for the CFRP where the heat capacity is a significant parameter, but there may be significant differences in thermal absorption rates on smaller time scales.

## 6. REFERENCES.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# APPENDIX A—ANALYTICAL LABORATORY REPORT

**Intertek**
160 James Drive East, Suite 100
St. Rose, Louisiana 70087
Ph: (504) 602-2192
Fax: (504) 471-6111

## Report of Analysis

**2010- 4513-PCL**

| | | | |
|---|---|---|---|
| **Client:** | Hughes Associates, Inc. | **Date Requested:** | Monday, June 28, 2010 |
| **Contact:** | Christopher Mealy | **Date Received:** | Monday, June 28, 2010 |
| **Client Reference No:** | PO# 2270-C | **Collected by:** | Client |

| Client Sample Description | Product | Sample ID |
|---|---|---|
| Wood | Other 1 | 4513-001-PCL |
| Composite | Other 1 | 4513-002-PCL |

**RESULTS:** SEE ATTACHED SHEETS

**APPROVED BY** _____ **DATE:** 7/25/2010

Intertek

This report has been reviewed for accuracy, completeness, and comparision against specifications when available. The reported results are only representative of the samples submitted for testing. This report shall not be reproduced expect in full without written approval of the laboratory.

**Report of Analysis**
**2010-4513-PCL**

| | |
|---|---|
| **Sample ID:** 4513-001 | **Date Received:** Monday, June 28, 2010 |
| **Product:** Wood and Composite | **Date Analyzed:** Monday, June 28, 2010 |

| Method | Test | Result | Units |
|---|---|---|---|
| TGA | TGA | see attached | |
| DSC | DSC | see attached | |
| GC-MS | Pyrolysis GC-MS Scan | see attached | |

Initial: _____

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**Report of Analysis**
**2010-4513-PCL**

| | |
|---|---|
| **Sample ID:** 4513-002 | **Date Received:** Monday, June 28, 2010 |
| **Product:** Wood and Composite | **Date Analyzed:** Monday, June 28, 2010 |

| Method | Test | Result | Units |
|---|---|---|---|
| TGA | TGA | see attached | |
| DSC | DSC | see attached | |
| GC-MS | Pyrolysis GC-MS Scan | see attached | |

Initial: _____

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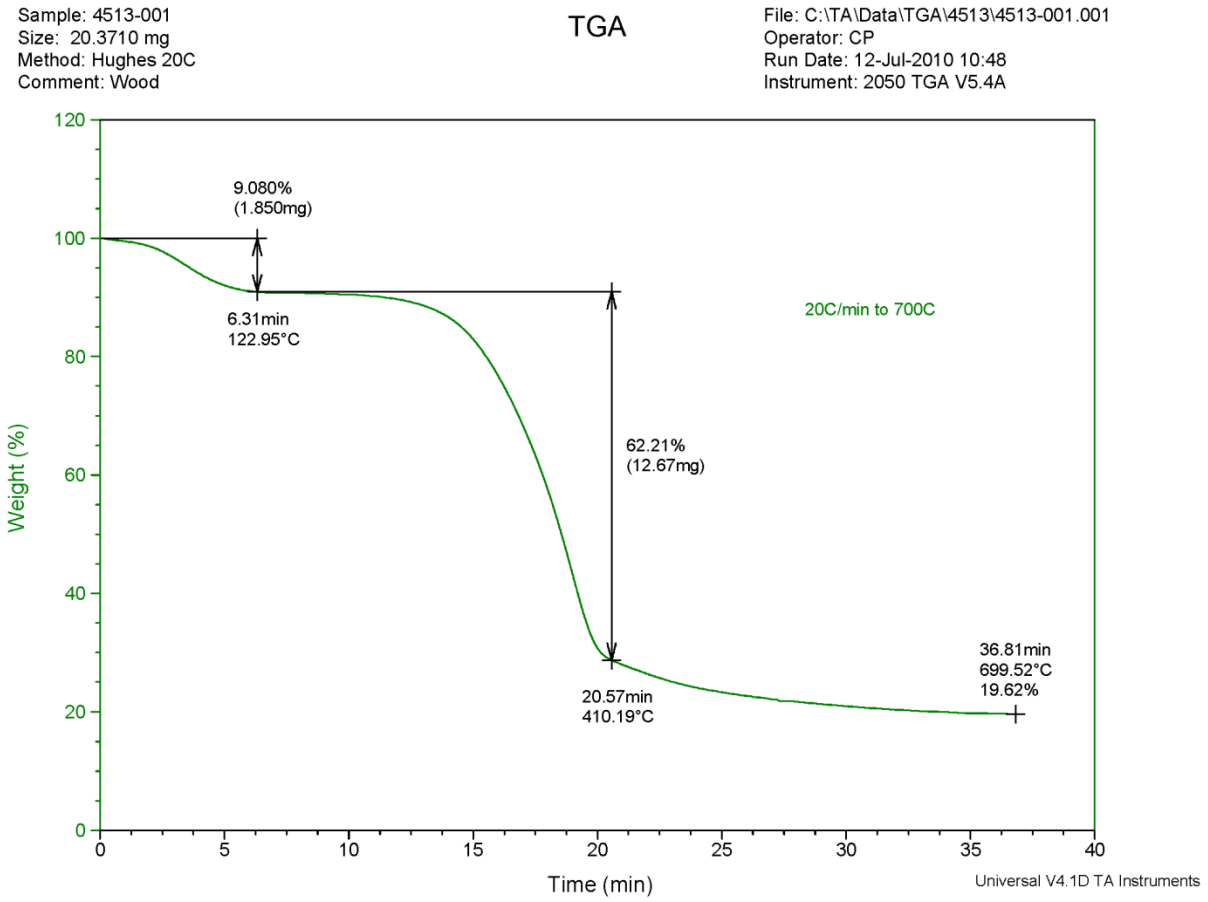
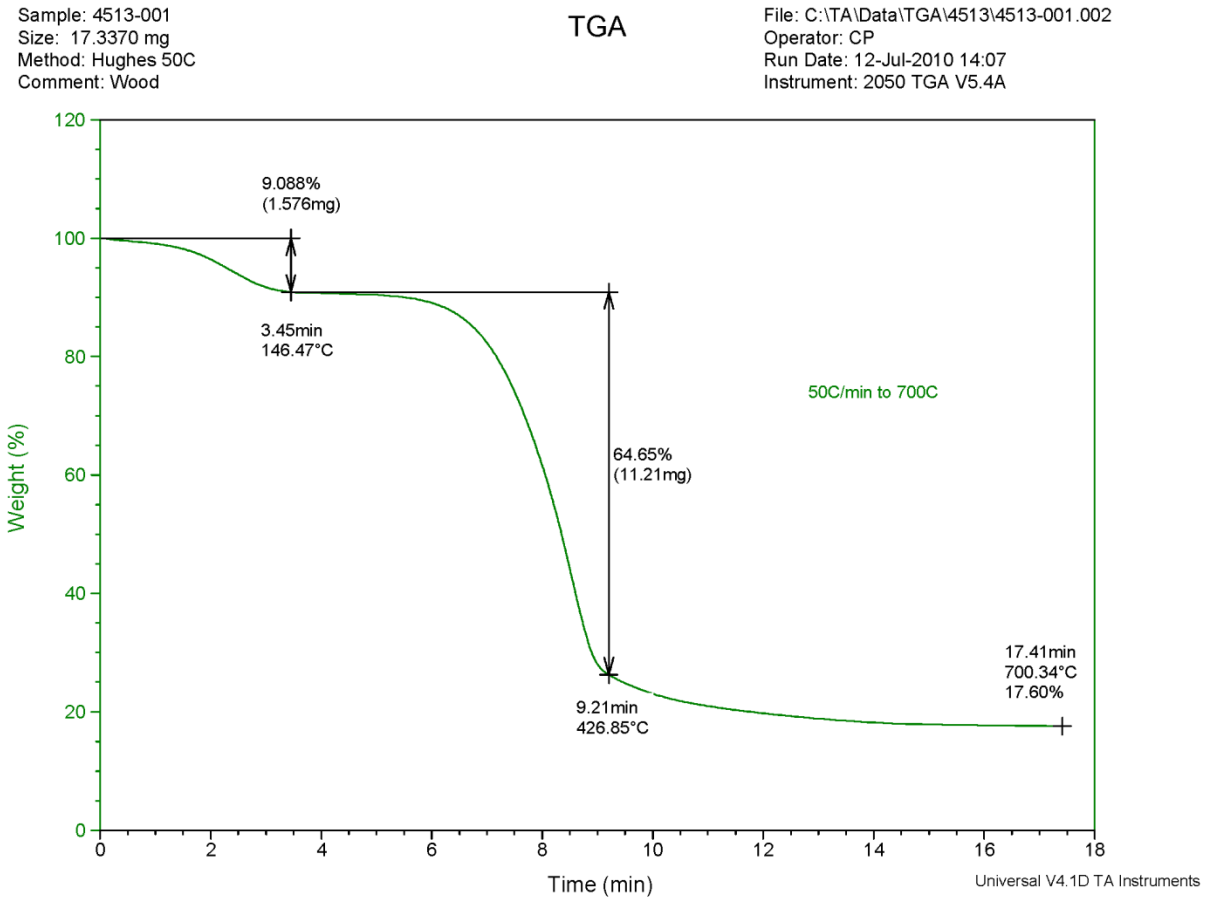
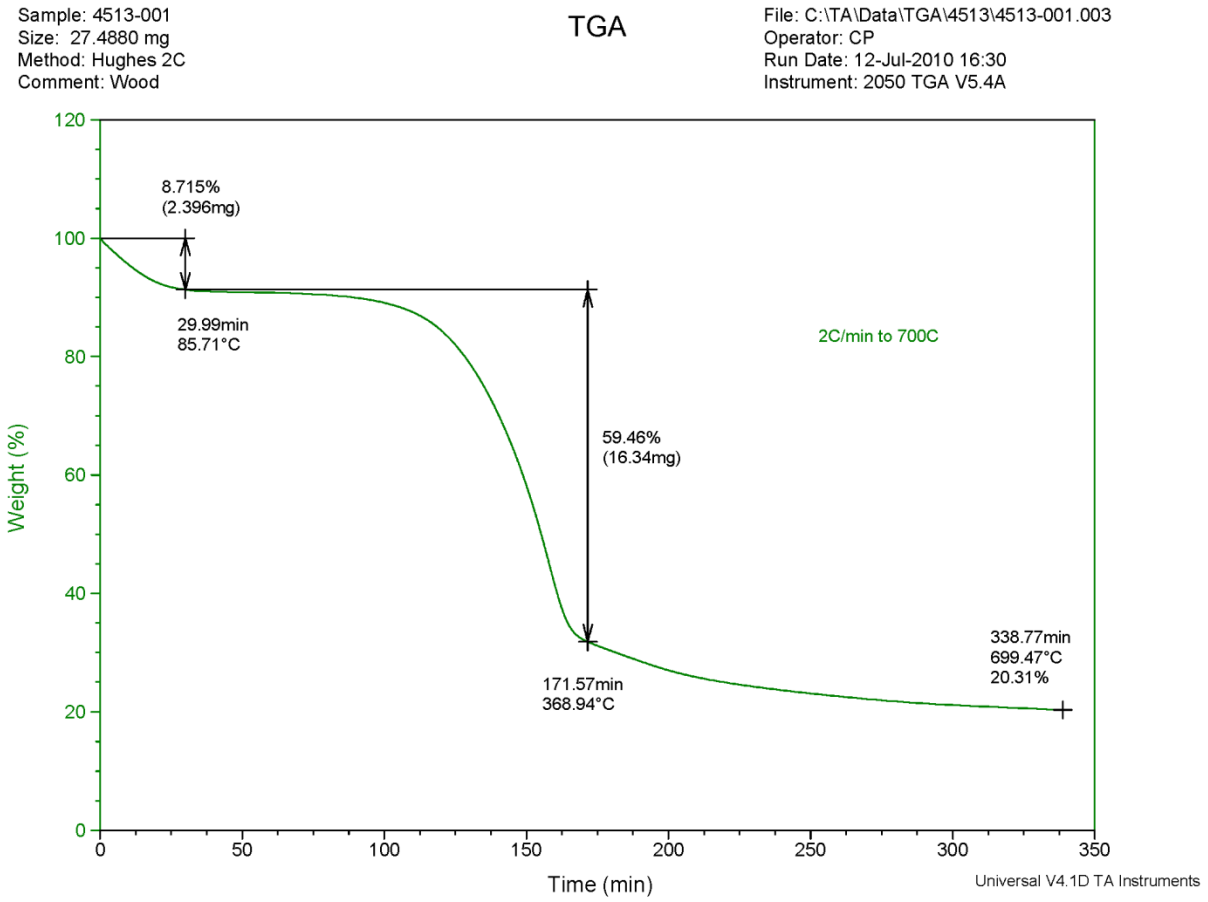
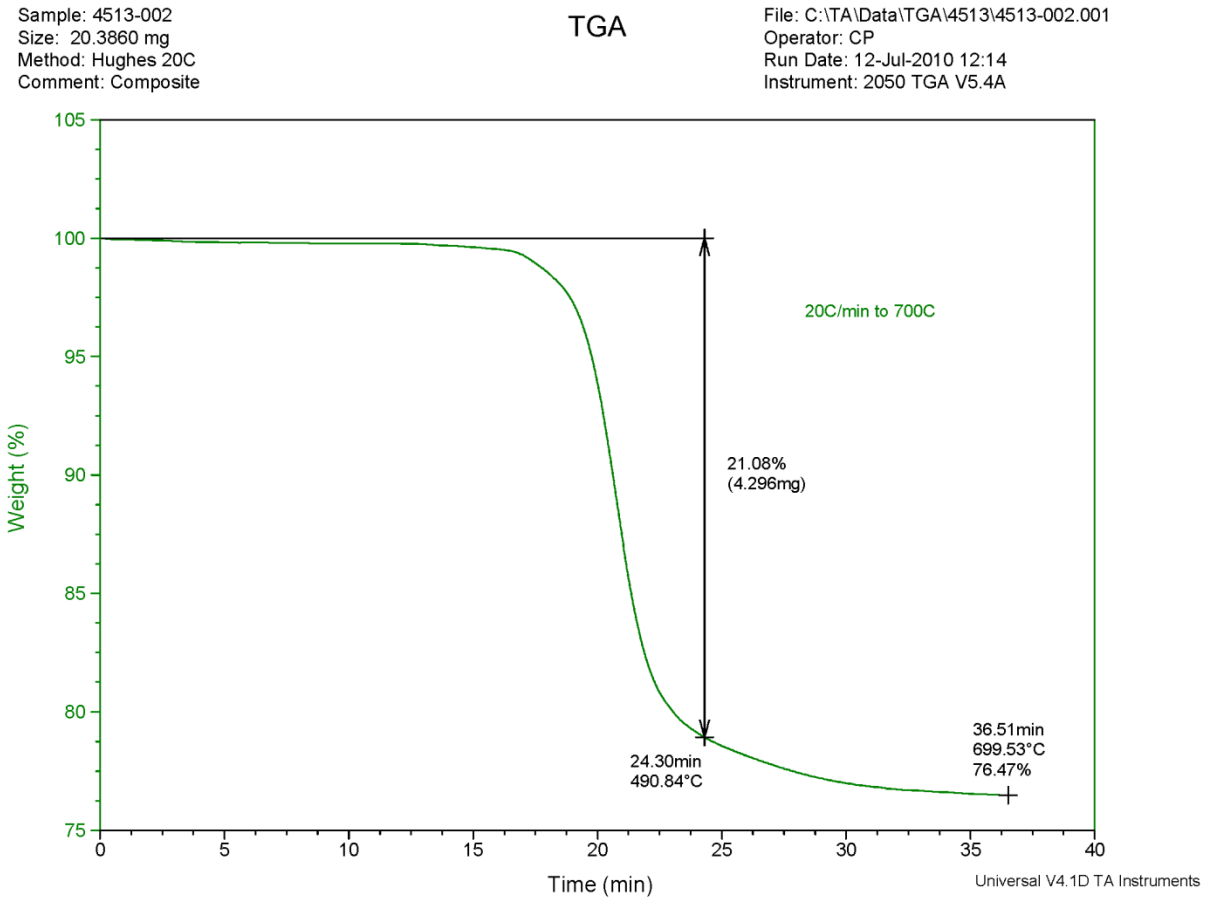
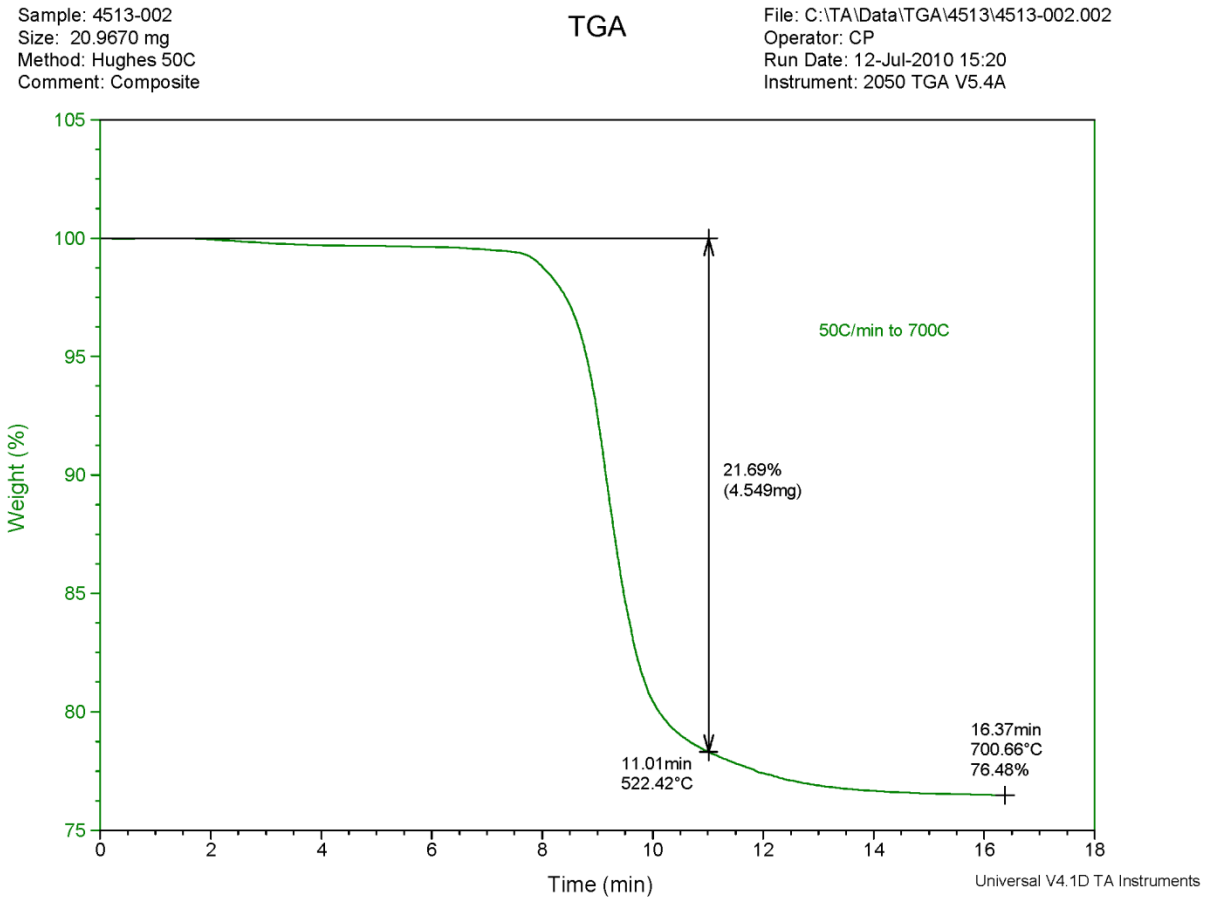
160 James Drive East, Suite 100, St. Rose, Louisiana 70087
Ph: (504) 602-2192, Fax: (504) 471-6111, www.intertek-cb.com

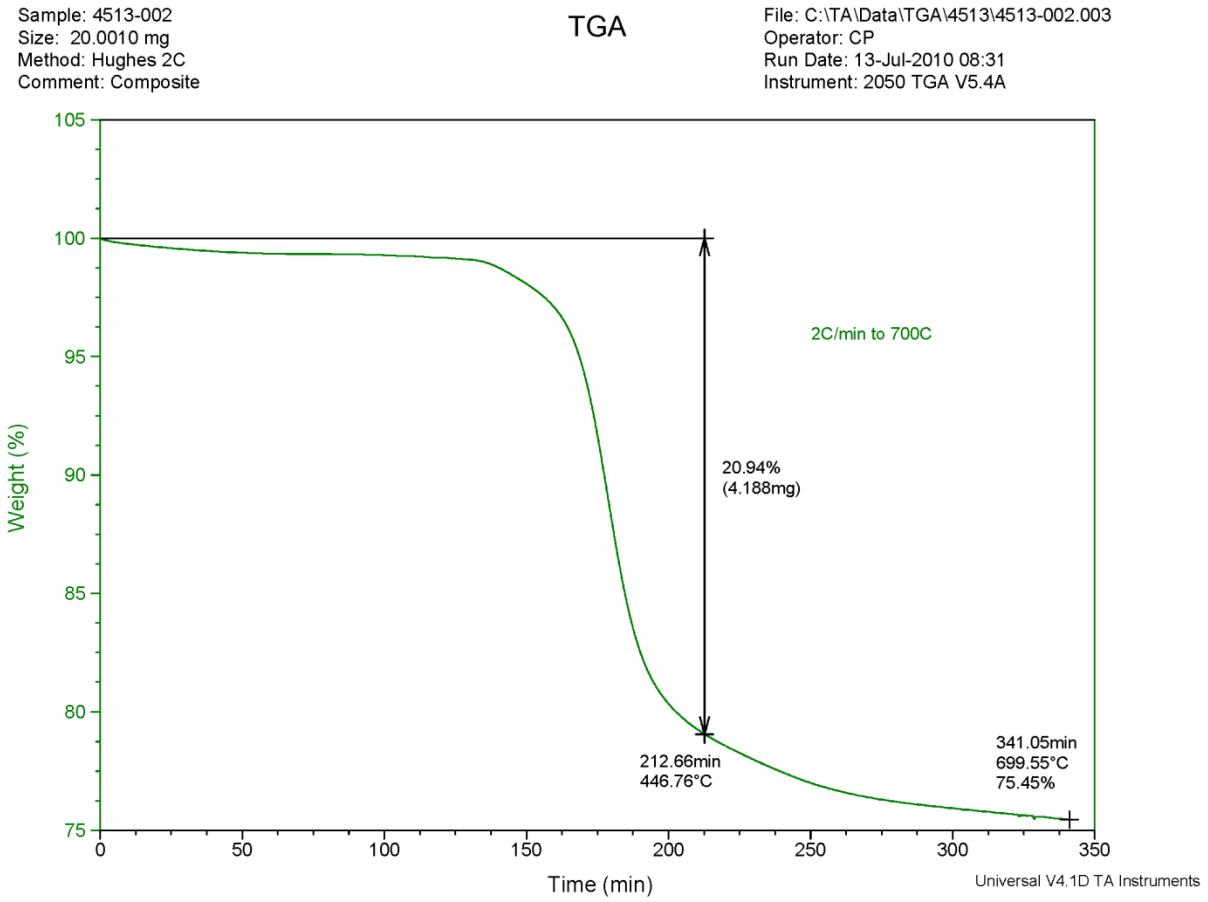

Sample: 4513-001
Size: 20.3710 mg
Method: Hughes 20C
Comment: Wood
TGA
File: C:\TA\Data\TGA\4513\4513-001.001
Operator: CP
Run Date: 12-Jul-2010 10:48
Instrument: 2050 TGA V5.4A
9.080%
(1.850mg)
6.31min
122.95°C
20C/min to 700C
62.21%
(12.67mg)
20.57min
410.19°C
36.81min
699.52°C
19.62%
Weight (%)
Time (min)
Universal V4.1D TA Instruments

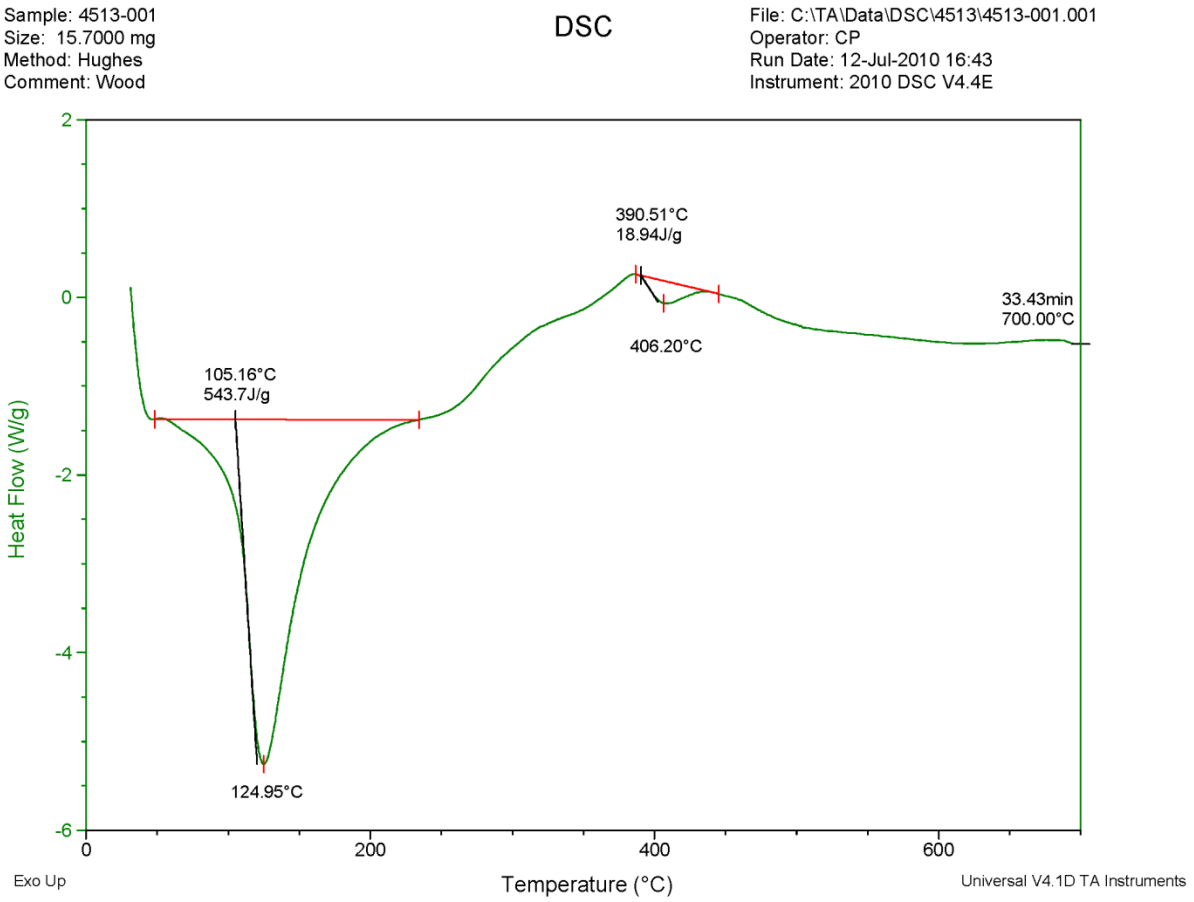

Sample: 4513-001
Size: 17.3370 mg
Method: Hughes 50C
Comment: Wood
TGA
File: C:\TA\Data\TGA\4513\4513-001.002
Operator: CP
Run Date: 12-Jul-2010 14:07
Instrument: 2050 TGA V5.4A
9.088%
(1.576mg)
3.45min
146.47°C
50C/min to 700C
64.65%
(11.21mg)
9.21min
426.85°C
17.41min
700.34°C
17.60%
Weight (%)
Time (min)
Universal V4.1D TA Instruments

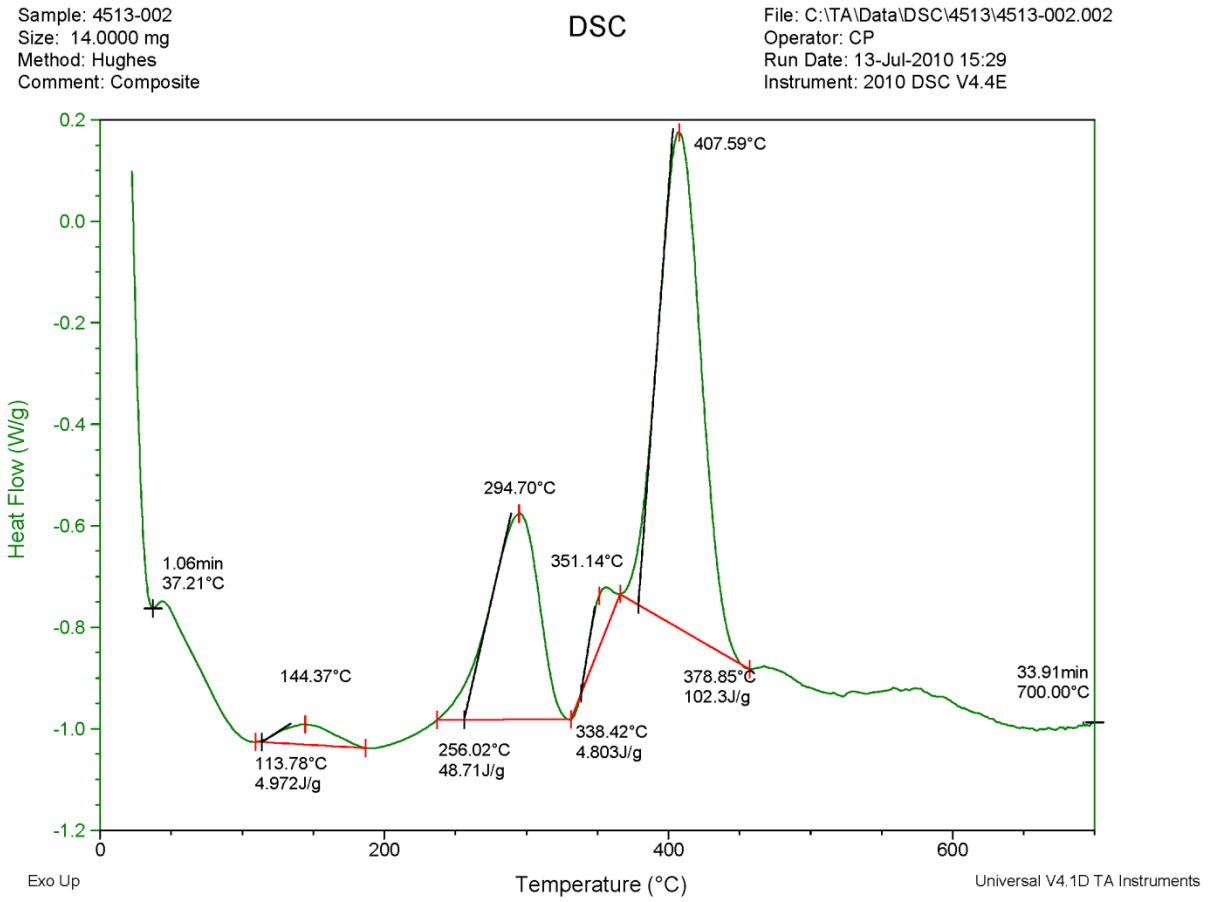

Sample: 4513-001
Size: 27.4880 mg
Method: Hughes 2C
Comment: Wood
TGA
File: C:\TA\Data\TGA\4513\4513-001.003
Operator: CP
Run Date: 12-Jul-2010 16:30
Instrument: 2050 TGA V5.4A
8.715%
(2.396mg)
29.99min
85.71°C
2C/min to 700C
59.46%
(16.34mg)
171.57min
368.94°C
338.77min
699.47°C
20.31%
Weight (%)
Time (min)
Universal V4.1D TA Instruments


Sample: 4513-002
Size: 20.3860 mg
Method: Hughes 20C
Comment: Composite
TGA
File: C:\TA\Data\TGA\4513\4513-002.001
Operator: CP
Run Date: 12-Jul-2010 12:14
Instrument: 2050 TGA V5.4A
20C/min to 700C
21.08%
(4.296mg)
24.30min
490.84°C
36.51min
699.53°C
76.47%
Weight (%)
Time (min)
Universal V4.1D TA Instruments

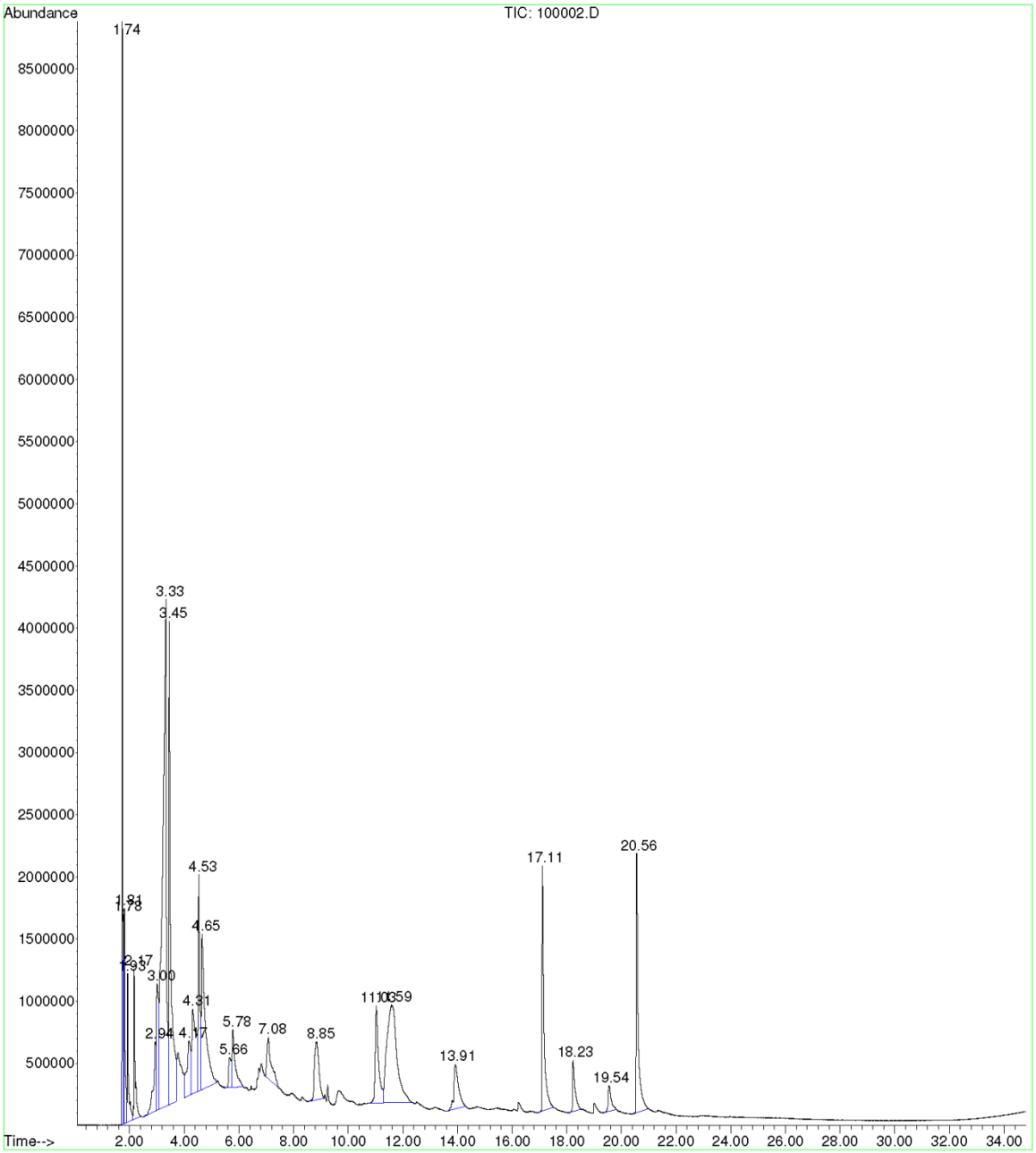

Sample: 4513-002
Size: 20.9670 mg
Method: Hughes 50C
Comment: Composite
TGA
File: C:\TA\Data\TGA\4513\4513-002.002
Operator: CP
Run Date: 12-Jul-2010 15:20
Instrument: 2050 TGA V5.4A
50C/min to 700C
21.69%
(4.549mg)
11.01min
522.42°C
16.37min
700.66°C
76.48%
Weight (%)
Time (min)
Universal V4.1D TA Instruments


Sample: 4513-002
Size: 20.0010 mg
Method: Hughes 2C
Comment: Composite
TGA
File: C:\TA\Data\TGA\4513\4513-002.003
Operator: CP
Run Date: 13-Jul-2010 08:31
Instrument: 2050 TGA V5.4A
2C/min to 700C
20.94%
(4.188mg)
212.66min
446.76°C
341.05min
699.55°C
75.45%
Weight (%)
Time (min)
Universal V4.1D TA Instruments


Sample: 4513-001
Size: 15.7000 mg
Method: Hughes
Comment: Wood
DSC
File: C:\TA\Data\DSC\4513\4513-001.001
Operator: CP
Run Date: 12-Jul-2010 16:43
Instrument: 2010 DSC V4.4E
390.51°C
18.94J/g
33.43min
700.00°C
406.20°C
105.16°C
543.7J/g
124.95°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.1D TA Instruments


Sample: 4513-002
Size: 14.0000 mg
Method: Hughes
Comment: Composite
DSC
File: C:\TA\Data\DSC\4513\4513-002.002
Operator: CP
Run Date: 13-Jul-2010 15:29
Instrument: 2010 DSC V4.4E
Heat Flow (W/g)
0.2
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
0
200
400
600
Temperature (°C)
Exo Up
Universal V4.1D TA Instruments
407.59°C
294.70°C
351.14°C
1.06min
37.21°C
144.37°C
378.85°C
102.3J/g
33.91min
700.00°C
338.42°C
4.803J/g
256.02°C
48.71J/g
113.78°C
4.972J/g

```
File        :C:\MSDChem\1\DATA\070710\100002.D
Operator    : SM
Acquired    :  7 Jul 2010  16:20     using AcqMethod HUGHES 070710
Instrument :   SP_GCMS_1
Sample Name: 4513-001
Misc Info   :
Vial Number: 1
```



Library Search Report

Data Path : C:\MSDChem\1\DATA\070710\
Data File : 10C002.D
Acq On : 7 Jul 2010 16:20
Operator : SM
Sample : 4513-001
Misc :
ALS Vial : 1 Sample Multiplier: 1

Search Libraries: C:\Database\NIST02.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: Chemstation Integrator - events.e

| Pk# | RT | Area% | Library/ID | Ref# | CAS# | Qual |
|---|---|---|---|---|---|---|
| 1 | 1.73 | 8.24 | C:\Database\NIST02.L | | | |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Ethylene oxide | 72 | 000075-21-8 | 3 |
| | | | Carbon dioxide | 78 | 000124-38-9 | 3 |
| 2 | 1.78 | 1.33 | C:\Database\NIST02.L | | | |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Ethylene oxide | 72 | 000075-21-8 | 3 |
| | | | Carbon dioxide | 78 | 000124-38-9 | 3 |
| 3 | 1.81 | 2.15 | C:\Database\NIST02.L | | | |
| | | | Ethylene oxide | 71 | 000075-21-8 | 5 |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Carbon dioxide | 78 | 000124-38-9 | 3 |
| 4 | 1.93 | 1.23 | C:\Database\NIST02.L | | | |
| | | | Acetone | 208 | 000067-64-1 | 9 |
| | | | Acetone | 205 | 000067-64-1 | 9 |
| | | | (3S)-(-)-3-Acetamidopyrrolidine | 11683 | 079286-74-1 | 9 |
| 5 | 2.17 | 1.03 | C:\Database\NIST02.L | | | |
| | | | 2,3-Butanedione | 1635 | 000431-03-8 | 72 |
| | | | 2,3-Butanedione | 1639 | 000431-03-8 | 50 |
| | | | Azetidine, 1-nitroso- | 1585 | 015216-10-1 | 9 |
| 6 | 2.23 | 0.49 | C:\Database\NIST02.L | | | |
| | | | Azetidine, 1-nitroso- | 1584 | 015216-10-1 | 10 |
| | | | Acetic acid ethenyl ester | 1651 | 000108-05-4 | 9 |
| | | | Acetic acid ethenyl ester | 1652 | 000108-05-4 | 9 |
| 7 | 2.94 | 1.69 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 255 | 000064-19-7 | 27 |
| | | | 2 Pentanone | 1686 | 000107-87-9 | 17 |
| | | | Acetic acid | 253 | 000064-19-7 | 16 |
| 8 | 3.00 | 2.86 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 254 | 000064-19-7 | 64 |
| | | | Acetic acid | 252 | 000064-19-7 | 59 |
| | | | Acetic acid | 256 | 000064-19-7 | 58 |
| 9 | 3.33 | 18.54 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 254 | 000064-19-7 | 90 |
| | | | Acetic acid | 253 | 000064-19-7 | 90 |
| | | | Acetic acid | 256 | 000064-19-7 | 90 |
| 10 | 3.45 | 8.97 | C:\Database\NIST02.L | | | |
| | | | 2-Propanone, 1-hydroxy- | 800 | 000116-09-6 | 9 |
| | | | Ethanamine, 2-methoxy- | 873 | 000109-85-3 | 9 |
| | | | 2 Propanone, 1 hydroxy | 799 | 000116-09-6 | 5 |
| 11 | 4.17 | 2.18 | C:\Database\NIST02.L | | | |
| | | | Hydroperoxide, 1-methylpentyl | 8581 | 024254-55-5 | 42 |
| | | | Hexane, 1-nitro- | 13416 | 000646-14-0 | 38 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | 1-Hepten-4-ol | 7258 | 003521-01-3 | 32 |
| 12 | 4.31 | 3.14 | C:\Database\NIST02.L | | | | |
| | | | | Methanecarbothiolic acid | 896 | 000507-09-5 | 37 |
| | | | | 2-Butanone, 1-(acetyloxy)- | 12810 | 001575-57-1 | 28 |
| | | | | Hexane, 3-bromo- | 32155 | 000377-87-5 | 25 |
| 13 | 4.53 | 3.79 | C:\Database\NIST02.L | | | | |
| | | | | Pentanol, 5-amino- | 4494 | 002508-29-4 | 9 |
| | | | | Propanoic acid, 2 oxo , methyl est er | 4148 | 000600 22 6 | 9 |
| | | | | 3-Amino-2-oxazolidinone | 4101 | 000080-65-9 | 9 |
| 14 | 4.66 | 5.97 | C:\Database\NIST02.L | | | | |
| | | | | 1H-Pyrazole, 3,5-dimethyl- | 2703 | 000067-51-6 | 80 |
| | | | | 1H-Imidazole, 4,5-dimethyl- | 2713 | 002302-39-8 | 80 |
| | | | | Furfural | 2683 | 000098-01-1 | 74 |
| 15 | 5.66 | 0.69 | C:\Database\NIST02.L | | | | |
| | | | | 1,6;2,3-Dianhydro-4-O-acetyl-.beta .-d-mannopyranose | 47172 | 1000139-89-7 | 53 |
| | | | | 1,6;2,3-Dianhydro-4-O-acetyl-.beta .-d-gulopyranose | 47170 | 1000139-89-8 | 53 |
| | | | | 1,6;2,3-Dianhydro-4-O-acetyl-.beta .-d-allopyranose | 47166 | 1000139-89-9 | 50 |
| 16 | 5.77 | 1.89 | C:\Database\NIST02.L | | | | |
| | | | | Methyl 1-methylcyclopropyl ketone | 3211 | 001567-75-5 | 47 |
| | | | | 2-Pentanone, 3-methylene- | 3175 | 004359-77-7 | 43 |
| | | | | 2,3-Dihydrooxazole, 2-t-butyl-4-(1 -hydroxy-1-methylethyl)-3-methoxyc arbonyl-5-methyl- | 3226 | 036808-01-2 | 38 |
| 17 | 6.74 | 0.50 | C:\Database\NIST02.L | | | | |
| | | | | Butyrolactone | 1629 | 000096-48-0 | 43 |
| | | | | Butyrolactone | 1625 | 000096-48-0 | 43 |
| | | | | Butyrolactone | 1623 | 000096-48-0 | 43 |
| 18 | 6.82 | 0.98 | C:\Database\NIST02.L | | | | |
| | | | | 1H-Imidazole, 4,5-dihydro-2-methyl | 1349 | 000534-26-9 | 50 |
| | | | | 2H-Pyran, 3,4-dihydro- | 1397 | 000110-87-2 | 49 |
| | | | | 3 Hexene, (E) | 1444 | 013269 52 8 | 46 |
| 19 | 7.08 | 3.18 | C:\Database\NIST02.L | | | | |
| | | | | 4H-1,2,4-Triazol-3-amine, 4-methyl | 2973 | 016681-76-8 | 47 |
| | | | | 2-Hexene | 1431 | 000592-43-8 | 45 |
| | | | | 2-Hexene, (Z)- | 1446 | 007688-21-3 | 45 |
| 20 | 8.85 | 2.95 | C:\Database\NIST02.L | | | | |
| | | | | 2,4 Imidazolidinedione, 3 methyl | 6903 | 006843 45 4 | 72 |
| | | | | 3-Cyclobutene-1,2-dione, 3,4-dihyd roxy- | 6883 | 002892-51-5 | 59 |
| | | | | 2H-Pyran, 3,4-dihydro-2-methoxy- | 7080 | 004454-05-1 | 59 |
| 21 | 9.24 | 0.28 | C:\Database\NIST02.L | | | | |
| | | | | D-Limonene | 15138 | 005989-27-5 | 86 |
| | | | | D-Limonene | 15136 | 005989-27-5 | 83 |
| | | | | Limonene | 15123 | 000138 86 3 | 70 |
| 22 | 9.66 | 0.68 | C:\Database\NIST02.L | | | | |
| | | | | Triethylenediamine | 6124 | 000280-57-9 | 43 |
| | | | | 2-Butenal, dimethylhydrazone | 6133 | 007422-95-9 | 35 |
| | | | | 3-Heptene, 3-methyl- | 6507 | 007300-03-0 | 35 |
| 23 | 11.03 | 3.32 | C:\Database\NIST02.L | | | | |
| | | | | Phenol, 2 methoxy | 10079 | 000090 05 1 | 95 |
| | | | | Mequinol | 10070 | 000150-76-5 | 94 |
| | | | | Mequinol | 10069 | 000150-76-5 | 93 |
| 24 | 11.58 | 10.01 | C:\Database\NIST02.L | | | | |

```
                 sec-Butylamine                          740 013952-84-6 40
                 Urea, trimethyl                        4117 000632 14 4 38
                 2-Butanamine, 3-methyl-                1886 000598-74-3 37

 25  13.91  2.16 C:\Database\NIST02.L
                 Phenol, 2-methoxy-4-methyl-          16723 000093-51-6 96
                 Phenol, 2-methoxy-4-methyl-          16717 000093-51-6 96
                 Phenol, 2-methoxy-4-methyl-          16722 000093-51-6 93

 26  17.11  4.78 C:\Database\NIST02.L
                 2-Methoxy-4-vinylphenol              23329 007786-61-0 94
                 4-Hydroxy-3-methylacetophenone       23387 000876-02-8 87
                 Ethanone, 1-(2-hydroxy-5-methylphe   23439 001450-72-2 86
                 nyl)-

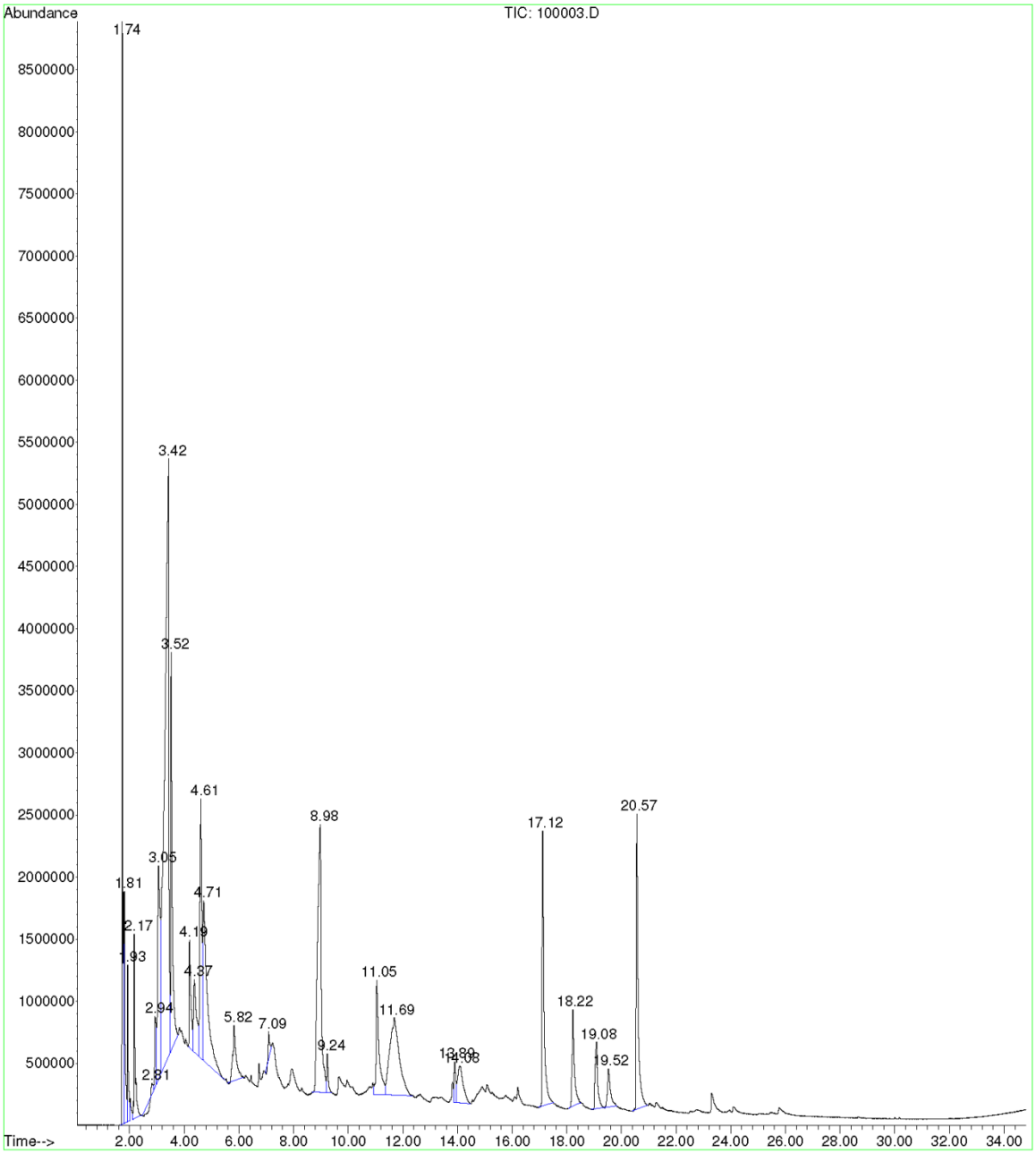
 27  18.23  1.33 C:\Database\NIST02.L
                 Eugenol                              31517 000097-53-0 98
                 Eugenol                              31518 000097 53 0 98
                 Phenol, 2-methoxy-3-(2-propenyl)-    31637 001941-12-4 98

 28  19.54  0.66 C:\Database\NIST02.L
                 Phenol, 2-methoxy-4-(1-propenyl)-,   31685 005912-86-7 94
                  (Z)-
                 Phenol, 2-methoxy-4-(1-propenyl)-    31635 000097-54-1 94
                 Vanillin                             24644 000121-33-5 90

 29  20.56  4.97 C:\Database\NIST02.L
                 Phenol, 2-methoxy-4-(1-propenyl)-,   31685 005912-86-7 98
                  (Z)-
                 Phenol, 2-methoxy-4-(1-propenyl)-    31635 000097-54-1 98
                 Phenol, 2-methoxy-4-(1-propenyl)-,   31637 005932-68-3 98
                  (E)-

DEFAULT.M Mon Jul 19 10:47:17 2010
```

```
File        :C:\MSDChem\1\DATA\070710\100003.D
Operator    : SM
Acquired    :  7 Jul 2010  17:14     using AcqMethod HUGHES 070710
Instrument  :   SP_GCMS_1
Sample Name: 4513-001
Misc Info   :
Vial Number: 2
```



Library Search Report

Data Path : C:\MSDChem\1\DATA\070710\
Data File : 10C003.D
Acq On : 7 Jul 2010 17:14
Operator : SM
Sample : 4513-001
Misc :
ALS Vial : 2 Sample Multiplier: 1

Search Libraries: C:\Database\NIST02.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: Chemstation Integrator - autoint1.e

| Pk# | RT | Area% | Library/ID | Ref# | CAS# | Qual |
|---|---|---|---|---|---|---|
| 1 | 1.74 | 9.12 | C:\Database\NIST02.L | | | |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Ethylene oxide | 72 | 000075-21-8 | 3 |
| | | | Carbon dioxide | 79 | 000124-38-9 | 3 |
| 2 | 1.81 | 2.12 | C:\Database\NIST02.L | | | |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Ethyne, fluoro- | 73 | 002713-09-9 | 3 |
| | | | Carbon dioxide | 78 | 000124-38-9 | 3 |
| 3 | 1.93 | 1.14 | C:\Database\NIST02.L | | | |
| | | | Acetone | 205 | 000067-64-1 | 9 |
| | | | Acetone | 208 | 000067-64-1 | 9 |
| | | | Acetone | 206 | 000067-64-1 | 7 |
| 4 | 2.17 | 1.74 | C:\Database\NIST02.L | | | |
| | | | 2,3-Butanedione | 1635 | 000431-03-8 | 64 |
| | | | 2,3-Butanedione | 1639 | 000431-03-8 | 59 |
| | | | 2-Pentanone | 1685 | 000107-87-9 | 9 |
| 5 | 2.81 | -0.18 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 256 | 000064-19-7 | 53 |
| | | | Acetic acid | 253 | 000064-19-7 | 53 |
| | | | Acetic acid | 255 | 000064-19-7 | 53 |
| 6 | 2.94 | 0.93 | C:\Database\NIST02.L | | | |
| | | | 1,2-Ethanediamine, N,N-diethyl- | 8037 | 000100-36-7 | 43 |
| | | | Diethylaminoacetone | 12516 | 001620-14-0 | 43 |
| | | | N-tert-Butylethylamine | 4088 | 004432-77-3 | 38 |
| 7 | 3.05 | 5.03 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 254 | 000064-19-7 | 53 |
| | | | Acetic acid | 253 | 000064-19-7 | 50 |
| | | | Acetic acid | 255 | 000064-19-7 | 50 |
| 8 | 3.41 | 22.36 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 253 | 000064-19-7 | 90 |
| | | | Acetic acid | 254 | 000064-19-7 | 90 |
| | | | Acetic acid | 256 | 000064-19-7 | 90 |
| 9 | 3.52 | 5.25 | C:\Database\NIST02.L | | | |
| | | | 2-Propanone, 1-hydroxy- | 800 | 000116-09-6 | 45 |
| | | | Ethanamine, 2-methoxy- | 873 | 000109-85-3 | 9 |
| | | | 2-Propanol, 1-amino- | 868 | 000078-96-6 | 5 |
| 10 | 4.19 | 1.69 | C:\Database\NIST02.L | | | |
| | | | 1-Butanol, 2-nitro- | 8759 | 000609-31-4 | 40 |
| | | | 1,2-Cyclopentanediol, trans- | 4216 | 005057-09-8 | 9 |
| | | | 1,2-Cyclopentanediol, trans- | 4221 | 005057-99-8 | 9 |
| 11 | 4.37 | 2.04 | C:\Database\NIST02.L | | | |
| | | | Propanoic acid, 2-methyl-, butyl e ster | 20098 | 000097-87-0 | 33 |

```
                Pentane, 3-ethyl-3-methyl-          7472 000609-26-7 38
                1 Butanol, 3 methyl , acetate      13036 000123 92 2 38

12   4.60  4.47 C:\Database\NIST02.L
                Propanoic acid, 2-oxo-, methyl est  4149 000600-22-6  9
                er
                Propanoic acid, 2-oxo-, methyl est  4147 000600-22-6  9
                er
                Propanoic acid, 2-methyl-, methyl   4255 000547-63-7  9
                ester

13   4.71  6.40 C:\Database\NIST02.L
                1H-Imidazole, 4,5-dimethyl-         2713 002302-39-8 72
                1H-Pyrazole, 3,5-dimethyl-          2707 000067-51-6 64
                Pyrazole, 1,4-dimethyl-             2698 001072-68-0 64

14   5.83  1.68 C:\Database\NIST02.L
                Methyl 1 methylcyclopropyl ketone   3211 001567 75 5 43
                2-Propanone, 1-(acetyloxy)-         7817 000592-20-1 43
                4-Penten-2-one, 3-methyl-           3162 000758-87-2 38

15   7.09  0.27 C:\Database\NIST02.L
                Aziridine, 2-methyl-3-(1-methyleth 17252 055669-77-7 37
                yl)-1-(2-propenyl)-, trans-
                Cyclohexanone                       3035 000108-94-1 32
                2 Hexene                            1431 000592 43 8 30

16   8.98  9.20 C:\Database\NIST02.L
                2,4-Imidazolidinedione, 3-methyl-   6903 006843-45-4 72
                1,4-Butanediamine, 2,3-dimethoxy-N 58184 026549-21-3 56
                ,N,N',N'-tetramethyl-, [S-(R*,R*)]
                N-tert-Butylacetamide               7605 000762-84-5 38

17   9.24  0.51 C:\Database\NIST02.L
                D-Limonene                         15138 005989-27-5 90
                D-Limonene                         15136 005989-27-5 90
                D-Limonene                         15139 005989-27-5 89

18  11.05  3.08 C:\Database\NIST02.L
                Phenol, 2-methoxy-                 10079 000090-05-1 95
                Mequinol                           10070 000150-76-5 94
                Mequinol                           10069 000150 76 5 90

19  11.69  6.65 C:\Database\NIST02.L
                1-Guanidinosuccinimide             18125 1000130-20-8 53
                2-Butanamine, (S)-                   741 000513-48-5 47
                sec-Butylamine                       740 013952-84-6 38

20  13.89  0.62 C:\Database\NIST02.L
                Phenol, 2 methoxy 4 methyl         16723 000093 51 6 98
                Phenol, 2-methoxy-4-methyl-        16717 000093-51-6 96
                Phenol, 2-methoxy-4-methyl-        16722 000093-51-6 93

21  14.08  1.88 C:\Database\NIST02.L
                Piperazine, 1,4-dimethyl-           7167 000106-58-1 37
                Cyclohexanone, 3-hydroxy-           7013 000823-19-8 30
                Oxirane, [(1-methylethoxy)methyl]-  7963 004016-14-2 22

22  17.12  4.53 C:\Database\NIST02.L
                2-Methoxy-4-vinylphenol            23329 007786-61-0 95
                4-Hydroxy-3-methylacetophenone     23387 000876-02-8 87
                4-Hydroxy-3-methylacetophenone     23388 000876-02-8 83

23  18.22  1.93 C:\Database\NIST02.L
                Phenol, 2-methoxy-3-(2-propenyl)-  31637 001941-12-4 98
                Eugenol                            31518 000097 53 0 98
                Eugenol                            31517 000097-53-0 98

24  19.08  1.55 C:\Database\NIST02.L
                Propanamide, 2-methoxy-N-methyl-    8280 1000196-12-6 27
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                Silane, ethyldimethyl-                1969 000758-21-4 87
                Silane, diethyldimethyl               8061 000756 81 0 25

 25  19.52  1.02 C:\Database\NIST02.L
                Phenol, 2-methoxy-4-(1-propenyl)-   31650 000097-54-1 92
                Phenol, 2-methoxy-4-(1-propenyl)-,  31685 005912-86-7 91
                 (Z)-
                Phenol, 2-methoxy-4-(1-propenyl)-   31635 000097-54-1 91

 26  20.57  5.00 C:\Database\NIST02.L
                Phenol, 2-methoxy-4-(1-propenyl)-   31635 000097-54-1 98
                Phenol, 2-methoxy-4-(1-propenyl)-,  31685 005912-86-7 98
                 (Z)-
                Phenol, 2-methoxy-4-(1-propenyl)-,  31687 005932-68-3 98
                 (E)-

POLYMERTD121608.M Mon Jul 19 10:59:28 2010
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File        :C:\MSDChem\1\DATA\070710\100002.D
Operator    : SM
Acquired    :  7 Jul 2010  16:20     using AcqMethod HUGHES 070710
Instrument :   SP_GCMS_1
Sample Name: 4513-001
Misc Info  :
Vial Number: 1
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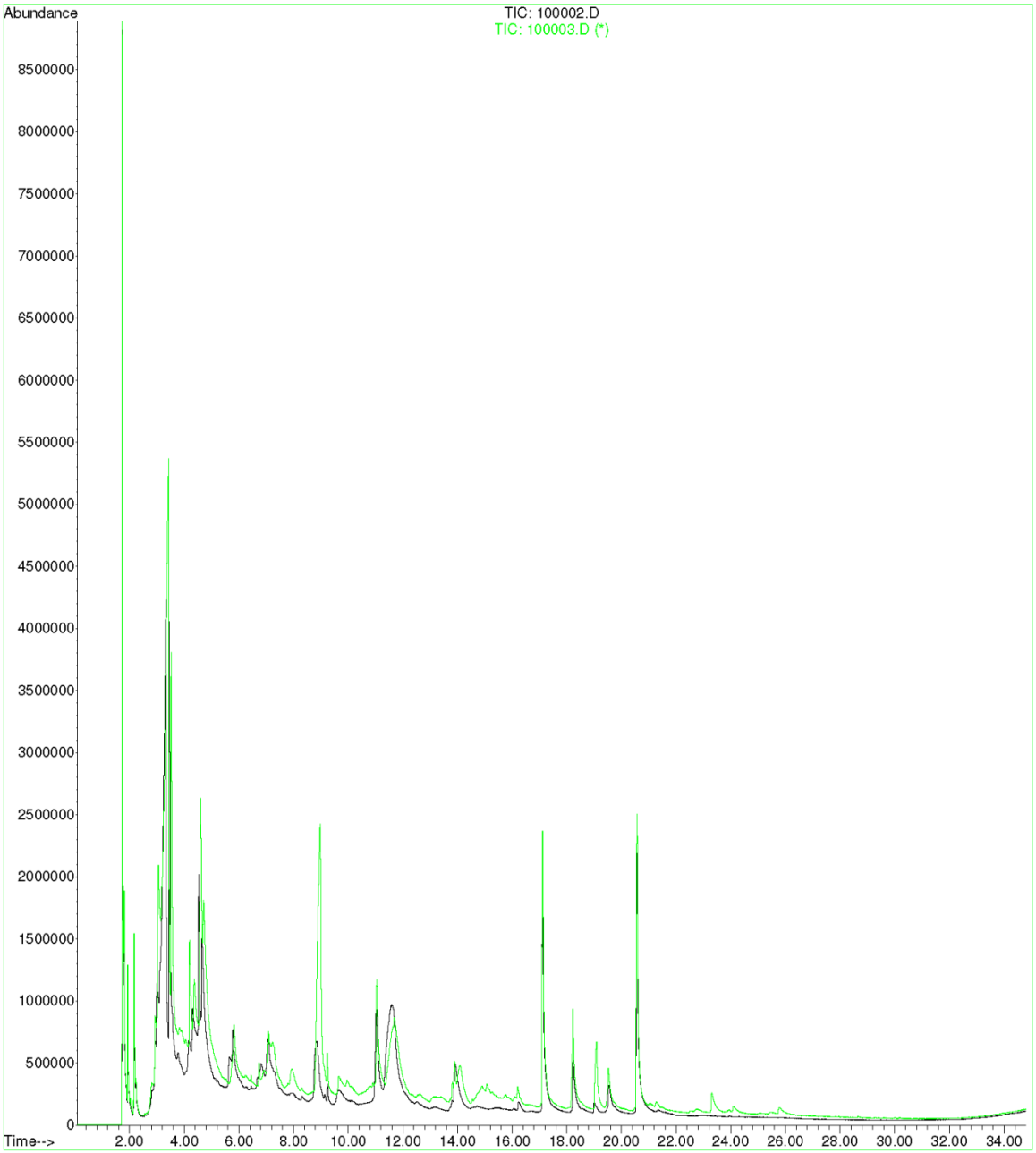

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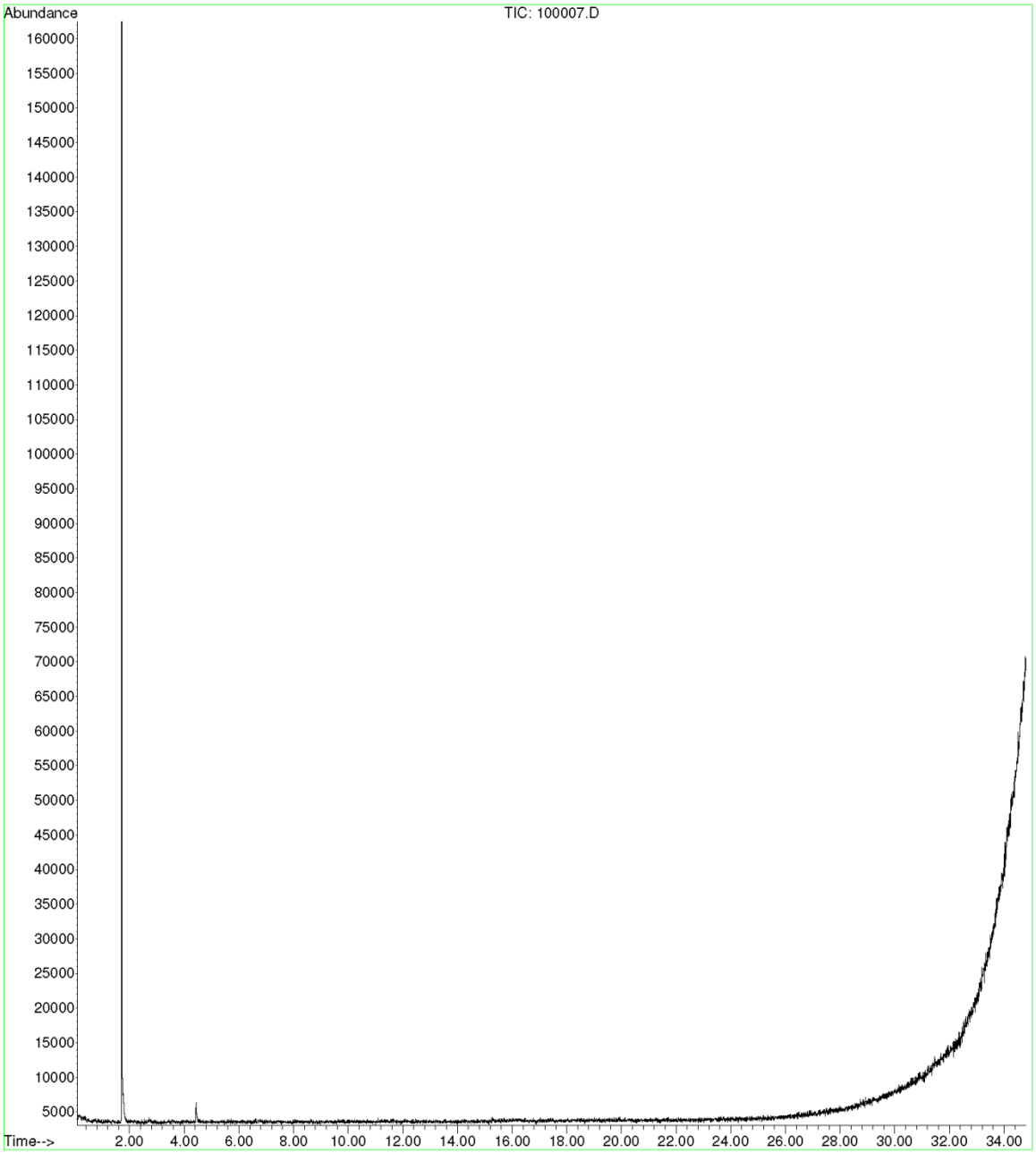
File        :C:\MSDChem\1\DATA\070710\100007.D
Operator    : SM
Acquired    :  8 Jul 2010  16:08     using AcqMethod HUGHES 070710
Instrument :   SP_GCMS_1
Sample Name: test
Misc Info   :
Vial Number: 6
```



Library Search Report

Data Path : C:\MSDChem\1\DATA\070710\
Data File : 100007.D
Acq On : 8 Jul 2010 16:08
Operator : SM
Sample : test
Misc :
ALS Vial : 6 Sample Multiplier: 1

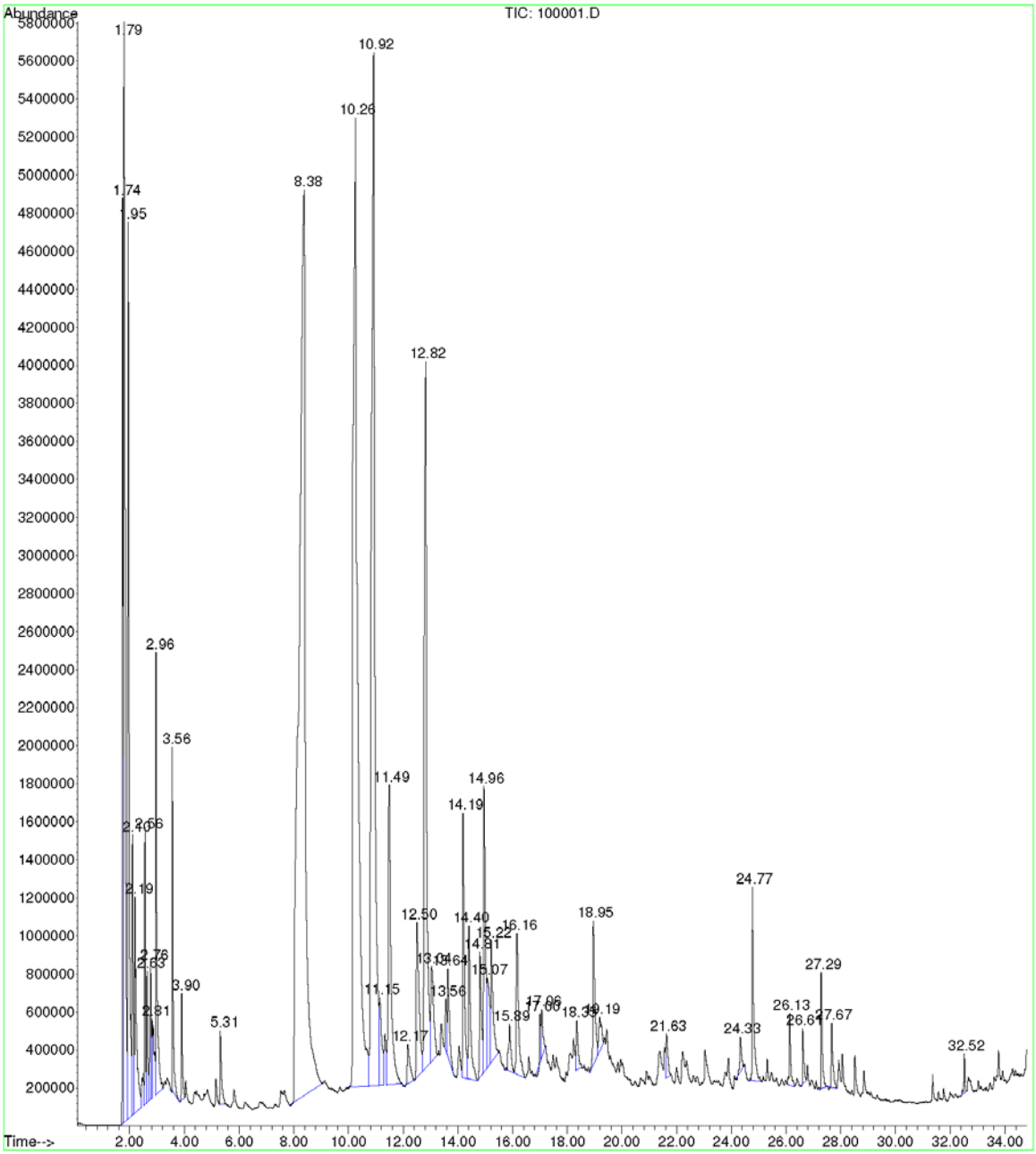
Search Libraries: C:\Database\NIST02.L Minimum Quality: 0

Unknown Spectrum: Peak average
Integration Events: Chemstation Integrator - autoint1.e

| Pk# | RT | Area% | Library/ID | Ref# | CAS# | Qual |
|---|---|---|---|---|---|---|
| 1 | 1.71 | 100.00 | C:\Database\NIST02.L | | | |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Carbon dioxide | 78 | 000124-38-9 | 3 |
| | | | Nitrous Oxide | 79 | 010024-97-2 | 3 |

POLYMERTD121608.M Sun Jul 25 11:34:41 2010

```
File        :C:\MSDChem\1\DATA\071210\100001.D
Operator    : SM
Acquired    : 12 Jul 2010  10:55     using AcqMethod HUGHES 070710
Instrument :   SP_GCMS_1
Sample Name: 4513-002
Misc Info   :
Vial Number: 1
```



Library Search Report

Data Path : C:\MSDChem\1\DATA\071210\
Data File : 10C001.D
Acq On : 12 Jul 2010 10:55
Operator : SM
Sample : 4513-002
Misc :
ALS Vial : 1 Sample Multiplier: 1

Search Libraries: C:\Database\NIST02.L Minimum Quality: 0

Unknown Spectrum: Peak average
Integration Events: Chemstation Integrator - autoint1.e

| Pk# | RT | Area% | Library/ID | Ref# | CAS# | Qual |
|---|---|---|---|---|---|---|
| 1 | 1.74 | 2.71 | C:\Database\NIST02.L | | | |
| | | | 1-Propanol | 280 | 000071-23-8 | 5 |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Acetaldehyde | 69 | 000075-07-0 | 4 |
| 2 | 1.79 | 6.21 | C:\Database\NIST02.L | | | |
| | | | Aminomethanesulfonic acid | 6020 | 013881-91-9 | 40 |
| | | | L-Alanine, 3-sulfo- | 35516 | 000498-40-8 | 40 |
| | | | Sulfur dioxide | 346 | 007446 09 5 | 38 |
| 3 | 1.95 | 4.08 | C:\Database\NIST02.L | | | |
| | | | 2-Propenal | 180 | 000107-02-8 | 30 |
| | | | Acetone | 207 | 000067-64-1 | 9 |
| | | | Diazene, dimethyl- | 202 | 000503-28-6 | 9 |
| 4 | 2.10 | 1.13 | C:\Database\NIST02.L | | | |
| | | | Chloromethyl propanoate | 9430 | 005402 53 9 | 38 |
| | | | 2-Propen-1-ol | 214 | 000107-18-6 | 9 |
| | | | Propane, 2-methyl-2-nitro- | 4465 | 000594-70-7 | 9 |
| 5 | 2.19 | 1.35 | C:\Database\NIST02.L | | | |
| | | | 2-Propenoic acid, pentyl ester | 18934 | 002998-23-4 | 32 |
| | | | 2-Pentene, (E)- | 544 | 000646-04-8 | 30 |
| | | | 2-Pentene, (Z)- | 546 | 000627-20-3 | 30 |
| 6 | 2.56 | 0.71 | C:\Database\NIST02.L | | | |
| | | | Benzene | 1004 | 000071-43-2 | 91 |
| | | | Benzene | 1001 | 000071-43-2 | 91 |
| | | | Benzene | 1003 | 000071-43-2 | 91 |
| 7 | 2.63 | 0.51 | C:\Database\NIST02.L | | | |
| | | | Furan, 2,5-dihydro-3-methyl- | 1419 | 001708-31-2 | 38 |
| | | | 2 Butanone, 4 hydroxy 3 (hydroxyme | 13861 | 006868 97 9 | 35 |
| | | | thyl)-3-methyl- | | | |
| | | | 3-Buten-2-one, 3-methyl- | 1406 | 000814-78-8 | 30 |
| 8 | 2.76 | 0.58 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 256 | 000064-19-7 | 47 |
| | | | Acetic acid | 253 | 000064-19-7 | 9 |
| | | | Acetic acid | 252 | 000064-19-7 | 9 |
| 9 | 2.82 | 0.29 | C:\Database\NIST02.L | | | |
| | | | 1,3,4-Oxadiazole, 2-(acetyloxy)-2, | 37714 | 066398-57-0 | 47 |
| | | | 5-dihydro-2,5,5-trimethyl- | | | |
| | | | 3-Buten-2-ol, 3-methyl- | 1754 | 010473-14-0 | 23 |
| | | | Methyl Isobutyl Ketone | 3732 | 000108-10-1 | 14 |
| 10 | 2.96 | 1.62 | C:\Database\NIST02.L | | | |
| | | | 2 Propanone, 1 hydroxy | 890 | 000116 09 6 | 38 |
| | | | Ethanamine, 2-methoxy- | 873 | 000109-85-3 | 9 |
| | | | Ethanamine, 2-methoxy- | 875 | 000109-85-3 | 5 |
| 11 | 3.56 | 1.10 | C:\Database\NIST02.L | | | |

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                Toluene                                2405 000108-88-3 95
                Toluene                                2402 000108-88-3 90
                Toluene                                2403 000108-88-3 90

12   3.90  0.38 C:\Database\NIST02.L
                2-Octene, (Z)-                         6488 007642-04-8 87
                2-Octene                               6450 000111-67-1 83
                2-Octene, (E)-                         6467 013389-42-9 83

13   5.31  0.40 C:\Database\NIST02.L
                o-Xylene                               4959 000095-47-6 95
                p-Xylene                               4957 000106-42-3 95
                p-Xylene                               4954 000106-42-3 95

14   8.38 21.42 C:\Database\NIST02.L
                Phenol                                 2538 000108-95-2 95
                Phenol                                 2536 000108-95-2 95
                Phenol                                 2540 000108-95-2 95

15  10.25 16.22 C:\Database\NIST02.L
                Phenol, 2-methyl-                      5243 000095-48-7 97
                Phenol, 2-methyl-                      5244 000095-48-7 96
                Phenol, 4-methyl-                      5240 000106-44-5 95

16  10.92 12.87 C:\Database\NIST02.L
                Phenol, 4-methyl-                      5235 000106-44-5 96
                Phenol, 4-methyl-                      5230 000106-44-5 94
                Phenol, 4-methyl-                      5240 000106-44-5 94

17  11.14  0.79 C:\Database\NIST02.L
                Phenol, 4-methyl-                      5235 000106-44-5 95
                Phenol, 4-methyl-                      5240 000106-44-5 95
                Phenol, 2-methyl-                      5243 000095-48-7 90

18  11.49  3.09 C:\Database\NIST02.L
                Phenol, 2,6-dimethyl-                  9624 000576-26-1 96
                Phenol, 2,6-dimethyl-                  9613 000576-26-1 95
                Phenol, 2,6-dimethyl-                  9618 000576-26-1 95

19  12.17  0.37 C:\Database\NIST02.L
                o-Toluidine                            5027 000095-53-4 62
                Aniline, N-methyl-                     5041 000100-61-8 58
                m-Ethylaniline                         9257 000587-02-0 58

20  12.50  1.57 C:\Database\NIST02.L
                Phenol, 3-ethyl-                       9600 000620-17-7 93
                Phenol, 3-ethyl-                       9593 000620-17-7 90
                Phenol, 3-ethyl-                       9599 000620-17-7 90

21  12.82  6.19 C:\Database\NIST02.L
                Phenol, 3,5-dimethyl-                  9620 000108-68-9 97
                Phenol, 2,4-dimethyl-                  9623 000105-67-9 96
                Phenol, 2,4-dimethyl-                  9616 000105-67-9 96

22  13.04  1.02 C:\Database\NIST02.L
                Benzenamine, N,4-dimethyl-             9285 000623-08-5 50
                Benzenamine, 3,4-dimethyl-             9316 000095-64-7 43
                Benzenamine, 3,4-dimethyl-             9315 000095-64-7 43

23  13.56  0.27 C:\Database\NIST02.L
                Benzenamine, N,N,3-trimethyl-         15068 000121-72-2 62
                Benzenamine, N,N,4-trimethyl-         15070 000099-97-8 60
                Benzenamine, N,N,4-trimethyl-         15071 000099-97-8 50

24  13.64  0.62 C:\Database\NIST02.L
                Phenol, 2-ethyl-4-methyl-             15856 003855-26-3 43
                Benzenemethanol, .alpha.,4-dimethy    15928 000536-50-5 41
                l-
                Phenol, 2-ethyl-6-methyl-             15852 001687-64-5 38
```

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25  14.19  1.65 C:\Database\NIST02.L
               Phenol, 2,4,6 trimethyl              15836 000527 60 6 92
               3,4-Dimethylanisole                  15810 004685-47-6 78
               Phenol, 2,3,6-trimethyl-             15834 002416-94-6 64

26  14.40  1.18 C:\Database\NIST02.L
               2-Propenal, 2-methyl-3-phenyl-       20660 000101-39-3 60
               1H-Benzimidazole, 2-ethyl-           21277 001848-84-6 58
               Benzofuran, 4,7-dimethyl-            20650 028715-26-6 53

27  14.81  0.86 C:\Database\NIST02.L
               Pyridine, 3,5-dimethyl-               5071 000591-22-0 53
               Phenol, 2-propyl-                    15801 000644-35-9 53
               Phenol, 2-propyl-                    15797 000644-35-9 53

28  14.96  1.89 C:\Database\NIST02.L
               Phenol, 2-ethyl-4-methyl-            15856 003855-26-3 90
               Benzene, 1 ethyl 4 methoxy           15883 001515 95 3 55
               Phenol, 2-ethyl-6-methyl-            15852 001687-64-5 55

29  15.08  0.88 C:\Database\NIST02.L
               Isoquinoline                         12651 000119-65-3 53
               Quinoline                            12647 000091-22-5 53
               Quinoline                            12648 000091-22-5 53

30  15.22  1.03 C:\Database\NIST02.L
               Isoquinoline                         12651 000119-65-3 89
               Quinoline                            12645 000091-22-5 86
               Benzeneacetonitrile, .alpha.-methy   12656 000495-10-3 86
               lene-

31  15.89  0.40 C:\Database\NIST02.L
               2-Methyl-6-propylphenol              22643 003520-52-3 50
               Phenol, 2 ethyl 6 methyl             15853 001687 64 5 46
               Phenol, 4-(1-methylpropyl)-          22663 000099-71-8 45

32  16.16  1.16 C:\Database\NIST02.L
               1H-Pyrrole, 1-phenyl-                19297 000635-90-5 93
               1H-Pyrrole, 1-phenyl-                19299 000635-90-5 78
               Isoquinoline, 3-methyl-              19302 001125-80-0 64

33  17.00  0.14 C:\Database\NIST02.L
               Benzene, 1-(2-butenyl)-2,3-dimethy   29406 054340-85-1 55
               l-
               Benzene, 1,3,5-trimethyl-2-(1-meth   29452 014679-13-1 55
               ylethenyl)-
               Benzene, 4-(2-butenyl)-1,2-dimethy   29441 054340-86-2 55
               l-, (E)-

34  17.06  0.30 C:\Database\NIST02.L
               Phenol, 4-(1-methylpropyl)-          22663 000099-71-8 38
               Phenol, 4-(1-methylpropyl)-          22670 000099-71-8 38
               Tricyclo[3.1.0.0(2,4)]hexane, 3,3,   15388 058987-01-2 35
               6,6-tetramethyl-, (1.alpha.,2.beta
               .,4.beta.,5.alpha.)-

35  18.35  0.36 C:\Database\NIST02.L
               Quinoline, 6 methyl                  19285 000091 62 3 80
               Quinoline, 8-methyl-                 19296 000611-32-5 42
               Quinoline, 7-methyl-                 19295 000612-60-2 42

36  18.95  1.07 C:\Database\NIST02.L
               1H-Pyrrole, 1-(4-methylphenyl)-      27870 000827-60-1 64
               Acetonitrile, 2-(pyrido[1,2-a]imid   28134 057892-77-0 62
               azol-2-yl)-
               1H Pyrrole, 1 (4 methylphenyl)       27871 000827 60 1 53

37  19.19  0.34 C:\Database\NIST02.L
               1H-Indole, 3-methyl-                 13513 000083-34-1 18
               1,5-Naphthyridine                    13325 000254-79-5 15
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                 1H-Indole, 2-methyl-                13507 000095-20-5 14

 38  21.63  0.31 C:\Database\NIST02.L
                 Indolizine, 2,5-dimethyl-           20376 000769-83-5 52
                 1H-Indole, 2,5-dimethyl-            20364 001196-79-8 46
                 Indolizine, 2,3-dimethyl-           20378 000769-65-3 46

 39  24.33  0.22 C:\Database\NIST02.L
                 4-(4-Methylphenyl)pyridine          35495 004423-10-3 90
                 3 Phenyl 3 (2 pyridyl) N,N dimethy  82564 000086 21 5 70
                 lpropylamine
                 [1,1'-Biphenyl]-2-amine             35481 000090-41-5 60

 40  24.77  1.08 C:\Database\NIST02.L
                 9H-Xanthene                         44103 000092-83-1 95
                 9H-Xanthene                         44104 000092-83-1 94
                 9H-Xanthene                         44102 000092-83-1 91

 41  26.13  0.39 C:\Database\NIST02.L
                 Pyridine, 2-[2-(4-aminophenyl)ethe  53521 001694-46-8 52
                 nyl]-, trans-
                 9-Anthracenamine, 9,10-dihydro-     52778 097825-91-7 35
                 Benzaldehyde, phenylhydrazone       53507 000588-64-7 35

 42  26.61  0.26 C:\Database\NIST02.L
                 9 Anthracenamine, 9,10 dihydro      52778 097825 91 7 46
                 Xanthene, 9,9-dimethyl-             62999 019814-75-6 43
                 (1H)-Imidazo[4,5-b]pyridine, 2-phe  52709 001016-93-9 35
                 nyl-

 43  27.28  0.53 C:\Database\NIST02.L
                 Ethanone, 1,2-diphenyl-             53531 000451-40-1 35
                 .gamma.-Chlorobutyrophenone         43638 000939-52-6 27
                 Benzene, 1,2,4 trimethyl             9111 000095 63 6 27

 44  27.67  0.35 C:\Database\NIST02.L
                 Acetanilide, 2'-(1-methylacetonyl)  59383 014300-16-4 43
                 Benzenamine, N-ethyl-N-methyl-      15081 000613-97-8 38
                 2-Phenyl-dl-alanine                 32759 006945-32-0 38

 45  32.52  0.12 C:\Database\NIST02.L
                 2,6 Di p tolylpyridine              93938 014435 88 2 25
                 Silane, (fluoromethyl)triphenyl-   113262 151842-47-6 22
                 1,4-Benzenediamine, N,N-dimethyl-N  93707 126893-37-6 16
                 '-[(5-nitro-2-furanyl)methylene]-

POLYMERTD121608.M Sun Jul 25 11:36:54 2010
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File        :C:\MSDChem\1\DATA\071210\100002.D
Operator    : SM
Acquired    : 12 Jul 2010  11:52     using AcqMethod HUGHES 070710
Instrument :   SP_GCMS_1
Sample Name: 4513-002
Misc Info   :
Vial Number: 2
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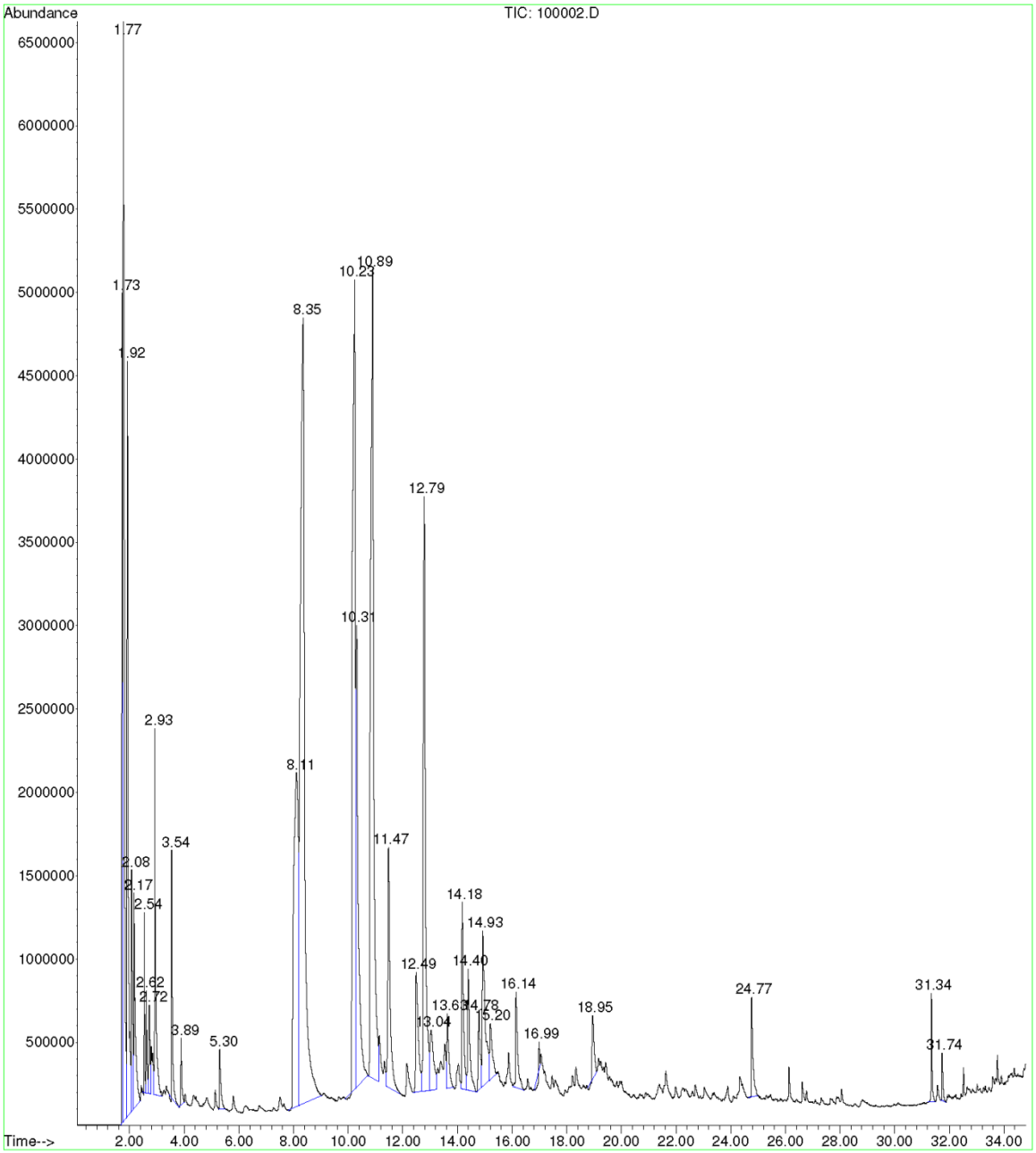

Library Search Report

Data Path : C:\MSDChem\1\DATA\071210\
Data File : 10C002.D
Acq On : 12 Jul 2010 11:52
Operator : SM
Sample : 4513-002
Misc :
ALS Vial : 2 Sample Multiplier: 1

Search Libraries: C:\Database\NIST02.L Minimum Quality: 0

Unknown Spectrum: Peak average
Integration Events: Chemstation Integrator - autoint1.e

| Pk# | RT | Area% | Library/ID | Ref# | CAS# | Qual |
|---|---|---|---|---|---|---|
| 1 | 1.73 | 3.25 | C:\Database\NIST02.L | | | |
| | | | Carbon dioxide | 77 | 000124-38-9 | 4 |
| | | | Acetaldehyde | 69 | 000075-07-0 | 4 |
| | | | Acetaldehyde | 69 | 000075-07-0 | 4 |
| 2 | 1.77 | 7.04 | C:\Database\NIST02.L | | | |
| | | | Aminomethanesulfonic acid | 6020 | 013881-91-9 | 50 |
| | | | L-Alanine, 3-sulfo- | 35516 | 000498-40-8 | 40 |
| | | | Sulfur dioxide | 346 | 007446 09 5 | 38 |
| 3 | 1.92 | 4.61 | C:\Database\NIST02.L | | | |
| | | | 2-Propenal | 180 | 000107-02-8 | 43 |
| | | | Acetone | 207 | 000067-64-1 | 9 |
| | | | Diazene, dimethyl- | 202 | 000503-28-6 | 9 |
| 4 | 2.08 | 1.27 | C:\Database\NIST02.L | | | |
| | | | 2 Propen 1 ol | 214 | 000107 18 6 | 42 |
| | | | Chloromethyl propanoate | 9430 | 005402-53-9 | 38 |
| | | | 2-Propen-1-ol | 213 | 000107-18-6 | 7 |
| 5 | 2.17 | 1.50 | C:\Database\NIST02.L | | | |
| | | | 1-Pentanol, 2-methyl- | 4345 | 000105-30-6 | 38 |
| | | | 2-Methyl-1-butene | 549 | 000563-46-2 | 32 |
| | | | 1-Pentanol, 2-methyl- | 4353 | 000105-30-6 | 28 |
| 6 | 2.54 | 0.56 | C:\Database\NIST02.L | | | |
| | | | Benzene | 1004 | 000071-43-2 | 91 |
| | | | Benzene | 1001 | 000071-43-2 | 91 |
| | | | Benzene | 1003 | 000071-43-2 | 91 |
| 7 | 2.61 | 0.43 | C:\Database\NIST02.L | | | |
| | | | 2-Butanone, 4-hydroxy-3-(hydroxyme | 13861 | 006868-07-9 | 47 |
| | | | thyl) 3 methyl | | | |
| | | | 3-Buten-2-one, 3-methyl- | 1406 | 000814-78-8 | 46 |
| | | | 3-Buten-2-one, 3-methyl- | 1410 | 000814-78-8 | 46 |
| 8 | 2.72 | 0.54 | C:\Database\NIST02.L | | | |
| | | | Acetic acid | 253 | 000064-19-7 | 47 |
| | | | Acetic acid | 256 | 000064-19-7 | 38 |
| | | | Acetic acid | 252 | 000064-19-7 | 38 |
| 9 | 2.93 | 1.89 | C:\Database\NIST02.L | | | |
| | | | Ethanamine, 2-methoxy- | 873 | 000109-85-3 | 9 |
| | | | 2-Propanone, 1-hydroxy- | 800 | 000116-09-6 | 9 |
| | | | 2,3-Pentanedione | 3591 | 000600-14-6 | 9 |
| 10 | 3.54 | 1.11 | C:\Database\NIST02.L | | | |
| | | | Toluene | 2405 | 000108-88-3 | 95 |
| | | | Toluene | 2403 | 000108 88 3 | 90 |
| | | | Toluene | 2402 | 000108-88-3 | 90 |
| 11 | 3.89 | 0.34 | C:\Database\NIST02.L | | | |
| | | | 1-Octene | 6449 | 000111-66-0 | 93 |

| | | | | | |
|---|---|---|---|---|---|
| | | | 2-Octene, (Z)- | 6468 | 007642-04-8 | 81 |
| | | | 2 Octene, (E) | 6467 | 013389 42 9 | 74 |
| 12 | 5.30 | 0.44 | C:\Database\NIST02.L | | | |
| | | | o-Xylene | 4959 | 000095-47-6 | 95 |
| | | | p-Xylene | 4953 | 000106-42-3 | 95 |
| | | | Benzene, 1,3-dimethyl- | 4977 | 000108-38-3 | 95 |
| 13 | 8.11 | 7.57 | C:\Database\NIST02.L | | | |
| | | | Aniline | 2442 | 000062 53 3 | 97 |
| | | | Aniline | 2444 | 000062-53-3 | 95 |
| | | | Aniline | 2443 | 000062-53-3 | 94 |
| 14 | 8.35 | 17.86 | C:\Database\NIST02.L | | | |
| | | | Phenol | 2538 | 000108-95-2 | 95 |
| | | | Phenol | 2536 | 000108-95-2 | 95 |
| | | | Phenol | 2540 | 000108-95-2 | 95 |
| 15 | 10.23 | 10.77 | C:\Database\NIST02.L | | | |
| | | | Phenol, 2-methyl- | 5243 | 000095-48-7 | 97 |
| | | | Phenol, 2-methyl- | 5244 | 000095-48-7 | 96 |
| | | | Phenol, 2-methyl- | 5233 | 000095-48-7 | 95 |
| 16 | 10.30 | 5.50 | C:\Database\NIST02.L | | | |
| | | | Aniline, N-methyl- | 5042 | 000100-61-8 | 93 |
| | | | Aniline, N methyl | 5035 | 000100 61 8 | 87 |
| | | | o-Toluidine | 5030 | 000095-53-4 | 87 |
| 17 | 10.89 | 12.61 | C:\Database\NIST02.L | | | |
| | | | Phenol, 4-methyl- | 5235 | 000106-44-5 | 95 |
| | | | Phenol, 4-methyl- | 5230 | 000106-44-5 | 94 |
| | | | Phenol, 4-methyl- | 5240 | 000106-44-5 | 94 |
| 18 | 11.48 | 2.83 | C:\Database\NIST02.L | | | |
| | | | Phenol, 2,6-dimethyl- | 9613 | 000576-26-1 | 97 |
| | | | Phenol, 2,6-dimethyl- | 9624 | 000576-26-1 | 96 |
| | | | Phenol, 2,6-dimethyl- | 9619 | 000576-26-1 | 95 |
| 19 | 12.49 | 1.61 | C:\Database\NIST02.L | | | |
| | | | Phenol, 3-ethyl- | 9600 | 000620-17-7 | 91 |
| | | | Phenol, 3-ethyl- | 9602 | 000090-00-6 | 87 |
| | | | Phenol, 3,4 dimethyl | 9622 | 000095 65 8 | 87 |
| 20 | 12.79 | 6.56 | C:\Database\NIST02.L | | | |
| | | | Phenol, 3,5-dimethyl- | 9620 | 000108-68-9 | 97 |
| | | | Phenol, 2,4-dimethyl- | 9616 | 000105-67-9 | 97 |
| | | | Phenol, 2,4-dimethyl- | 9623 | 000105-67-9 | 96 |
| 21 | 13.04 | 1.15 | C:\Database\NIST02.L | | | |
| | | | Phenol, 2,5 dimethyl | 9619 | 000095 87 4 | 50 |
| | | | Phenol, 2,5-dimethyl- | 9626 | 000095-87-4 | 46 |
| | | | Benzenamine, N,4-dimethyl- | 9285 | 000623-08-5 | 46 |
| 22 | 13.63 | 0.77 | C:\Database\NIST02.L | | | |
| | | | Phenol, 3-(1-methylethyl)- | 15871 | 000618-45-1 | 50 |
| | | | Phenol, 3,4,5-trimethyl- | 15829 | 000527-54-8 | 46 |
| | | | Phenol, 4-(1-methylethyl)- | 15866 | 000099-89-8 | 46 |
| 23 | 14.18 | 1.65 | C:\Database\NIST02.L | | | |
| | | | Phenol, 2,4,6-trimethyl- | 15836 | 000527-60-6 | 95 |
| | | | Phenol, 2,3,6-trimethyl- | 15834 | 002416-94-6 | 90 |
| | | | Phenol, 2,3,5-trimethyl- | 15832 | 000697-82-5 | 90 |
| 24 | 14.40 | 1.27 | C:\Database\NIST02.L | | | |
| | | | 2-Propenal, 2-methyl-3-phenyl- | 20656 | 000101-39-3 | 53 |
| | | | 1H Benzimidazole, 2 ethyl | 21277 | 001848 84 6 | 53 |
| | | | Benzofuran, 4,7-dimethyl- | 20650 | 028715-26-6 | 53 |
| 25 | 14.78 | 0.75 | C:\Database\NIST02.L | | | |
| | | | Pyridine, 3,5-dimethyl- | 5057 | 000591-22-0 | 58 |

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                 Acetamide, N-(3-methylphenyl)-      22577 000537-92-8 58
                 Pyridine, 3,5 dimethyl               5071 000591 22 0 58

26  14.92  2.23 C:\Database\NIST02.L
                 Phenol, 2-ethyl-4-methyl-           15856 003855-26-3 83
                 Phenol, 2-(1-methylethyl)-          15867 000088-69-7 53
                 Phenol, 2-(1-methylethyl)-          15873 000088-69-7 49

27  15.20  0.75 C:\Database\NIST02.L
                 Phenol, 2 ethyl 4 methyl            15856 003855 26 3 60
                 Phenol, 2-ethyl-6-methyl-           15852 001687-64-5 60
                 3-Methylene-1,5,5-trimethylcyclohe  15265 016609-28-2 52
                 xene

28  16.15  1.01 C:\Database\NIST02.L
                 1H-Pyrrole, 1-phenyl-               19297 000635-90-5 64
                 Isoquinoline, 3-methyl-             19302 001125-80-0 60
                 1H Pyrrole, 1 phenyl                19299 000635 90 5 60

29  16.99  0.00 C:\Database\NIST02.L
                 2,5,6-Trimethylbenzimidazole        29215 003363-56-2 91
                 Naphthalene, 1,2,3,4-tetrahydro-1,  29456 021564-91-0 86
                 5-dimethyl-
                 Benzene, 4-(2-butenyl)-1,2-dimethy  29441 054340-86-2 64
                 l-, (E)-

30  18.95  0.59 C:\Database\NIST02.L
                 1H-Pyrrole, 1-(4-methylphenyl)-     27871 000827-60-1 76
                 1-Amino-2-methylnaphthalene         27863 002246-44-8 64
                 1-Naphthalenemethanamine            27848 000118-31-0 59

31  24.77  0.77 C:\Database\NIST02.L
                 9H-Xanthene                         44103 000092-83-1 95
                 9H Xanthene                         44104 000092 83 1 94
                 9H-Xanthene                         44102 000092-83-1 91

32  31.34  0.51 C:\Database\NIST02.L
                 Benzonitrile, 2,4,6-trimethyl-      20387 002571-52-0 35
                 Aniline                              2444 000062-53-3 18
                 Aniline                              2442 000062-53-3 18

33  31.74  0.25 C:\Database\NIST02.L
                 Quinoline, 5,6,7,8-tetrahydro-3-me  21360 028712-62-1 43
                 thyl-
                 2-(1-Methyl-1,2,3,4-tetrahydro-iso  59482 1000275-38-4 43
                 quinolin-1-yl)-propan-2-ol
                 Benzocycloheptene                   20694 001075-16-7 38

POLYMERTD121608.M Sun Jul 25 11:38:39 2010
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Operator    : SM
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Instrument :   SP_GCMS_1
Sample Name: 4513-002
Misc Info   :
Vial Number: 1
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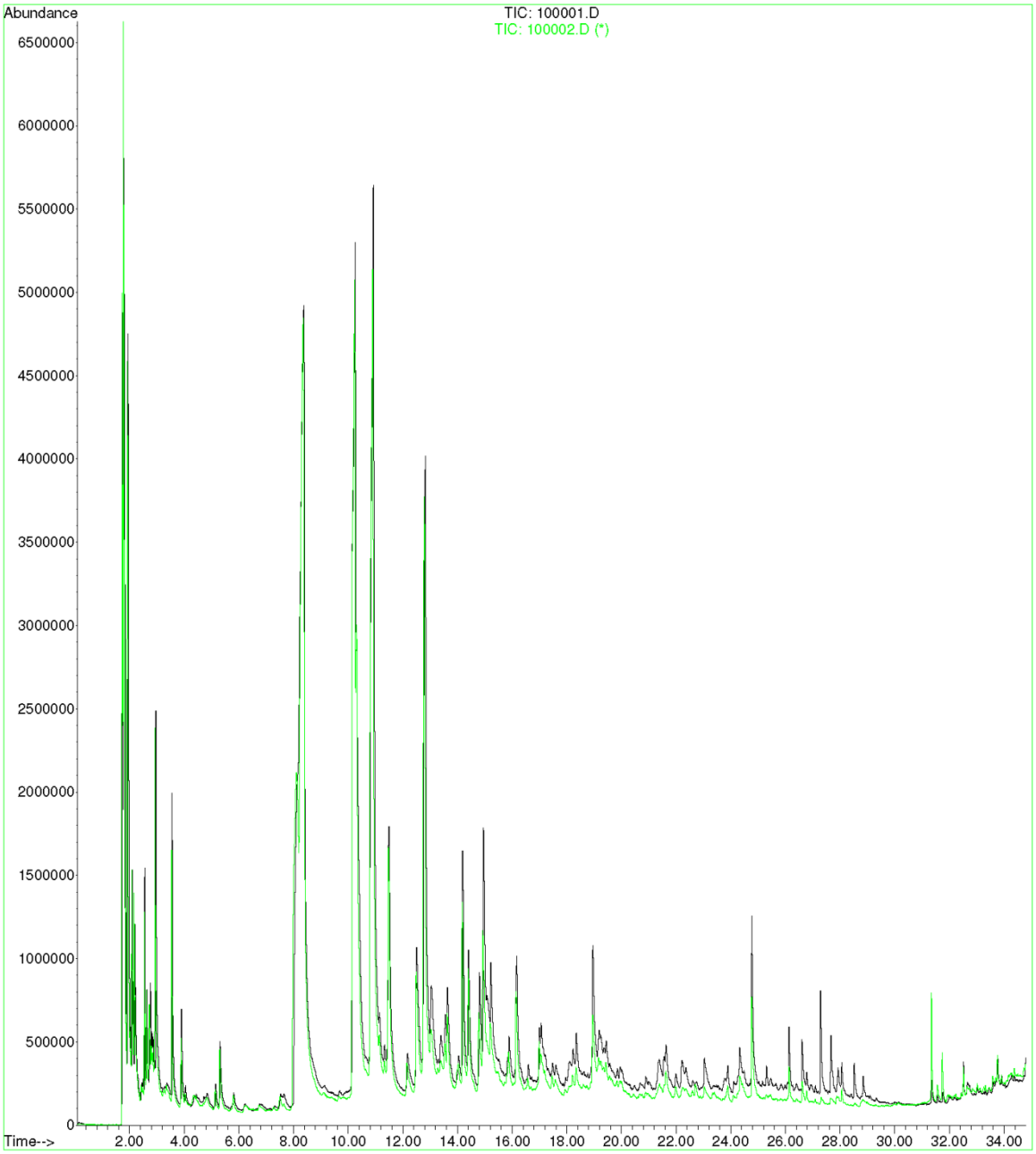

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Operator    : SM
Acquired    : 12 Jul 2010  12:46     using AcqMethod HUGHES 070710
Instrument :   SP_GCMS_1
Sample Name: blank
Misc Info   :
Vial Number: 3
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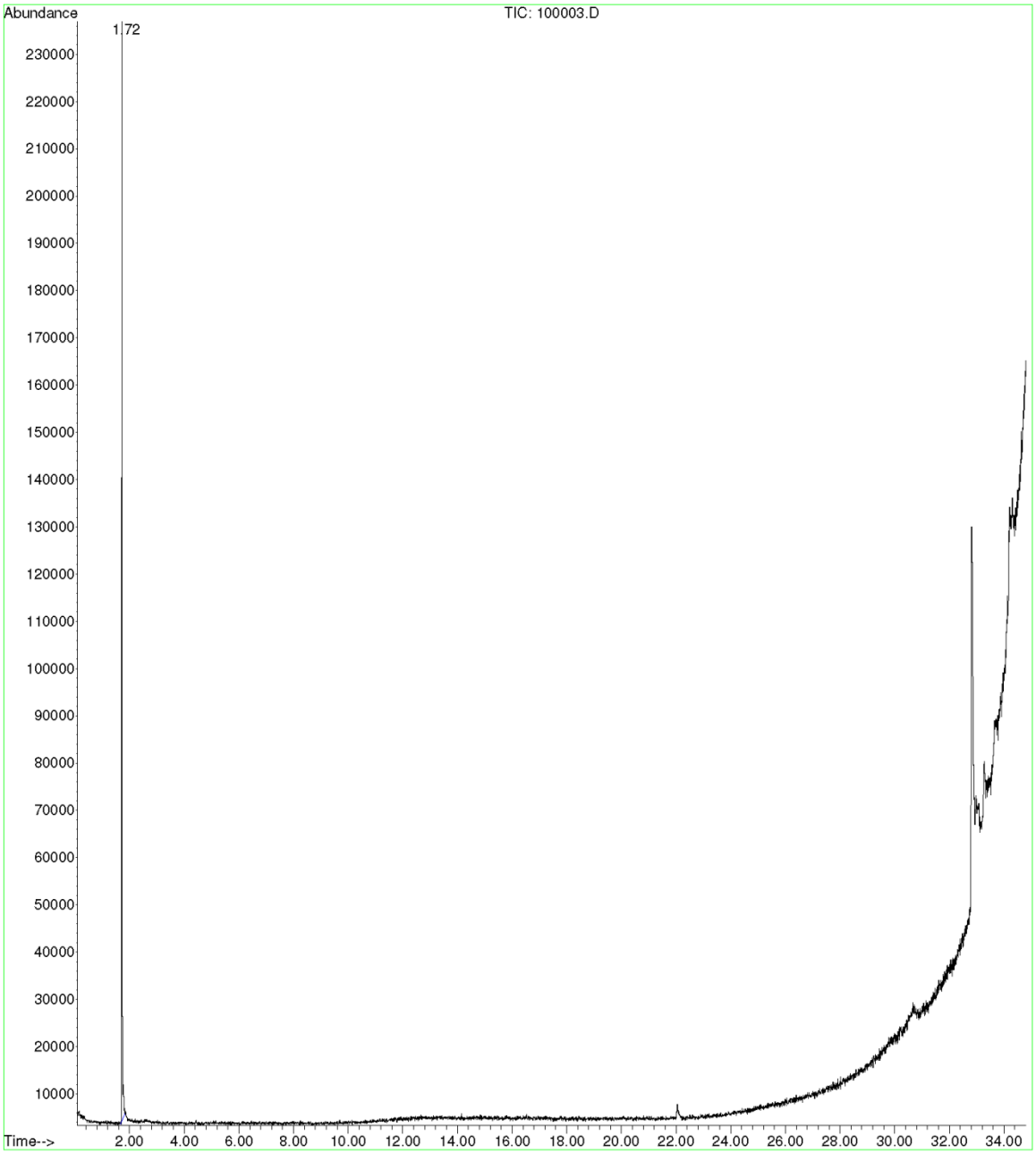

Library Search Report

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  Data File : 100003.D
  Acq On    : 12 Jul 2010  12:46
  Operator  : SM
  Sample    : blank
  Misc      :
  ALS Vial  : 3   Sample Multiplier: 1

  Search Libraries:   C:\Database\NIST02.L              Minimum Quality:   0

  Unknown Spectrum:   Peak average
  Integration Events: Chemstation Integrator - autoint1.e

Pk#    RT  Area%          Library/ID                 Ref#     CAS#   Qual
_____________________________________________________________________________
  1   1.72 100.00 C:\Database\NIST02.L
                 Carbon dioxide                        77 000124-38-9  4
                 Carbon dioxide                        78 000124-38-9  3
                 Nitrous Oxide                         79 010024-97-2  3


POLYMERTD121608.M Sun Jul 25 11:40:16 2010
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