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Studies of Ion Exchange Resin Integrity under Flowsheet Extremes: Part II

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SUMMARY

This task addressed four items related to SuperLig[®] 644 ion exchange resin stability under nominal to extreme conditions:¹

A process upset analysis of resin in contact with 1 molar sodium permanganate at 25 and 40 °C identified runaway reaction behavior that was characterized by acute self-heating and sample ejection with only condensable gas (e.g. steam) evolution. An analogous test of the resin-permanganate reaction under 2000 kPa prevented sample ejection, displayed a temporary pressure rise, but showed no net evolution of noncondensable gases. Follow-on work done before this report was finalized suppressed sample ejection by testing a range of initial elevated pressures.²

A simulation of the accelerated aging of resin during normal dilute acid (0.5 M) regeneration operations identified no measurable reactivity.

The behavior of resin in 5 molar nitric acid at ambient temperatures was characterized by a slow, steady evolution of noncondensable gases (NO_x and CO₂), and dissolved 80% of the resin after 21 days. The neutralization of this acidic “SuperLig[®] 644 resin solution” with sodium hydroxide was characterized as a rapid exothermic reaction without foaming, bubbling, or precipitation.

Resin exposure to water at 60 °C showed no measurable gas evolution, and dissolved 55% of the resin mass after 7 days. The cause for the loss in resin mass could not be definitively determined. It might have been due largely to the presence of a substantial amount of potassium bicarbonate contained within the as-received resin supplied by the vendor. Substantial dissolution of the resin in water was not anticipated and further work is needed to determine what is occurring.

Work to follow that reported here shall address the potassium bicarbonate content of the as-received resin by pretreating the resin. The presence of approximately 50 wt% potassium bicarbonate in the resin was a recent discovery.³ The bicarbonate affected batch distribution and mass loss/dissolution work up to the point that it was discovered.

Differential scanning calorimetry (DSC) analysis of solid and liquid residues showed small heat flow, evidence that the potential reactivity of these resin-nitric acid reaction products is small to negligible. Volatile organic analysis (VOA) did not identify any reactive degradation products. Semi-volatile organic compound analysis (SVOC) of liquid residues did not identify typical low molecular weight resin degradation products. Liquid reaction products have been analyzed by liquid chromatography-mass spectrometry (LC-MS). In summary, this work characterized the reactivity SuperLig[®] 644 ion exchange resin under various conditions as part of the validation of waste treatment technologies to be employed by the Hanford River Protection Project-Waste Treatment Plant (RPP-WTP). The quantified impact of the results from these tests on facility design will be addressed in a separate document by the design authority.

INTRODUCTION

Incidents involving undesirable self-accelerated exothermic reactions between organic resins and nitric acid solutions have been well documented.^{3,4,5,6,7,8} The work reported here is intended to address safety concerns during normal and off-normal operations during the use of SuperLig[®] 644 ion exchange resin to separate cesium from low activity waste streams. This report covers the thermal behavior characterization of resin under various conditions, and an evaluation of the potential energy release from the solid, liquid and gaseous reaction products. This work is a sequel to the resin-nitric acid stability testing at high temperatures.³ The objective of this work is to characterize the reactivity of SuperLig[®] 644 and identify a margin of safety for plant process operations. These results are intended to be used by the design authority, along with additional testing that has been performed at SRTC and elsewhere, to determine the impact on facility design safety. The design authority will develop the bounding facility conditions (e.g., safe concentration of permanganate solution, pressure relief capacity, etc.).

Background on Ion Exchange Processing and Resin Dissolution

The RPP-WTP process for separating cesium and technetium from low-activity waste solutions uses SuperLig[®] 644 and SuperLig[®] 639 resins, respectively. RPP-WTP plans to elute the SuperLig[®] 644 resin with 0.5M nitric acid and concentrate the eluate at 50 – 60 °C under sufficient vacuum to cause boiling and concentrate the solution to 80% total salts saturation and ~5M nitric acid. SuperLig[®] 644 resin particles could potentially exit from the ion exchange column during elution due to failure of the resin retention mechanism (Johnson wedge-wire screen) or attrition of resin particles. Therefore, resin could be boiled with the eluate solution, leading to potential adverse reactions (e.g. energy release).

The initial study of resin-nitric acid mixtures evaluated the energetics and decomposition products from contacting SuperLig[®] 644 resin particles at 90 °C in 5M nitric acid to simulate off-normal operating temperatures during plant evaporation and concentration processes. These results show that SuperLig[®] 644 will likely completely dissolve in the 5M nitric acid solution with evolution of nitrous oxide and carbon dioxide, but not hydrogen gas. Differential scanning calorimetry of the evaporated liquid and the solids residues from the incomplete digestion of resin and 5M nitric acid showed no exotherms. As a control blank, an additional test of resin in water showed that about 50% of the resin dissolved after contacting for 24 hours at temperatures of 90 to 100 °C.

These initial results suggest that it may be possible to safely dissolve SuperLig[®] 644 in 5M nitric acid (or water at elevated temperatures). Resin dissolution at ambient temperature is desirable to avoid additional process equipment and controls. This may provide a means to dispose of the spent resin by feeding it to the LAW melter as opposed to radioactive solid waste. To evaluate the possibility of resin dissolution, the reaction of SuperLig[®] 644 in nitric acid at ambient temperature and in water at 60 °C needs to be studied. For each reaction, the energetics, pressure rate increase, temperature, composition of gases evolved, and extent of dissolution were determined and documented in order to evaluate potential safety hazards.

In addition, the RPP-WTP process for eluting cesium from SuperLig[®] 644 involves contact with 0.5M nitric acid solution for prolonged periods at nominally 25 °C. The stability of the resin in contact with the dilute elution acid at off-normal temperatures needs to be evaluated. The energetics, pressure rate increase, temperature, and composition of gases evolved were determined and documented to evaluate potential safety hazards and identify operational limits.

The behavior of SuperLig[®] 644 resin under conditions of 0.5 M and 5 M nitric acid under conditions of room temperature for periods of up to one month were already addressed by IBC Technologies.⁸ Resin exposed to 0.5 M nitric acid was found to not dissolve much and to still perform after one month of exposure. IBC also confirmed that 5 M and 12.2 M nitric acid dissolved most to all of the resin in just a few minutes.

Background on Off-normal Contact of Sodium Permanganate with SuperLig[®] 644

The RPP-WTP process for separating Sr⁹⁰ and transuranic elements for Envelope C solutions uses sodium hydroxide, strontium nitrate, and sodium permanganate solutions. A hazard review has postulated a scenario in which sodium permanganate solution could be inadvertently transferred into the feed vessel for the cesium ion exchange column, and then into columns containing SuperLig[®] 644 resin. To evaluate this potential safety hazard, the pressure rate increase, temperature rate increase, and composition of gases evolved were determined and documented.

This work addresses four items related to SuperLig[®] 644 resin stability: (1) process upset evaluation of resin in contact with 1 molar sodium permanganate at 25 and 40 °C, (2) accelerated aging with nitric acid solution used during normal regeneration operations, (3) prolonged contacting of SuperLig[®] 644 resin with 5 molar nitric acid at ambient temperature, and (4) prolonged contacting of SuperLig[®] 644 resin with deionized water at 60 ± 5 °C.

This task fulfills all the requests as defined in Task Technical Plan BNF-003-98-0225. Quality Assurance requirements were applied to this work as described in BNF-003-98-0225, Studies of Ion Exchange Resin Integrity under Flowsheet Extremes: Part II, W. Crooks, C. Nash, D. McCabe, H. Saito.

EXPERIMENTAL

Apparatus

The Reactive System Screening Tool (RSST) was used to characterize the thermal effects of the reactions between SuperLig[®] 644 organic resin and process chemicals. The RSST calorimeter is traditionally used for the efficient screening of runaway reactions as a function of temperature, and also serves as a reactor vessel. The RSST is a nearly adiabatic calorimeter that heats an open, well-insulated 14-mL test cell within a sealed 350-mL Parr bomb (Figures 1 and 2). The test cell mixture is stirred and heated at a nearly

constant rate and the Parr bomb and test cell are monitored for temperature and pressure, respectively. All tests utilized SuperLig[®] 644 (batch # 981020MB48-563), as received.

One deficiency of the RSST control/data recording software to be noted is that it could only run for just less than 24 hours at a time. Runs exceeding 24 hours had to be restarted manually. Gaps in the 21-day run WJC-IV-021 (Test 8) were days that the equipment was not restarted. Other runs were shorter than 24 hours or were restarted to allow continuous data recording. A software upgrade to allow longer term data recording became available and was implemented after this test program was complete.

Procedure

Accelerated aging tests, the permanganate reaction tests, and the resin digestion/dissolution tests were performed using the RSST. The general procedure used for conducting RSST tests is described below:

1. SuperLig[®] 644 resin and a process solvent (nitric acid or water) was added to the test cell. However, sodium permanganate was added as described below.
2. The test cell was loaded into the Parr bomb, sealed, and the mixture was stirred and heated at a programmed rate. Alternately, because of the vigorous reactivity of 1M sodium permanganate reactions with SuperLig[®] 644 resin at ambient temperature, the sodium permanganate solution is added via an extension tube to the dry resin loaded in the test cell within a sealed Parr bomb. The initial composition of the reaction mixture for all RSST tests in this work is described in **Table 1**. Tests 1-6 were designed to evaluate process upset due to resin in contact with 1 molar sodium permanganate. Test 7 was designed to simulate the effect of accelerated aging of resin in contact with the dilute nitric acid solution used during normal column regeneration operations. Tests 8 and 9 were designed to evaluate the dissolution of resin in either nitric acid or water.
3. The test cell mixture and the Parr bomb were monitored for temperature and pressure, respectively.

Table 1. Summary of Reactants for Resin Stability Tests in RSST

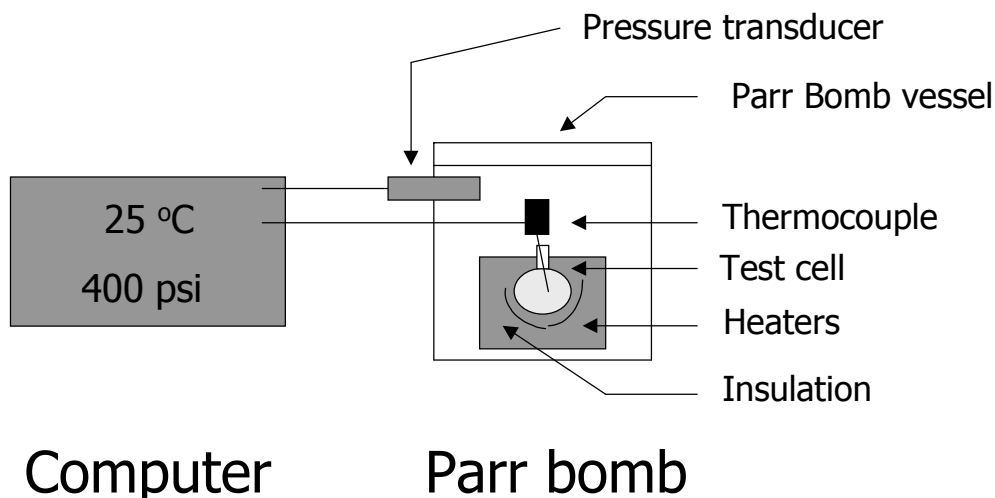
Test No.	Record Reference	Initial Temp. °C	Resin g	1M NaMnO ₄ g	5M HNO ₃ g	Water g	0.5M HNO ₃	Initial Press. psig
1	WJC-IV-025	ambient	2.51	7.93				0
2	WJC-IV-027	ambient	2.51	7.93				0
3	WJC-IV-033	ambient	2.50	7.92				0
4	WJC-IV-035	40	2.51	7.93				0
5	WJC-IV-109	ambient	2.52			9.36		290
6	WJC-IV-111	ambient	2.52	8.68				291
7	WJC-IV-009	ambient	2.50				7.98	0
8	WJC-IV-021	ambient	0.40		11.58			0
9	WJC-IV-045	ambient	0.40			10.00		0

4. The pressure-temperature data were processed to generate plots of temperature vs. time, pressure vs. time, self-heating rate vs. temperature, rate of pressure change vs. temperature, and pressure vs. temperature.
5. The Analytical Development Section (ADS) analyzed sample chemistry per quality assurance requirements as well as tracking samples within the LIMS system. ADS analyzed liquid samples by SVOC and gaseous samples by VOA Halocarbon (5mL purge/trap) and VOA Halocarbon (direct gas loop). These organic analysis methods are consistent with EPA SW-846 methods, although the laboratory is not certified by the South Carolina Department of Health and Environmental Control for NPDES discharge compliance monitoring.
6. The Materials Technology Section (MTS) provided differential scanning calorimetry (DSC) analyses.
7. The Savannah River Ecology Lab performed Ion Cyclotron Mass Spectrometry (ICMS) of selected samples of resin mixtures in an effort to identify volatile and/or potentially energetic compounds. Samples were neutralized to about pH 2 to 3. The neutralized samples were then loaded onto a C-18 SPE column, eluted with methanol, diluted 1:1 with water, and 50 μ L of acetic acid was added. Samples were not separated into components by chromatography. Chemical ionization (CI) was used to introduce the sample into the ICMS. CI is a “soft” ionization method that generally will not cause fragmentation of molecules, and therefore, tends to produce parent molecular ions plus one proton, the $(M+1)^+$ peak. The spectra in this report were measured at low resolution (~ 0.02 m/z). Since samples were not separated into pure components by liquid chromatography, each spectrum contains the $(M+1)^+$ peak of each component, in addition to some fragmentation products. The SREL laboratory is not certified by the South Carolina Department of Health and Environmental Control for NPDES discharge compliance monitoring.
8. Data were recorded in a controlled laboratory notebook.⁹

FIGURE 1. PHOTOGRAPH OF THE PARR BOMB OF THE RSST



FIGURE 2. SKETCH OF THE RSST



RESULTS AND DISCUSSION

1. “Process upset” test of SuperLig[®] 644 resin in contact with 1M sodium permanganate at 25 °C, 40 °C, and at 25 °C and 2000 kPa

Sodium permanganate is a powerful oxidizer that is expected to damage organic ion exchange resins. The purpose of this work was to characterize the reaction that will occur as a result of the accidental addition of sodium permanganate solution to the SuperLig[®] 644 ion exchange column. To simulate resin packed in a column, the liquid:solid ratio was 3:1 (mass:mass). Resin was mixed with a solution of 1 molar sodium permanganate in the RSST at a specified temperature and pressure. Significant self-heating was observed although no heat was applied. The reactivity of the system was characterized by monitoring the temperature and pressure changes. Some of the samples were analyzed for gaseous products but none were identifiable.

Scoping Test A: Reactivity assessment

This test was performed in accord with the National Fire Protection Agency (NFPA) guidelines for working with potentially energetic compounds, as approved by the DOE Explosives Safety Committee.^{10,11} As suggested by the NPFA guidelines, the reactivity scoping test was performed in a chemical hood with less than 50 mg of material, and personal protective equipment included leather gloves and a face shield. In addition, a Plexiglas[™] barrier was in place between the test reaction and personnel. SuperLig[®] 644 resin (39.7 mg) was placed on a watchglass. A drop of 1 molar sodium permanganate was added, and no evidence of reaction was observed. No bubbling or foaming was observed. After 5 minutes, 5 additional drops were added, and a few small bubbles slowly formed and burst. These small bubbles could be the liberation of trapped air (lower solubility)

due to self-heating from the exothermic reaction. The purple sodium permanganate turned brown upon addition. Although little to no gas is generated, the resin appeared to degrade and partially dissolve, forming a brown MnO_2 -resin slurry. No RSST test was performed.

Scoping Test B: Scale-up reactivity at 25 °C.

Based on the intensity of the reactivity scoping test, a mass scale-up test was performed in a test cell. Since this test simulates resin packed in a column, the target liquid:solid ratio was about 3:1. The RSST test cell was loaded with SuperLig[®] 644 resin (2.501 grams). To the test cell was added 1 molar sodium permanganate (4.5 grams). In less than two seconds, the reaction mixture bubbled (probably generating steam), and was gently ejected from the test cell. The reaction mixture remaining in the test cell was 80 °C and cooling. The test cell was quickly placed in the Parr bomb and quickly sealed but no pressure generation was observed. Gas analysis by VOA showed only a trace of carbon dioxide. The resulting mixture was a brown slurry. No purple color remained, qualitative evidence that the permanganate ions were completely consumed in the reaction. This scoping test showed that a rapid addition of NaMnO_4 to SuperLig[®] 644 resin at atmospheric pressure leads to ejection of the reaction mixture from the test cell, although the reaction was complete before the system could be sealed, prohibiting measurement of gas evolution.

Scale-up reactivity tests in a *sealed* RSST at ambient and 40 °C.

Tests 1-3. Based on the minimal intensity of the preceding scoping test, gram-scale tests were performed in a *sealed* RSST. Using the target liquid:solid ratio of about 3:1, the RSST test cell was charged with a SuperLig[®] 644 resin and loaded into the RSST. Before the RSST test was started, the 1 molar sodium permanganate solution was added to the test cell by injecting the solution through an extension tube. The RSST reaction profiles shown in Figures 3-6 characterize the runaway reactions. Note that all pressures reported are gauge rather than absolute. All three tests showed rapid temperature and pressure excursions upon addition of sodium permanganate. Maximum self-heating rates were on the order of 500 to 1000 °C/min. The flattened portion of the self-heating profile is evidence of vapor saturation (i.e. boiling). Two minutes after addition, the temperature and pressure decrease, evidence that the sodium permanganate has been completely consumed.

Gas analyses from Test 1 (1-gas) and Test 2 (2-gas) are shown in the Results section.

Test 4. For the test starting at 40 °C, the dry resin and permanganate solution were each pre-equilibrated in a water bath at temperature prior to being mixed. These results, which are included in Figures 3-6, started at a higher initial temperature but had similar reaction profiles to the tests starting at ambient temperature with one exception. The maximum pressure was much lower for the 40 °C test. One explanation is that initial reaction rate was higher at 40 °C (i.e. rate doubles with 10 °C increase). A more rapid reaction could result in ejection of sample before the reaction is complete. This result is consistent with the unusually charred condition of the test cell at the completion of Test 4.

If a noncondensable gas is evolved during the permanganate tests, then a net pressure rise should be observed. The observed pressure rise is the sum of the pressure due to the charge of the liquid into the closed system and any pressure generated from the reaction. An initial test (performed in triplicate) showed that the addition of 10 mL of water to the sealed RSST results in a pressure rise of 3 kPa \pm 0.1 (theoretical pressure rise is \sim 3 kPa gauge). Therefore, to correct for pressure associated with addition of the 10-mL liquid reactant, the net pressure change, ΔP_{net} , is given by the following equation:

$$\Delta P_{\text{net}} = \Delta P_{\text{exp}} - 3 \text{ kPa}$$

Table 2 shows the net pressure changes associated with the addition of sodium permanganate to resin, after subtracting the pressure change associated with the addition of 10 mL of liquid. The negligible to small net pressure changes in tests 1-4 suggest that noncondensable gases (e.g. carbon dioxide) are not evolved during the reaction.

Table 2. Net Pressure Changes Associated with Sodium Permanganate Addition to SuperLig[®] 644

Test No.	Record Name	Initial Temp. °C	ΔP_{net} Pa
1	WJC-IV-025	ambient	0
2	WJC-IV-027	ambient	1
3	WJC-IV-033	ambient	5
4	WJC-IV-035	40	0

Test 5. To suppress sample ejection during sodium permanganate addition to resin, high-pressure tests were performed. Dry resin was placed in the Parr bomb and pressurized to about 2000 kPa with nitrogen. Note that all pressures reported are gauge rather than absolute. A high-pressure syringe was loaded with the water, and attached to the extension tube of the RSST. After the pressure reading stabilized, the water was charged into the test cell using a 690 kPa overpressure (about 2690 kPa) on the high-pressure syringe. No reaction was expected but pressure changes associated with the addition of liquid to the sealed Parr bomb, and temperature changes associating with the mixing process were evaluated in this control run. The RSST reaction profiles are shown in Figures 7-10. Pressure and temperature changes before the system was stabilized to begin the test are included for completeness. The addition of water causes an immediate 0.5 °C temperature rise (Figure 7) and 50 kPa pressure rise (Figure 8). After the system came to thermal equilibrium, the net pressure rise was 35 kPa. Therefore, an increase in system pressure of about 35 kPa is associated with the addition of the liquid to the sealed system.

Test 6. The addition of sodium permanganate solution to resin was characterized by a rapid temperature and pressure excursion upon addition of sodium permanganate (Figures 11-14). However, the maximum self-heating rate of 45 °C/min occurred at 30 °C. In contrast to Tests 1-3 in which vapor saturation was observed, the high pressure suppressed

the solvent vaporization, and no sample was ejected from the test cell. The net pressure rise was 30 kPa. This value is smaller than the net pressure rise from the water test (35 kPa). The pressure rate plot in Figure 14 shows an initial pressure rate spike associated with addition of sodium permanganate, and a small pressure rate increase from 40 to 50 °C which is likely associated with the vaporization of solvent. This evidence suggests that noncondensable gases are not generated from the reaction of sodium permanganate and SuperLig[®] 644 resin.

Ion Cyclotron Mass Spectrometry (ICMS) was performed on the brown slurry residue from Test 6. The mass spectrum contained a large distribution of peaks with a wide range of mass/charge (m/z) ratio. This large distribution of compounds is consistent with the general degradation of the organic resin, however, no peak predominated and no peaks could be identified. The spectrum is not included in this report.

2. “Accelerated aging” test of SuperLig[®] 644 resin with nitric acid solution used during normal operation

Resin in 0.5 molar nitric acid was heated in the RSST. The reactivity of the system was characterized by monitoring temperature and pressure changes. Solid, liquid and gaseous degradation products were analyzed.

Scoping test. In preparation for the RSST, the addition of 1 mL of 0.5 molar nitric acid to dry SuperLig[®] 644 resin resulted in a immediate foaming and fizzing, causing a resin-acid mixture to be ejected from the test cell. The ejected material was a pale-green/brown color, with a white foamy head. The run was aborted.

Test 7. An alternate preparation was devised to prevent ejection of sample during the test cell loading. To the 2.5 grams of dry resin at room temperature was added 7.19 grams of water followed incremental addition of 50 to 100 µL aliquots of 5 M nitric acid (total 790 µL) over a 30 minute period. Total acid additions provided the desired 0.5 M nitric acid. The RSST was then sealed, and the mixture was run with an imposed self-heating rate of 0.25 °C/min to 90 °C, and held for 24 hours. The RSST reaction profiles are shown in Figures 15-18. No exotherm was observed. The only evidence of reaction was the net pressure increase was 5 kPa gauge(ambient temperature). The gas was not analyzed. Dried solids and evaporated and dried liquid samples were analyzed by DSC (Table 4, Figures 34 and 35)). The analysis of the liquid and solid samples identified no reactive compounds.

The resin residue (1.2431 g) and residue recovered in the rinses (0.0181 g) was combined to give 1.2612 g of solid mass recovered from the original 2.50 g (50%). The mass lost during the treatment was 15%. Assuming, all mass lost was due to evaporation, 1.21 grams of liquid was evaporated during the test. The remaining 6.88 grams of liquid has a specific gravity of 1.0717 g/mL, and therefore, a volume of 6.42 mL. The organics recovered from the evaporation of 1 mL of liquid was 172.9 mg. The amount of resin dissolved in the liquid is calculated by 6.42 mL x 172.9 mg/mL x 1 g/1000 mg = 1.11 g (44%). The mass balance due to solid and liquid was 94%.

3. Prolonged Contacting of SuperLig[®] 644 resin with 5M nitric acid solution at ambient temperature

For this RSST test (0.4 g resin), a larger-scale beaker test (10 g acid) was conducted simultaneously. The larger-scale beaker test was used to visually monitor the dissolution of resin. The RSST was run continuously and the Parr bomb was kept sealed until termination of the test at 21 days. The target liquid:solid ratio of about 25:1 will be used for both RSST-scale and the larger-scale beaker tests. Resin was contacted with 5M nitric acid for 21 days in the RSST. Solid, liquid, and gaseous degradation products were analyzed.

Test 8. RSST test. SuperLig[®] 644 resin (0.4 grams) and 5 molar nitric acid (10.0 grams) were loaded into the RSST, and containment was closed without backpressure. The system was slowly heated from ambient temperature to 25 ± 5 °C at a rate of 15 °C/hr (0.25 °C/min), and held at 25.0 ± 1.1 °C until the test was terminated. The objective of a similar and parallel large-scale test (not performed in the RSST) was solely to observe the reaction to determine when dissolution is complete, so that the activity in the concurrent RSST test could be inferred. The larger-scale beaker test contained SuperLig[®] 644 resin (4 grams) and 5 molar nitric acid (100 grams), and was rapidly stirred with a magnetic stir bar. The beaker was covered with a watchglass and visually monitored. The beaker-scale test did not completely dissolve the resin in 21 days. The RSST test was terminated, and the off-gas was collected in a Tedlar[™] bag.

The RSST reaction profiles shown in Figures 19-22 characterize the reactivity of the system. A slow steady pressure rise is evidence of gas evolution associated with slow degradation. Gaps in the data shown in Figure 19 and 20 are days that the RSST data collection system was not restarted. The data acquisition software could only run up to 24 hours at a time. Manual restart was needed after that for data collection to continue. It must also be noted that the temperature rose above 32 °C briefly on days 7 and 8. These were caused by heater control problems. The heater temperature control was adjusted at the time in each case and temperature decreased back to desired levels.

The mass loss in the test was 4%, and is attributed to solvent evaporation and condensation on the sides of the vessel. The residue recovered from the evaporation of 1 mL of liquid was 32.6 mg. The mass of resin that dissolved was 0.313 g (78%). The recovered solid had a mass of 0.0843 g (21%) after drying in vacuum at 40 to 50 °C for 8 hours. The mass balance due to liquid and solid recovery was 99%. The gas was analyzed by VOA (Table 3, 8-gas) and the liquid was analyzed by SVOC (Table 3, 8-liquid). The SVOC analysis indicated several peaks that had fragmentation patterns consistent with ethers or other oxygenated organics. Since the structure of the resin material is proprietary and not known to SRTC, a positive identification of soluble degradation products was not feasible, and the concentration shown (4.9 mg/L) is the sum total of the estimated concentrations. The presence of toluene was also identified in the “blank acid” run of the RSST,³ and likely originates from degradation of polymeric internals, such as insulation. Dried solids and evaporated and dried liquid samples were analyzed by DSC (Table 4,

Figures 36 and 37). The analysis of the gas, liquid and solid samples identified no reactive compounds. In addition, VOA analyses were performed on blank runs B1_N2 (nitrogen in Tedlar[®] bag) and B2_air (air in Tedlar[®] bag), and the results are shown in Table 3.

Ion Cyclotron Mass Spectrometry was performed on yellow liquid from Test 8 (partial dissolution) is shown in Figure 31. The mass spectrum contained three sets of peaks: 938-939, 640-681, and 165-281 m/z. The three sets of peaks are consistent with the sequential fragmentation of a resin copolymer into smaller units. However, no specific compounds could be identified.

Preparation of a SuperLig[®] 644 solution for neutralization test. SuperLig[®] 644 resin (4 grams) was dissolved in 5 molar nitric acid (100 grams) by heating at about 90 °C for 8 hours.

ICMS performed on yellow liquid from Test 8 (complete dissolution) is shown in Figure 32. The mass spectrum contained two broad distributions of peaks (~200 m/z and ~550 m/z). The multitude of peaks suggests a more general degradation occurred under these harsher (higher temperature) conditions relative to the “partial dissolution”. However, no specific compounds could be identified.

Beaker Neutralization Test (5-mL). A 5-mL aliquot of “dissolved SuperLig[®] 644 solution” (8L = 8 liquid) was placed in a beaker, and 5 molar sodium hydroxide was added to achieve pH 12-13 (by pH paper). For the RSST test, a 5-mL aliquot of “SuperLig[®] 644 solution” was loaded into the RSST. The amount of sodium hydroxide required to attain pH 12-13 in the 5-mL beaker test was added externally to a sealed Parr bomb. A syringe was used to force the sodium hydroxide through an extension tube into the test cell. Sample was not ejected during the neutralization tests, and no foaming or precipitation was observed.

The neutralization (N) test of the 8L solution was performed four times (8L-N1, 8L-N2, 8L-N3 and 8L-N4) with pH 7 being exceeded twice, as shown in **Table 5**. No foaming was observed during the neutralization reaction. The results indicate that little nitric acid was consumed in the dissolution, as the equal volume of 5M sodium hydroxide solution very nearly neutralized the “dissolved SuperLig[®] 644 solution”. Interestingly, a distinct orange-yellow to golden/brown color change occurred at approximately pH 7.

The RSST profiles in Figures 23-26 are consistent with a rapid, exothermic acid-base neutralization reaction. Figure 24 shows a small net pressure change that is associated with the addition of a liquid to a sealed system.

4. Prolonged Contacting of SuperLig[®] 644 resin with deionized water at intermediate temperature

Work described in this section was intended to determine if the resin will completely dissolve in deionized water at 60 °C.

Test 9. SuperLig[®] 644 resin (0.4 grams) and deionized water (10.0 grams) were loaded into the RSST, and containment was closed without backpressure. The system was heated from ambient to 60 °C at a rate of 15 °C/hr (0.25 °C/min), and held at about 60 °C for one week. A same-scale beaker test did not completely dissolve during the test period.

The off-gas was analyzed by VOA (Table 3, 9-gas). The dried solids and the evaporated and dried liquid samples were analyzed by DSC (Table 4, Figures 38 and 39). The analysis of the gas, liquid and solid samples identified no reactive compounds.

The RSST reaction profiles in Figures 27-30 reflect temperature changes due to daily shutdown and restarts, however, no exotherms or net pressure change was identified. As expected, this evidence suggests gases were not generated from the contact of water and SuperLig[®] 644 resin.

The 17% mass lost in the test was attributed to solvent evaporation (9.3 g total mass remaining) (which condenses on the vessel wall). The residue recovered from the evaporation of 1 mL of liquid was 28.4 mg. The mass of resin that dissolved was 0.231 g (55%). The recovered solid had a mass of 0.1790 g (45%) after air and vacuum drying at room temperature. The mass balance of resin due to liquid and solid recovery was 100%. The gas was analyzed by VOA (Table 3, 9-gas), and the dried solids and evaporated and dried liquid samples were analyzed by DSC (Table 4). The analysis of the gas, liquid and solid samples identified no reactive compounds.

ICMS performed on brown liquid from Test 9 is shown in Figure 33. The mass spectrum contains a single peak at 223 m/z. Since this is the (M+1)⁺ peak, the molecular weight of this single component is 222 grams/mole. However, the identity of this component could not be determined.

Table 3. Analysis of Gases and Liquid Residue Using VOA, SVOA and SVOC

Test No.	Analyte, mg/L			Gas Concentrations, %			Method
	Aliphatic Hydrocarbons	Ethers and Oxygenated Organics	Toluene	N ₂	CO ₂	N ₂ O	
1-gas	0.13	-	0.11	99%+	-	-	VOA
2-gas	0.024	0.035	0.061	99%+	-	-	VOA
8-gas	0.21	0.052	0.2	96	3.0	1.0	VOA
9-gas	0.088	0.025	0.097	99%+	0.28		VOA
B1_N2	-	0.031	-	99%+			VOA
B2_air	-	0.020	-	99%+	0.033		VOA
8-liquid		4.9					SVOC

1 = Resin and 1 M NaMnO₄R

2 = Resin and 1 M NaMnO₄R

8 = Resin and 5 M nitric heated to 25 °C

9 = Resin and water heated to 60 °C

B1_N2 = blank run 1 containing nitrogen

B2_air = blank run 2 containing air

Table 4. Analysis of Solid and Liquid Residue DSC

Test No.	Peak Temperature °C	Peak Height W/g
7-liquid	93.8	-2.5
	107	-0.07
	325	1.48
	356	0.3
7-solid	104	-0.44
	327	-0.17
	404	0.7
	410	0.47
	415	0.54
	423	0.18
8-liquid	91.1	-0.226
	110	0.548
	132	-0.8
	336	2.6
	393	2.68
8-solid	87.5	-0.378
9-liquid	110	-3.2
	150	-0.47
	301	0.23
	336	1.3
	346	0.81
	356	1.02
	502	0.52
9-solid	119	-0.629
	329	-0.2
	400	0.17
	442	0.381

Table 5. Caustic Neutralization of SuperLig[®] 644 solution

Test	Mass of 5.000 mL of SL644 (aq) g	Mass of 5M NaOH g	Final pH	Solution Color	Evidence of Solids, Sample Ejected or Foam
8L-N1	5.895	6.1	2	orange/yellow	None
8L-N2	5.916	6.7	4	golden/yellow-brown	None
8L-N3	5.907	6.7	>11	golden/brown	None
8L-N4	5.896	7.3	>11	golden/brown	None

CONCLUSIONS

This task addressed four items related to SuperLig[®] 644 ion exchange resin stability under nominal to extreme conditions.

1. A process upset analysis of resin in contact with 1 molar sodium permanganate at 25 and 40 °C identified runaway reaction behavior that was characterized by acute self-heating and sample ejection, however, only condensable gas was evolved. An analogous test of the resin-permanganate reaction under 2000 kPa prevented sample ejection, displayed a temporary pressure rise, but also showed no net evolution of noncondensable gases.
2. A simulation of the accelerated aging of resin during normal dilute acid regeneration operations identified no measurable reactivity. The 24-hour treatment at 90 °C in 0.5M nitric acid dissolved 44% of the resin. One precaution is that the addition of even dilute nitric acid to dry resin results in rapid foaming, although no self-heating was observed.
3. The behavior of resin in 5M nitric acid at ambient temperatures was characterized as a slow steady evolution of non-condensable gases (NO_x and CO₂), and 78% of the resin dissolved after 21 days. Caustic neutralization of a “SuperLig[®] 644 resin solution” was characterized as a rapid exothermic reaction without foaming, bubbling, or precipitation.
4. Resin exposure to water at 60 °C showed no measurable gas evolution, and dissolved 55% of the resin after 7 days.
5. Differential scanning calorimetry (DSC) analysis of solid and liquid residues from nitric acid and water tests showed small heat flows, evidence that the potential reactivity of these resin-nitric acid reaction products is small to negligible. Volatile organic analysis (VOA) did not identify any reactive compounds in the gaseous degradation products. Semi-volatile organic analysis (SVOA) of a liquid residue did not identify typical low molecular weight resin degradation products. For the detection of low molecular weight organic acids, liquid chromatography-mass spectrometry (LC-MS) of samples was performed. Since the resin structure is proprietary, the mass peaks could not be assigned to specific compounds.
6. Dissolution of 55% of the resin in warm water was not anticipated. A portion of the mass loss may be attributable to dissolution of potassium, which was recently identified by IBC Advanced Technologies as present in this resin batch. The measured concentration of potassium is 15.5 wt% of the as-received mass. It is also known that approximately 8% of the as-received mass is due to absorbed moisture, which would have been removed in this experiment. Since the solution is colored, it may also contain low molecular weight polymerized ligand. The possibility of the presence of a low molecular weight polymer has implications for the fate of the wash solution. If the polymer contains the cesium-binding ligand, it may bind the cesium in solution, prohibiting absorption by the column.

The resin wash solutions should not be combined with ion exchange feed solution until it can be proved that this does not occur.

The objective of this work was to characterize the reactivity of SuperLig[®] 644 and identify a margin of safety for plant process operations. These results are intended to be used by the design authority, along with additional testing that has been performed at SRTC and elsewhere, to determine the impact on facility design safety. The design authority will develop the bounding facility conditions (e.g., safe concentration of permanganate solution, rupture disk capacity, etc.). In summary, this work characterized the reactivity SuperLig[®] 644 ion exchange resin under various conditions as part of the validation of waste treatment technologies to be employed by the Hanford River Protection Project.

Figure 3. SuperLig® 644 resin and 1 molar sodium permanganate

AT 0 PASCAL: TEMPERATURE VS. TIME

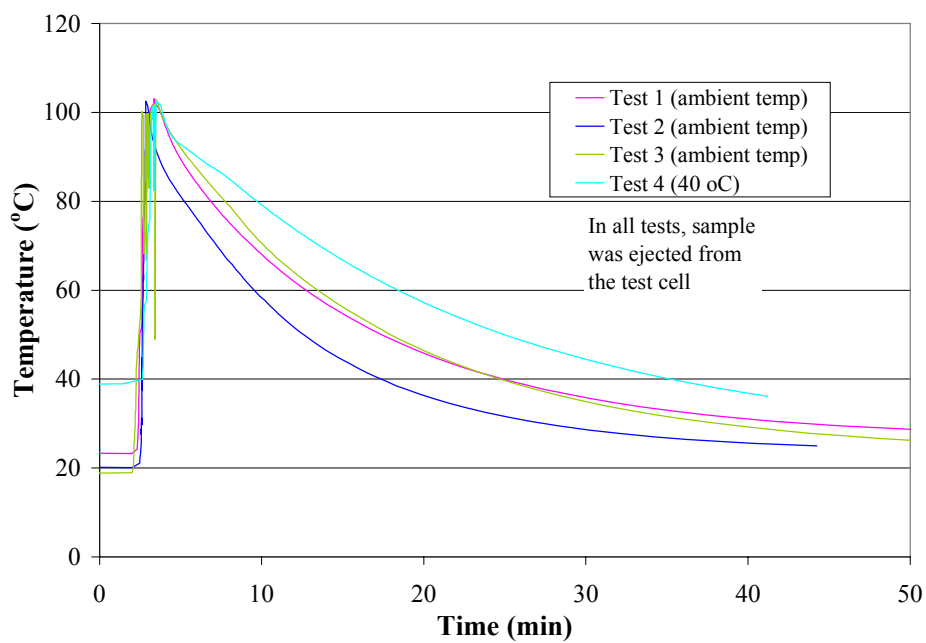


Figure 4. SuperLig® 644 resin and 1 molar sodium permanganate

0 PASCAL: PRESSURE VS. TIME

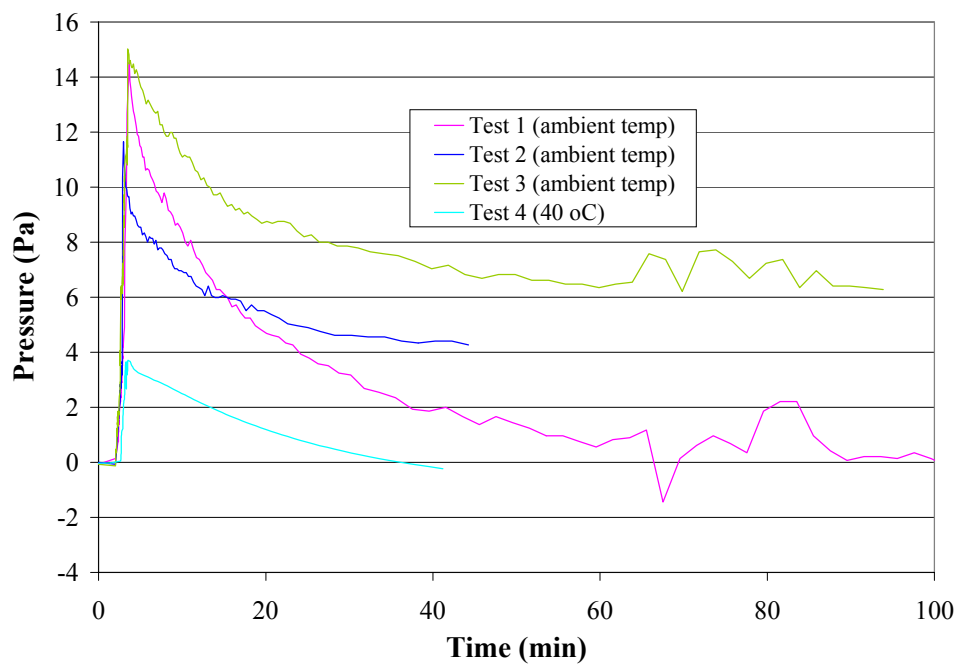


Figure 5. SuperLig[®] 644 resin and 1 molar sodium permanganate
AT 0 PASCAL: SELF-HEATING VS. TEMPERATURE

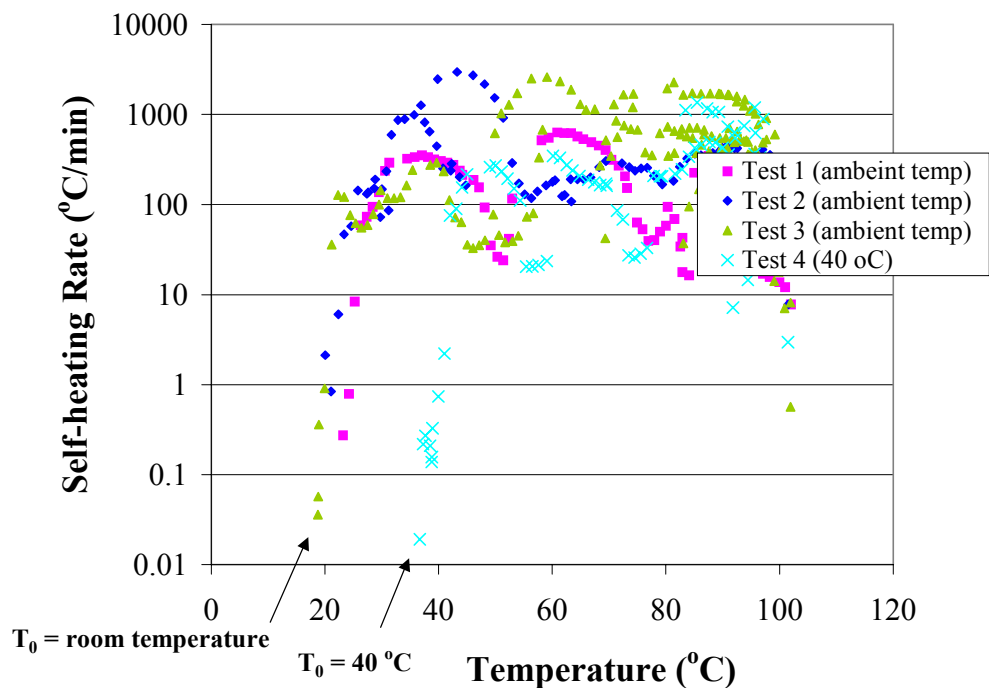


Figure 6. SuperLig[®] 644 resin and 1 molar sodium permanganate
AT 0 PASCAL: PRESSURE RATE VS. TEMPERATURE

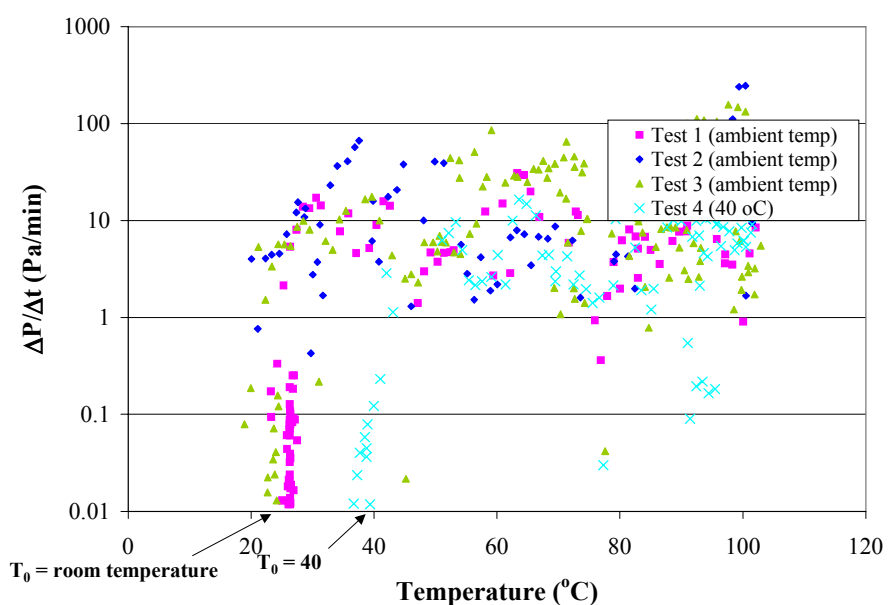


Figure 7. SuperLig[®] 644 resin and Water
AT 2000 KILOPASCAL: TEMPERATURE VS. TIME

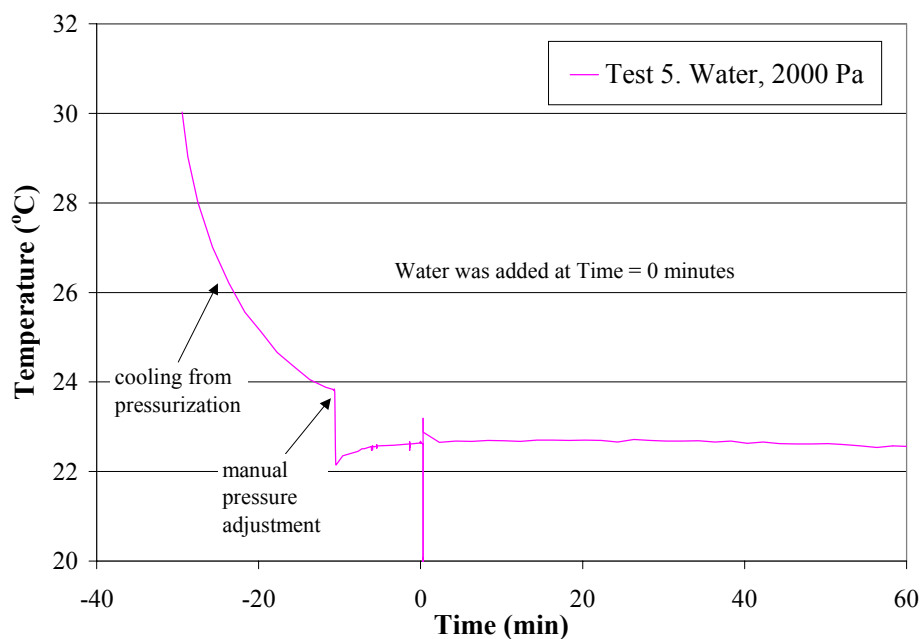


Figure 8. SuperLig[®] 644 resin and Water
AT 2000 KILOPASCAL: PRESSURE VS. TIME

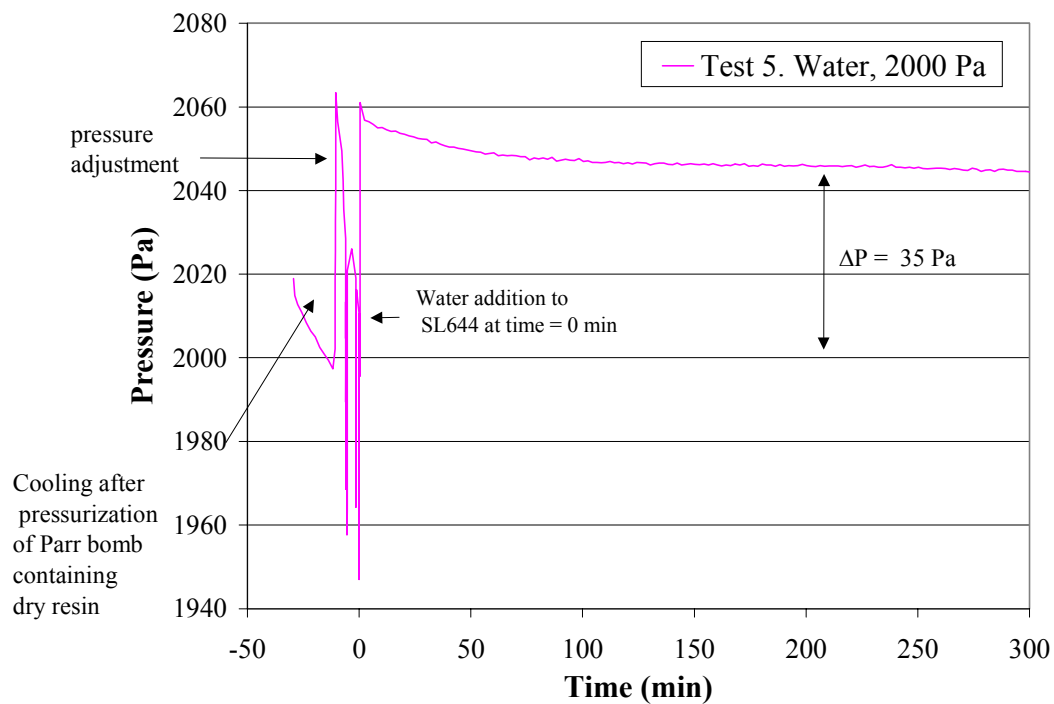


Figure 9. SuperLig[®] 644 resin and Water
AT 2000 KILOPASCAL: SELF-HEATING VS. TEMPERATURE

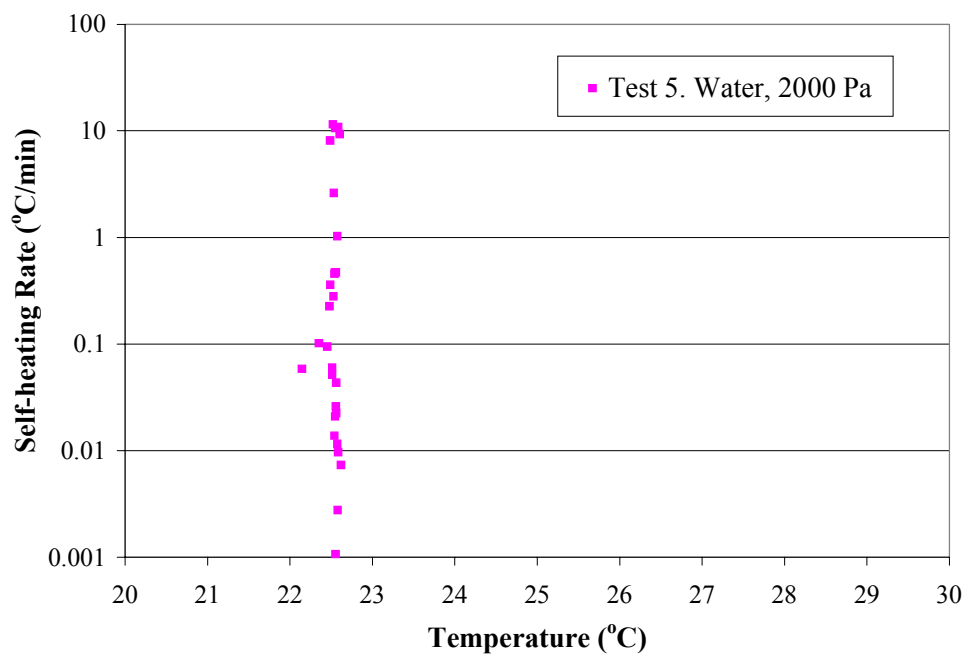


Figure 10. SuperLig[®] 644 resin and Water
AT 2000 KILOPASCAL: PRESSURE RATE VS. TEMPERATURE

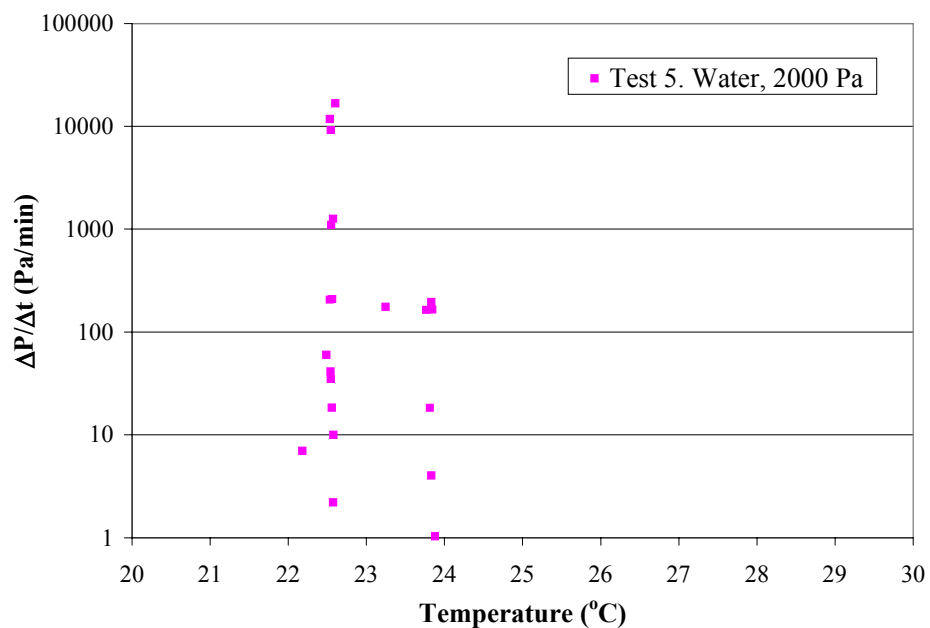


Figure 11. SuperLig[®] 644 resin and 1 molar sodium permanganate
AT 2000 Pa: TEMPERATURE VS. TIME

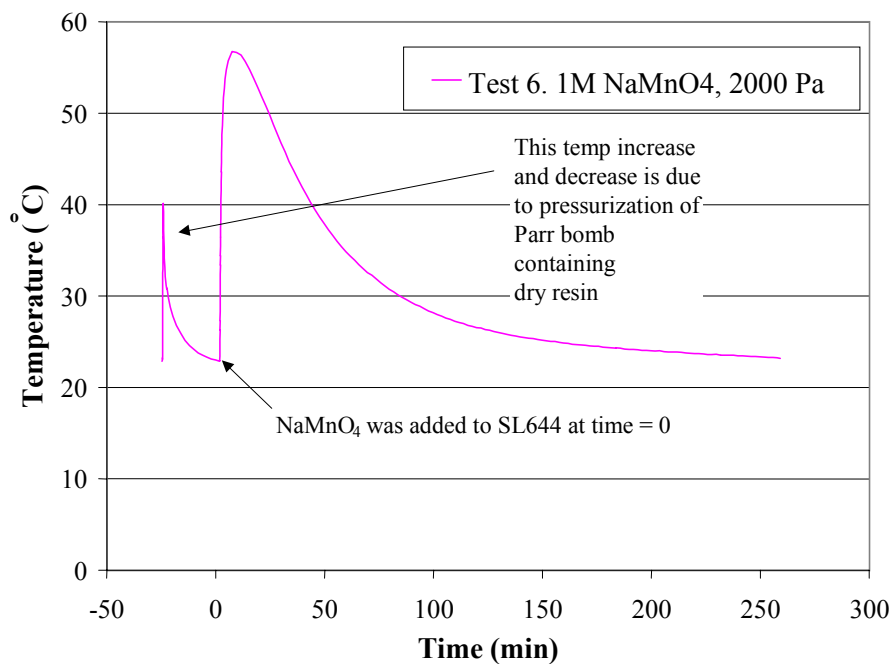


Figure 12. SuperLig[®] 644 resin and 1 molar sodium permanganate
AT 2000 Pa: PRESSURE VS. TIME

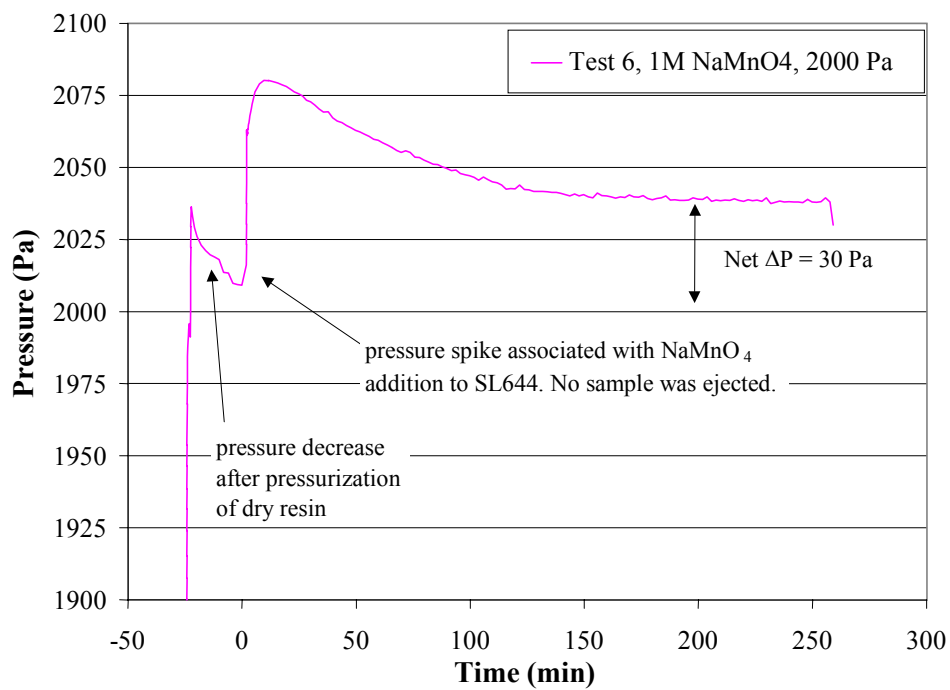


Figure 13. SuperLig[®] 644 resin and 1 molar sodium permanganate
AT 2000 PA: SELF-HEATING VS. TEMPERATURE

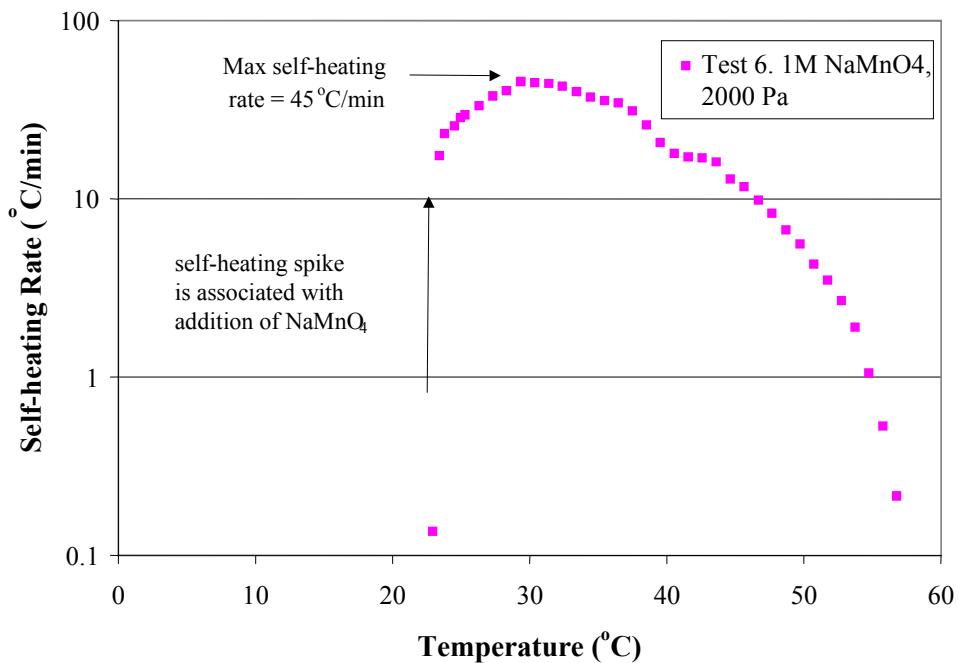


FIGURE 14. SUPERLIG[®] 644 RESIN AND 1 MOLAR SODIUM PERMANGANATE
AT 2000 PA: PRESSURE RATE VS. TEMPERATURE

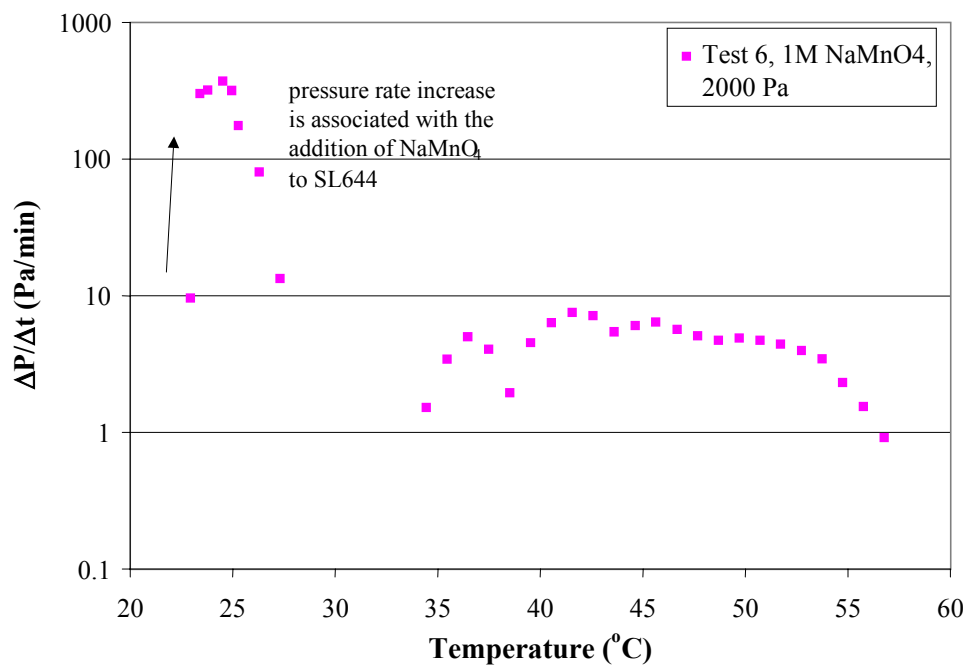


Figure 15. SuperLig[®] 644 resin and 0.5 molar nitric Acid
AT 0 PASCAL: TEMPERATURE VS. TIME

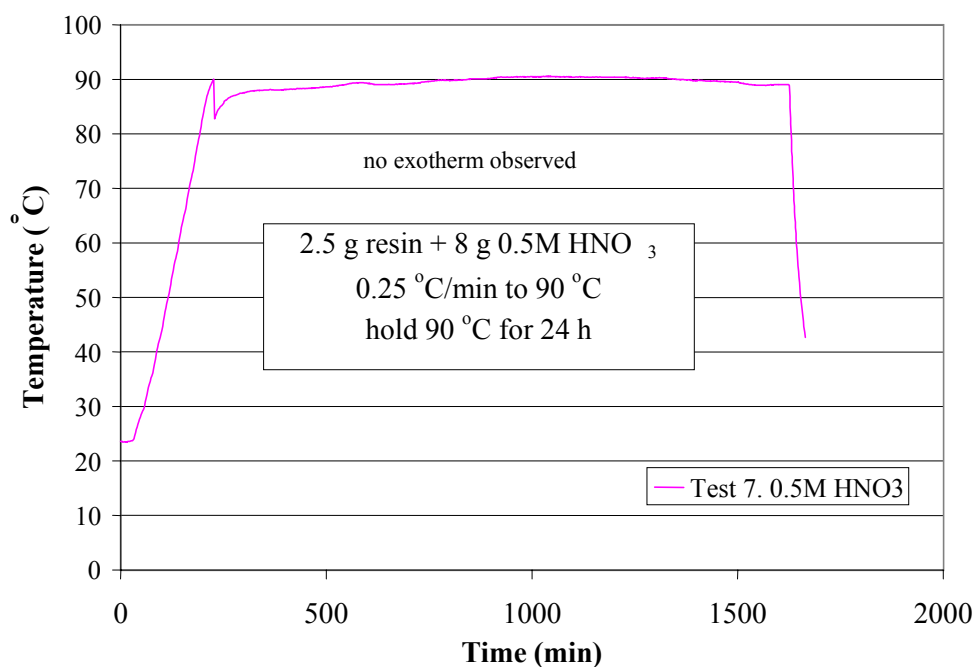


FIGURE 16. SUPERLIG[®] 644 RESIN AND 0.5 MOLAR NITRIC ACID
AT 0 PASCAL: PRESSURE VS. TIME

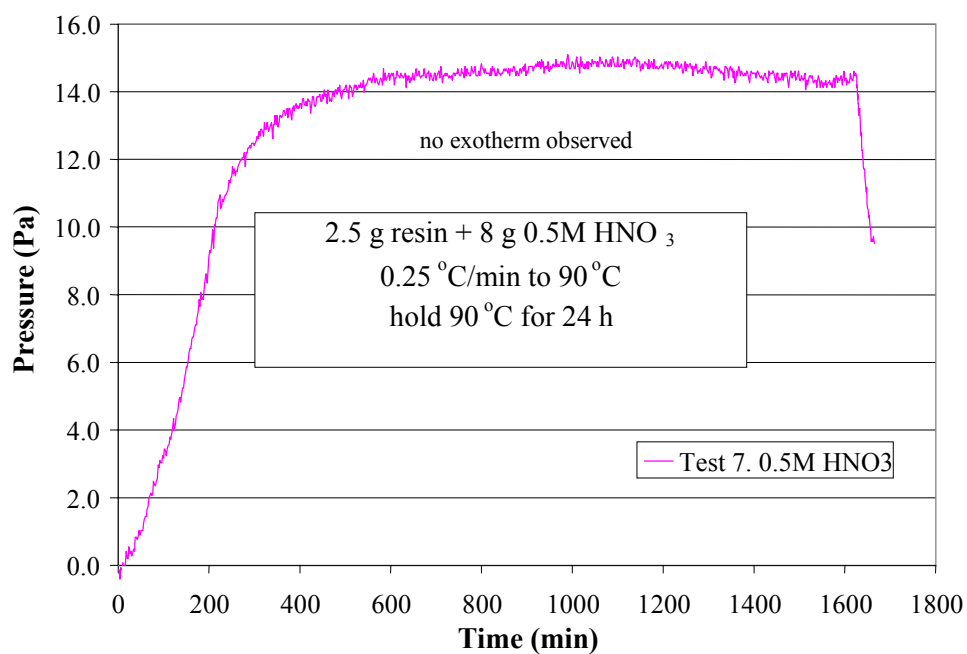


Figure 17. SuperLig[®] 644 resin and 0.5 molar nitric Acid
AT 0 PASCAL: SELF-HEATING VS. TEMPERATURE

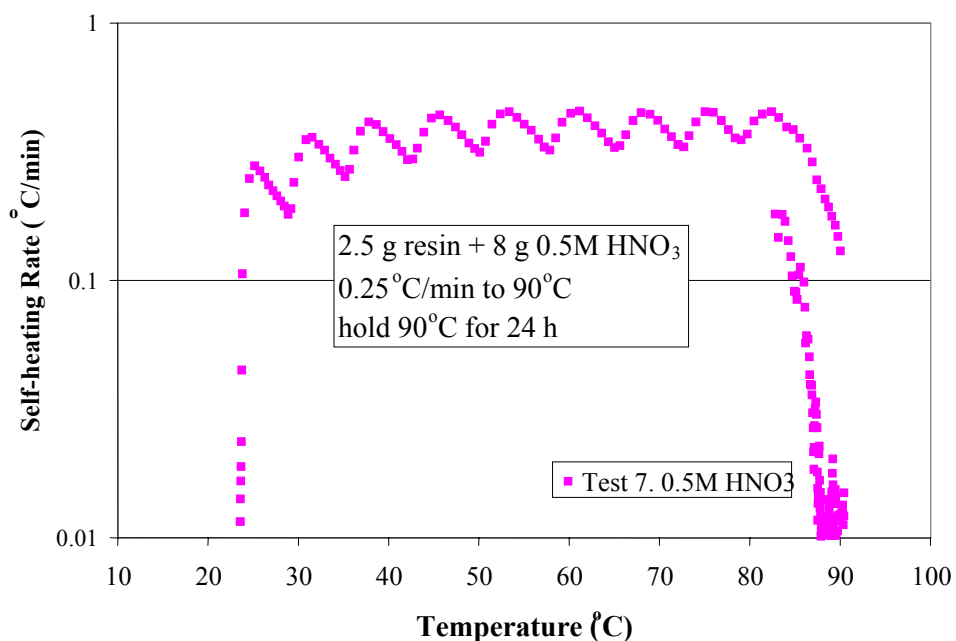


Figure 18. SuperLig[®] 644 resin and 0.5 molar nitric Acid
AT 0 PASCAL: PRESSURE RATE VS. TEMPERATURE

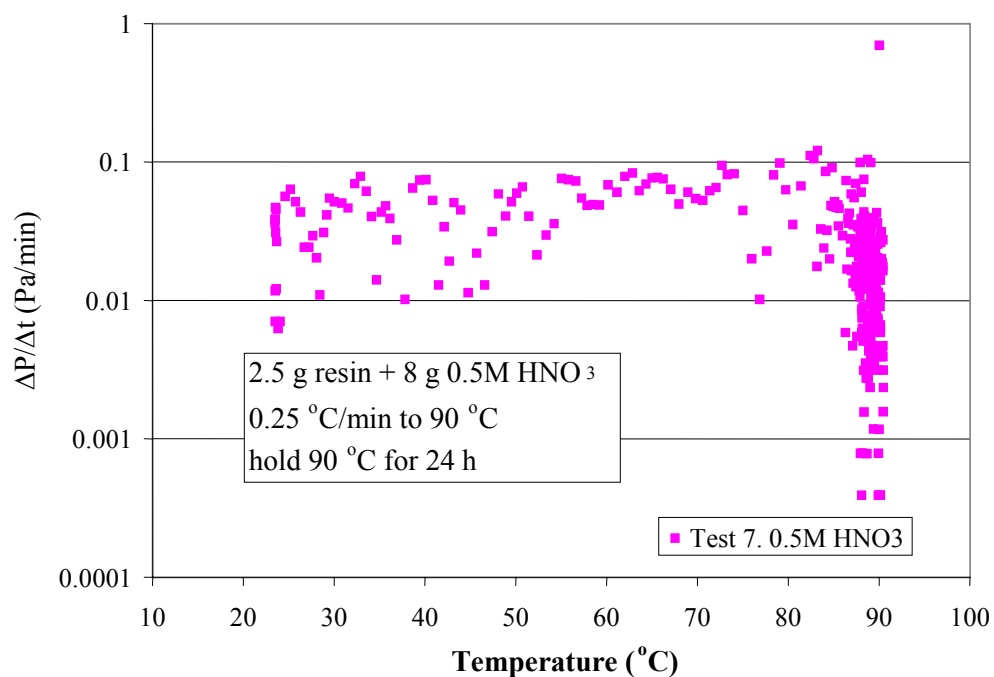


Figure 19. SuperLig[®] 644 resin and 5 molar Nitric Acid

FROM AMBIENT T: TEMPERATURE VS. TIME

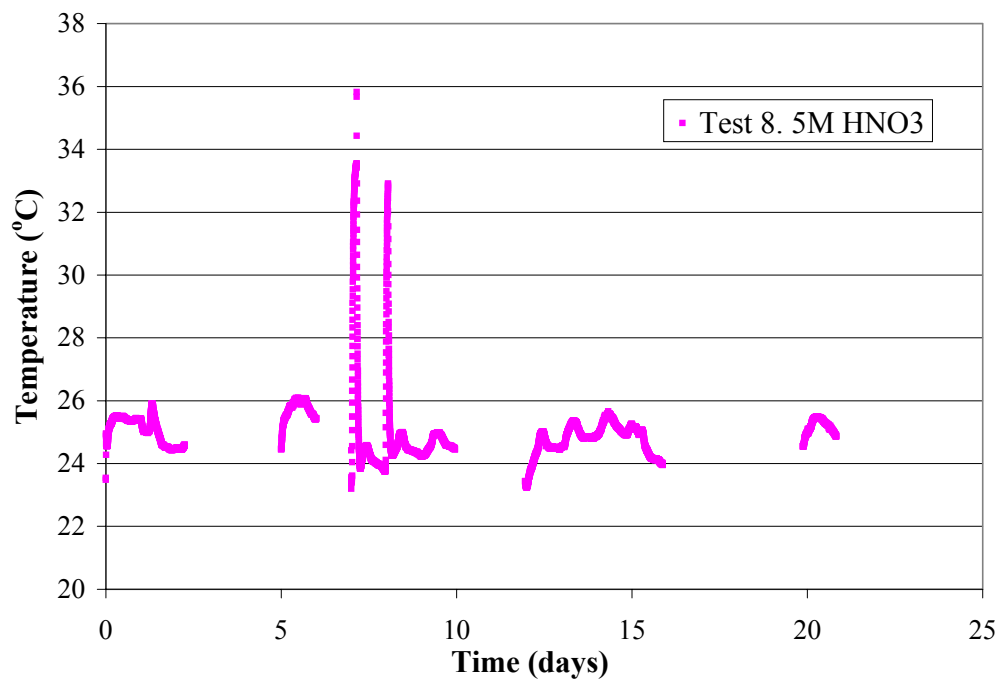


Figure 20. SuperLig[®] 644 resin and 5 molar Nitric Acid

FROM AMBIENT T: PRESSURE VS. TIME

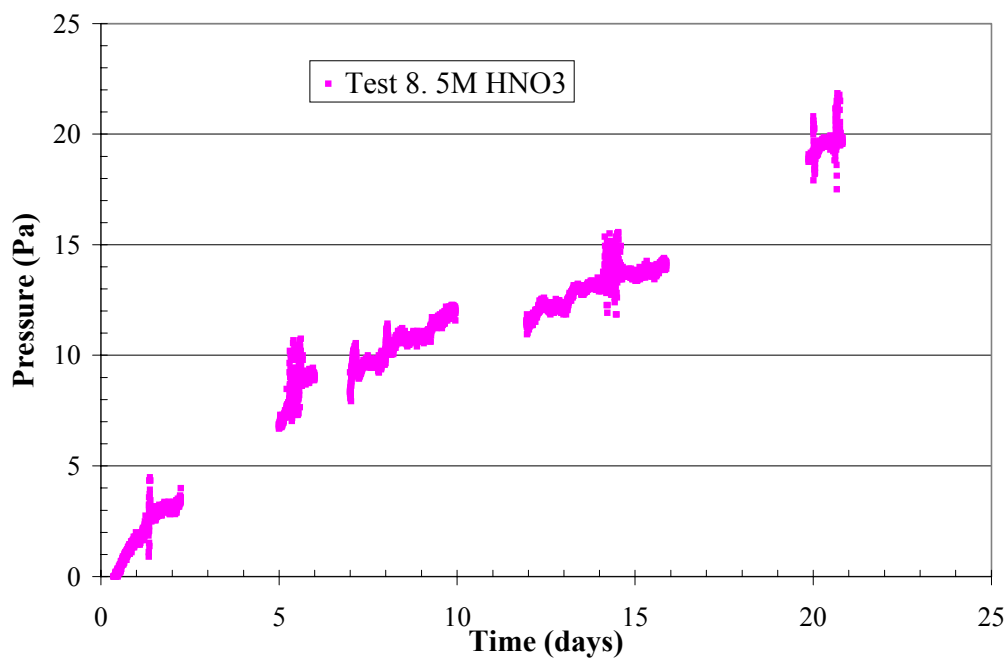


Figure 21. SuperLig[®] 644 resin and 5 molar Nitric Acid
FROM AMBIENT T: SELF-HEATING VS. TEMPERATURE

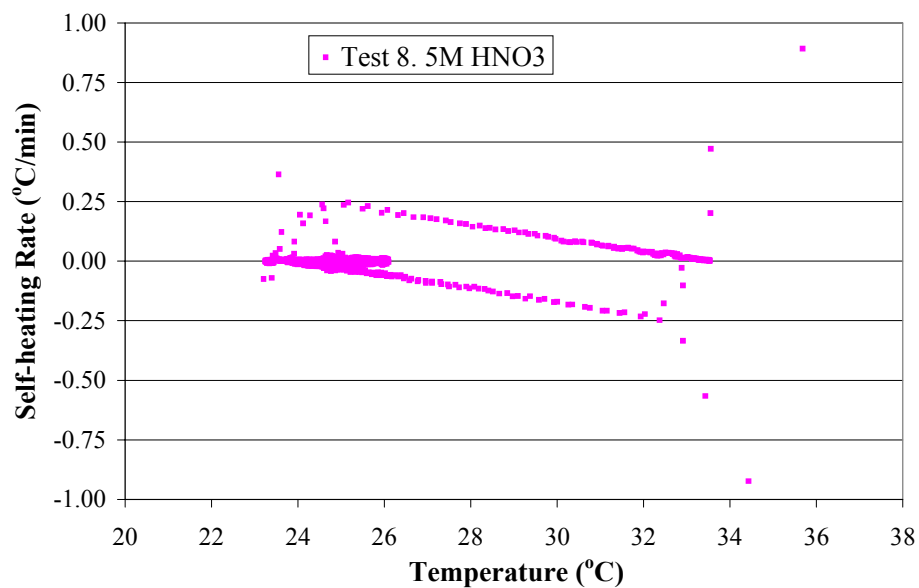


Figure 22. SuperLig[®] 644 resin and 5 molar Nitric Acid
FROM AMBIENT T: PRESSURE RATE VS. TEMPERATURE

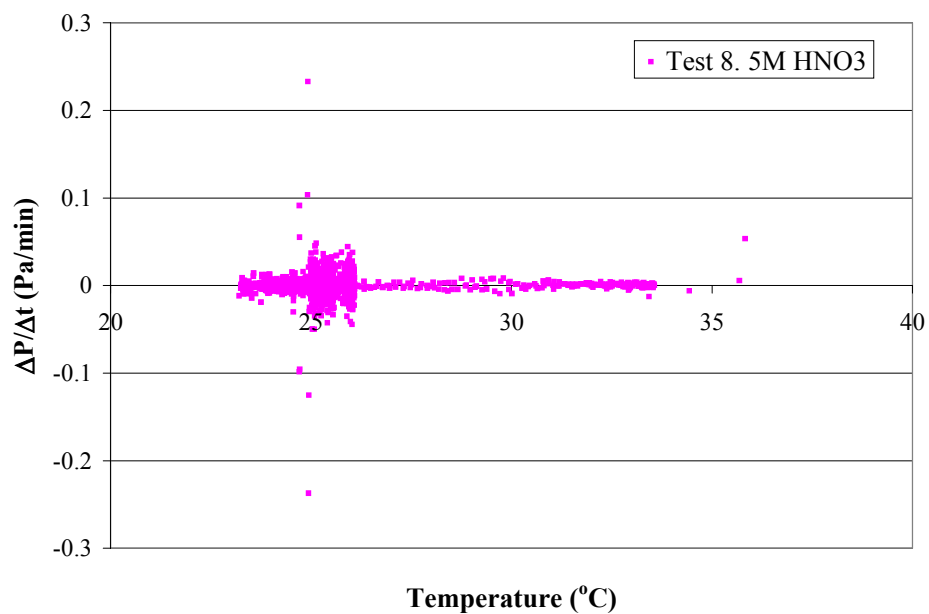


Figure 23. Caustic Neutralization of “SuperLig® 644 solution”

AT 0 PASCAL: TEMPERATURE VS. TIME

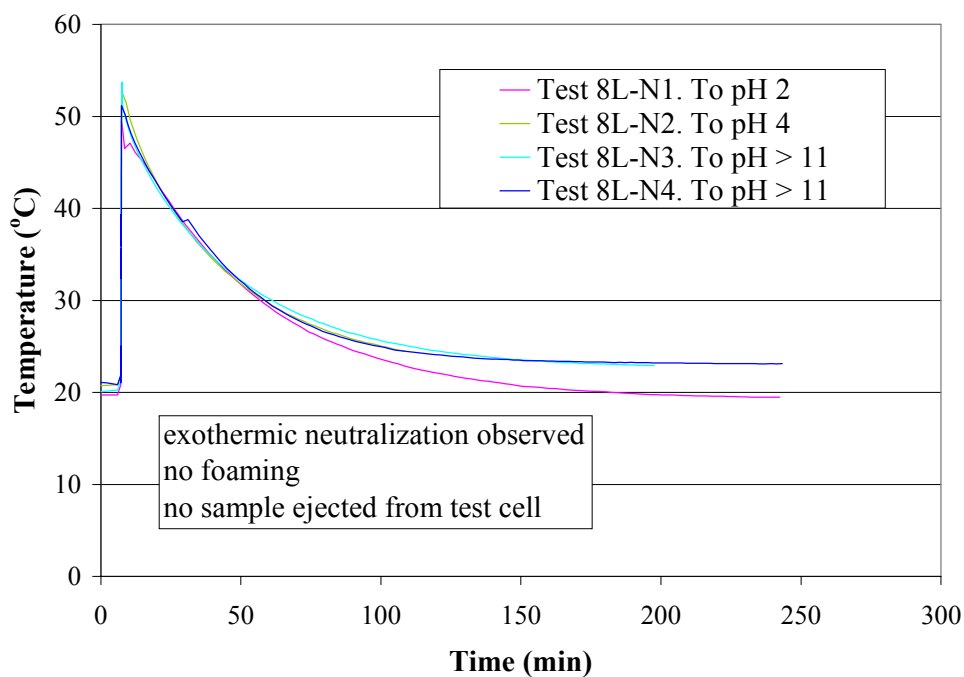


Figure 24. Caustic Neutralization of “SuperLig® 644 solution”

AT 0 PASCAL: PRESSURE VS. TIME

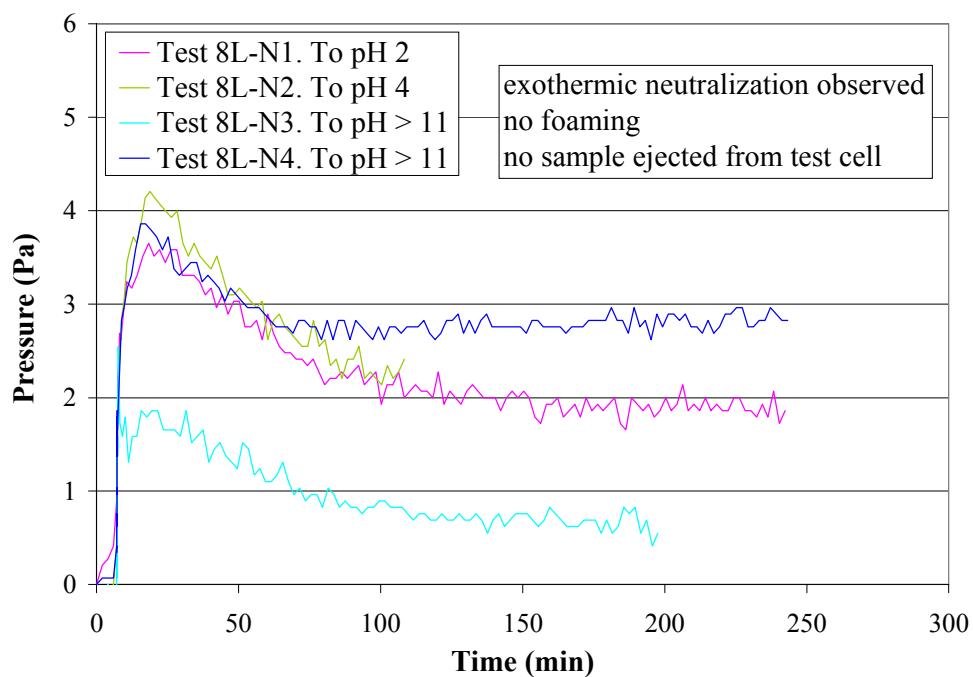


Figure 25. Caustic Neutralization of “SuperLig® 644 solution”

AT 0 PASCAL: SELF-HEATING VS. TEMPERATURE

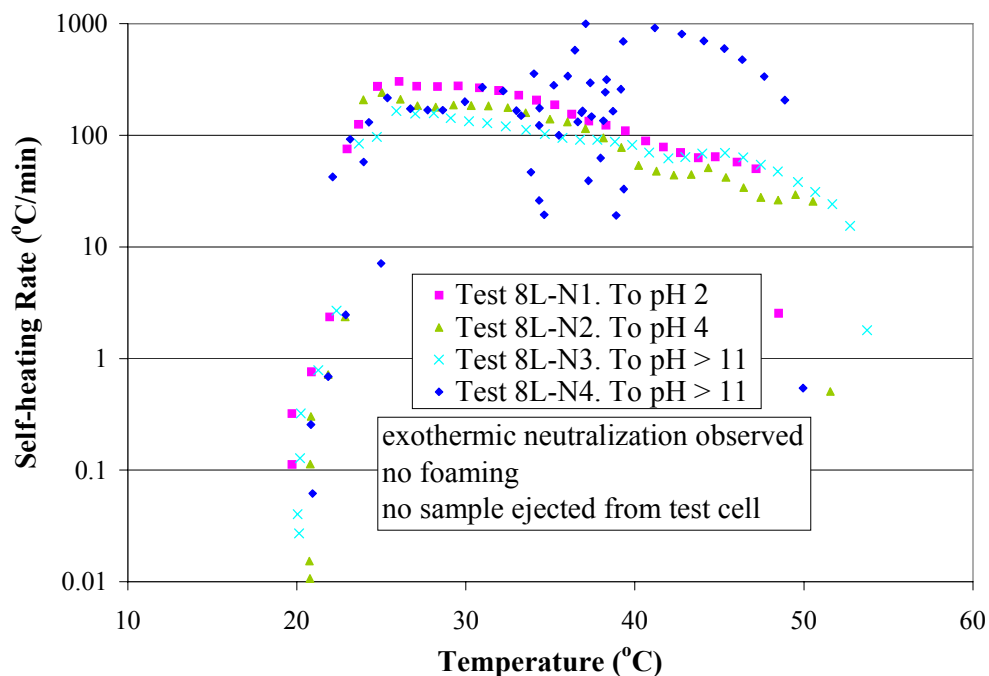


Figure 26. Caustic Neutralization of “SuperLig® 644 solution”

AT 0 PASCAL: PRESSURE RATE VS. TEMPERATURE

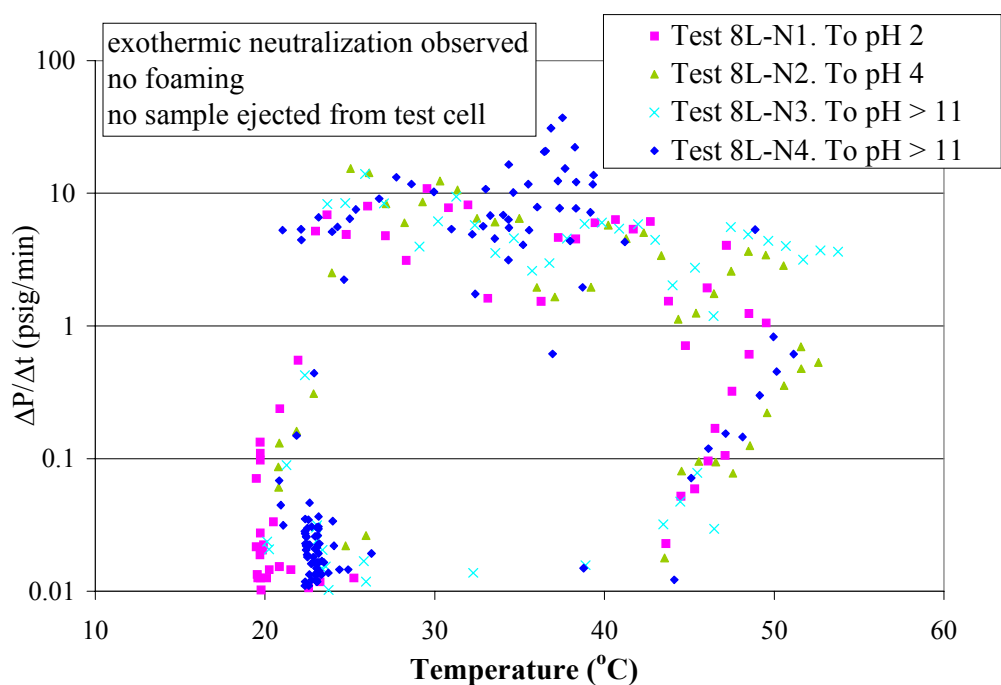


Figure 27. SuperLig[®] 644 resin and Water
FROM AMBIENT T: TEMPERATURE VS. TIME

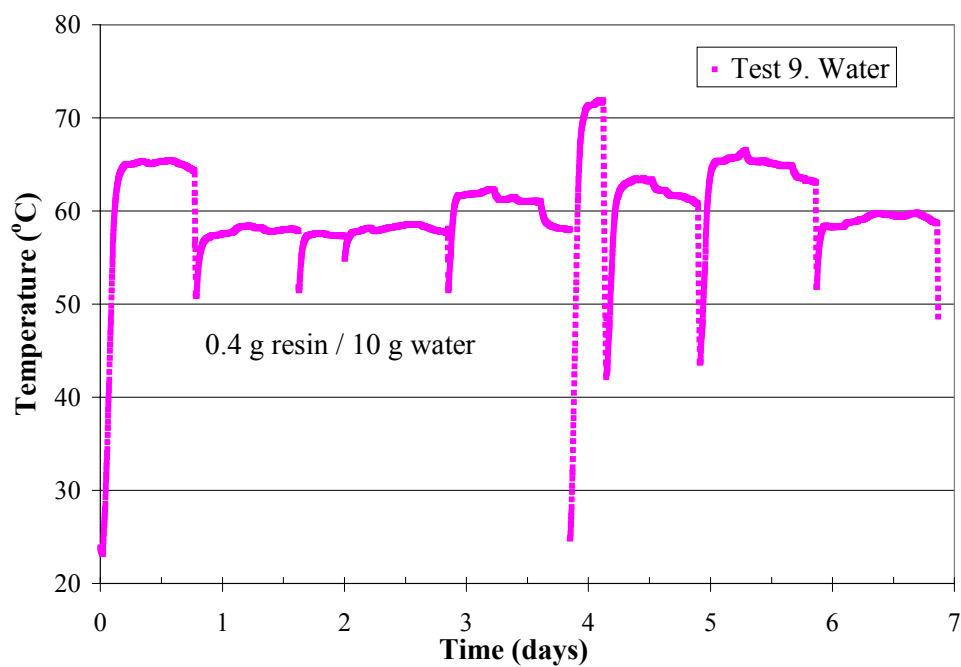


Figure 28. SuperLig[®] 644 resin and Water
FROM AMBIENT T: PRESSURE VS. TIME

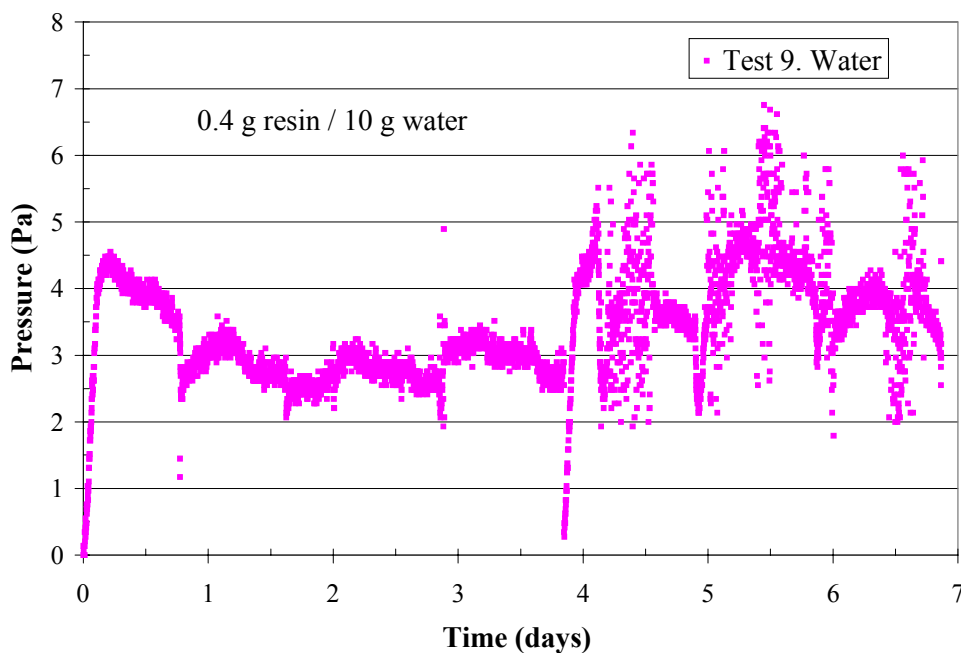


Figure 29. Caustic Neutralization of “SuperLig[®] 644 solution”

AT 0 PA: SELF-HEATING VS. TEMPERATURE

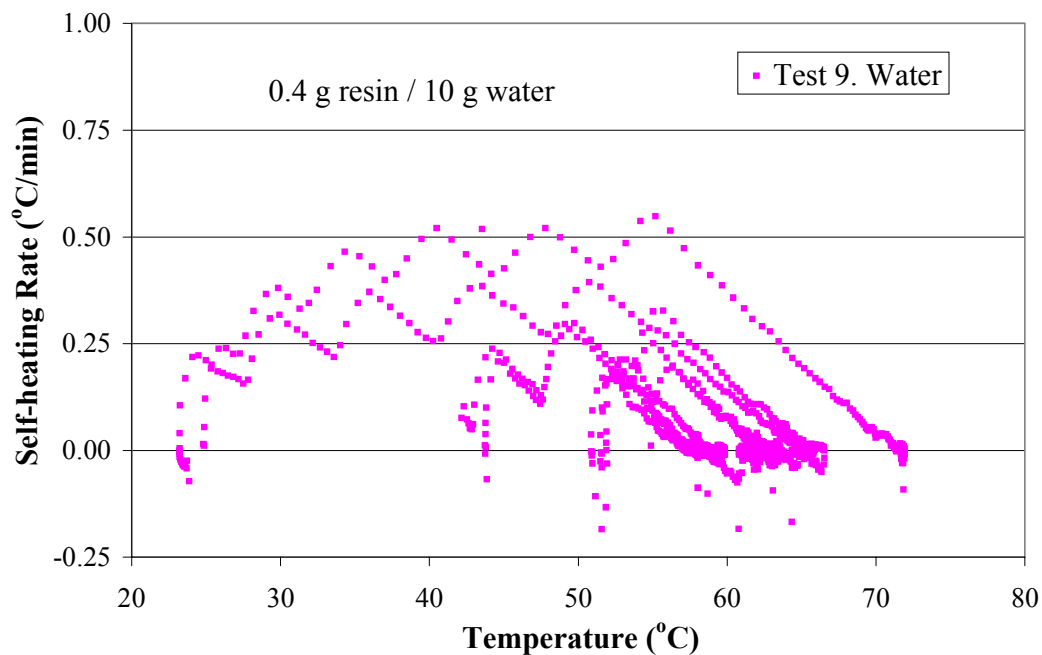


Figure 30. Caustic Neutralization of “SuperLig[®] 644 solution”

AT 0 PA: PRESSURE RATE VS. TEMPERATURE

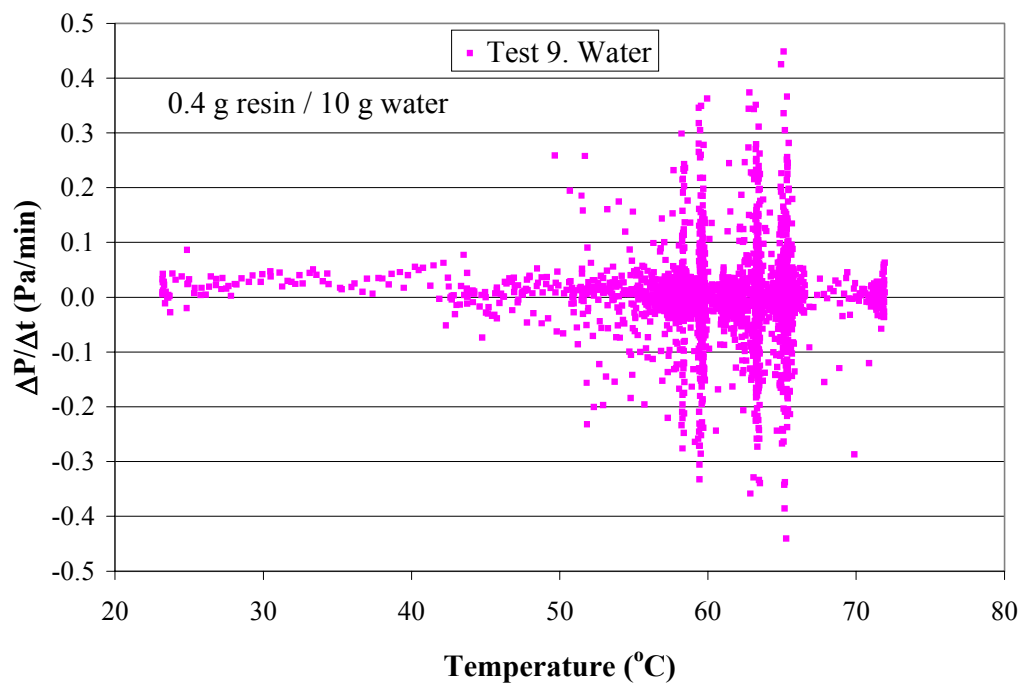


Figure 31. ICMS of the product from Test 8: "Partial" dissolution of SL644 in 5M nitric acid at 25 °C for 21 days

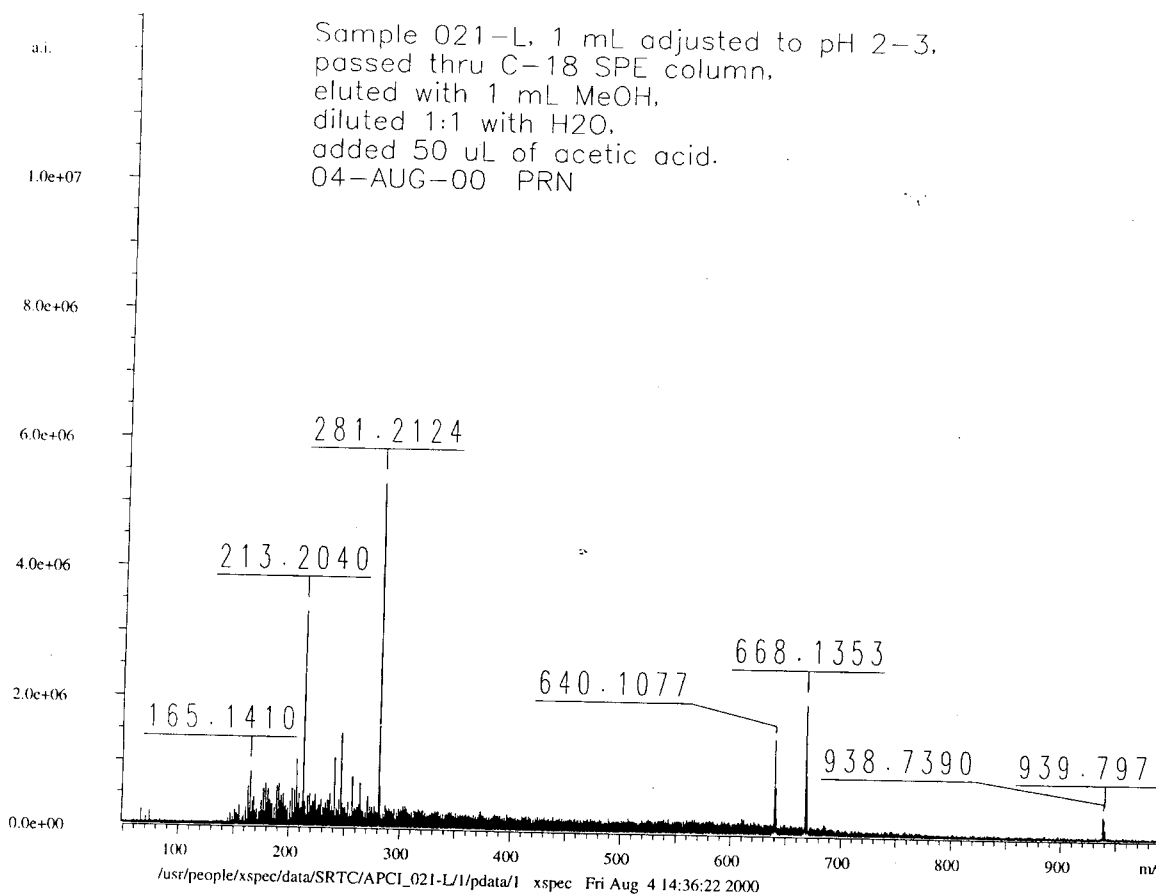


Figure 32. ICMS of the product from Test 8: “Complete” dissolution of SL644 in 5M nitric acid at 90 °C for 8 hours

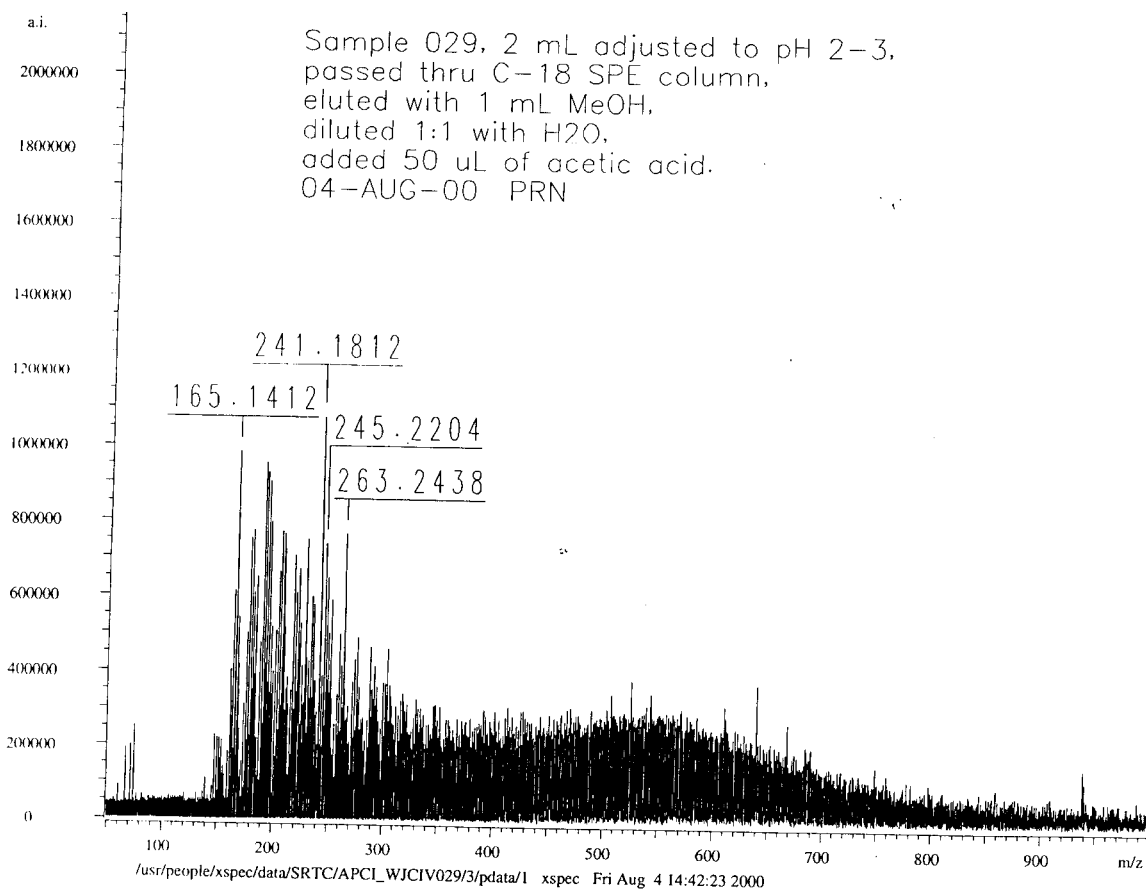


Figure 33. ICMS of the product from Test 9: Partial dissolution of SL644 in Distilled Water at 60 °C for 7 days

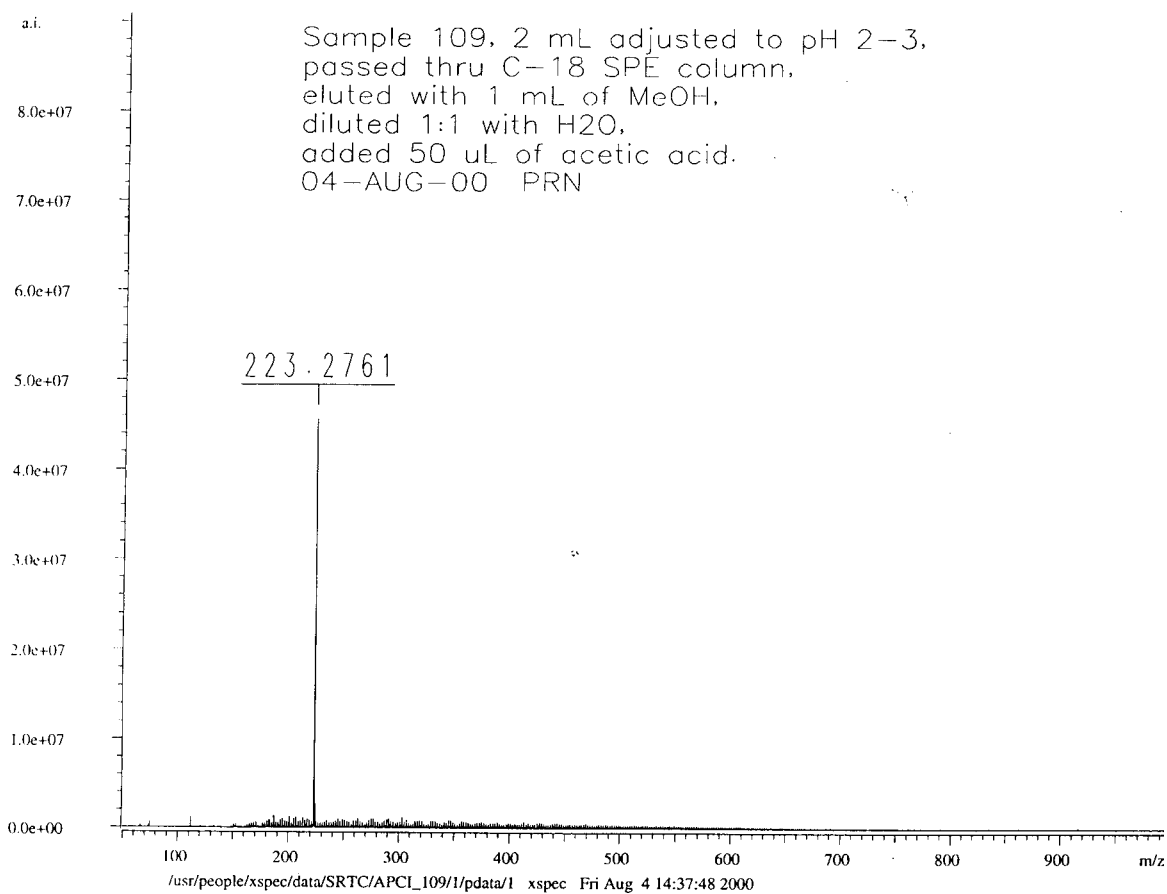


Figure 34. DSC of Test 7: Resin + 0.5M HNO₃ at 90 °C for 24 hours → Liquid Residue

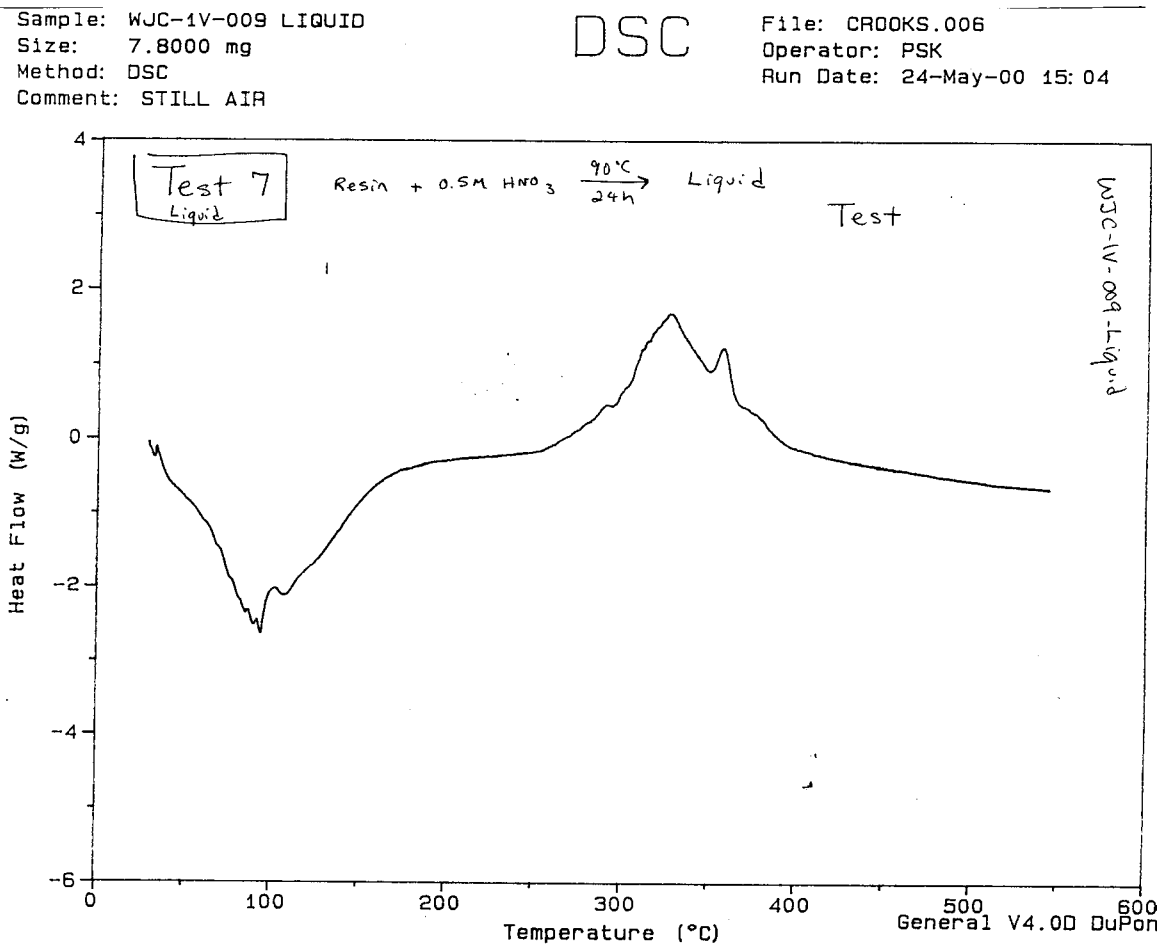


Figure 35. DSC of Test 7: Resin + 0.5M HNO₃ at 90 °C for 24 hours → Solid Residue

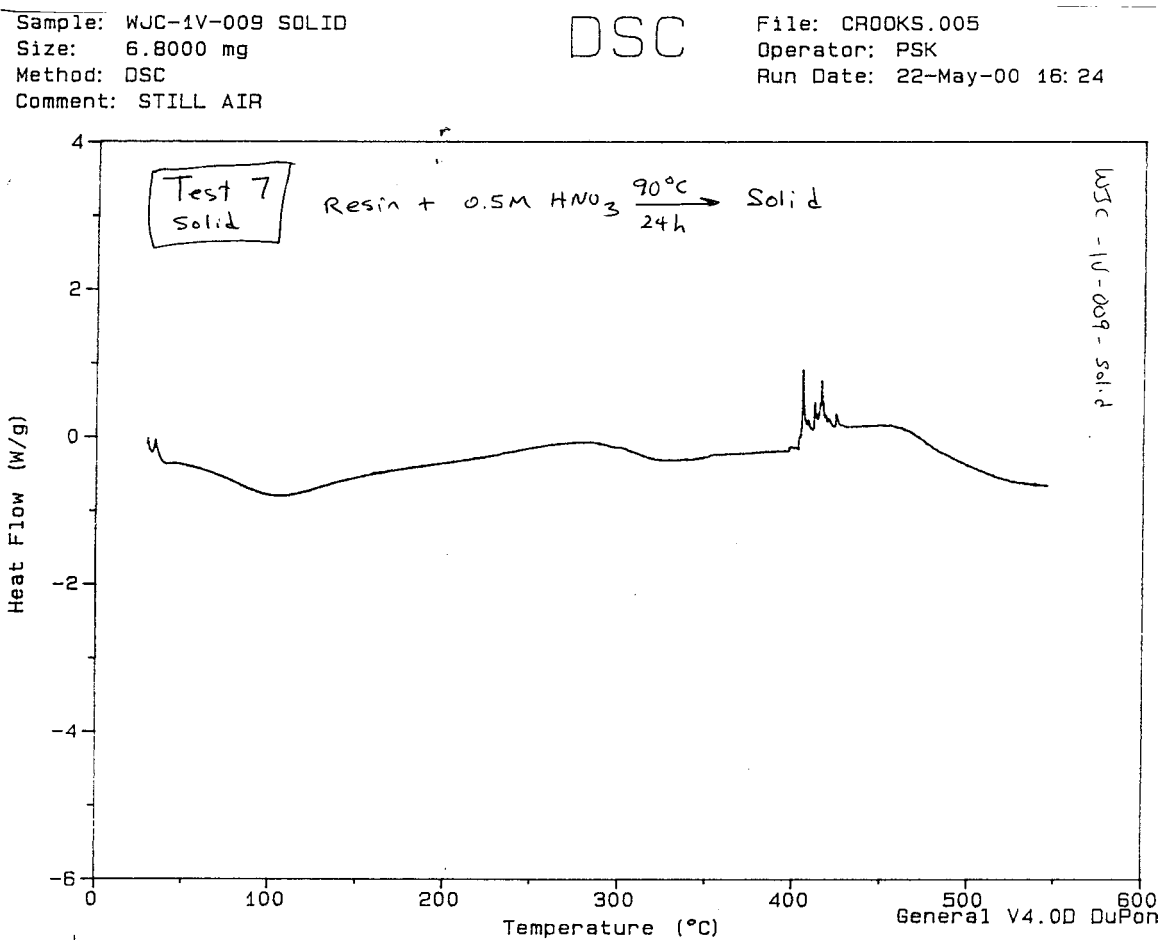


Figure 36. DSC of Test 8: Resin + 5M HNO₃ at ambient temperature for 21 days → Liquid Residue

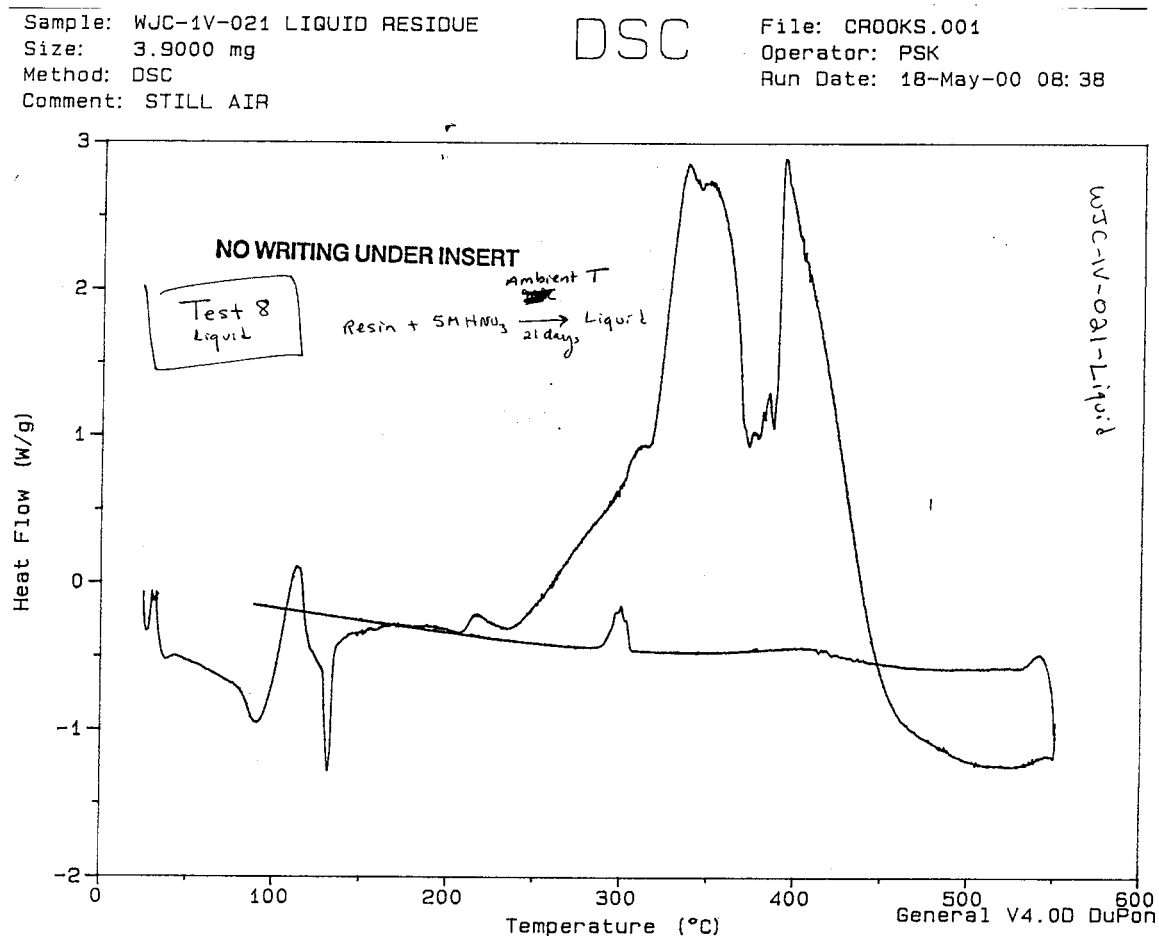


Figure 37. DSC of Test 8: Resin + 5M HNO₃ at ambient temperature for 21 days → Solid Residue

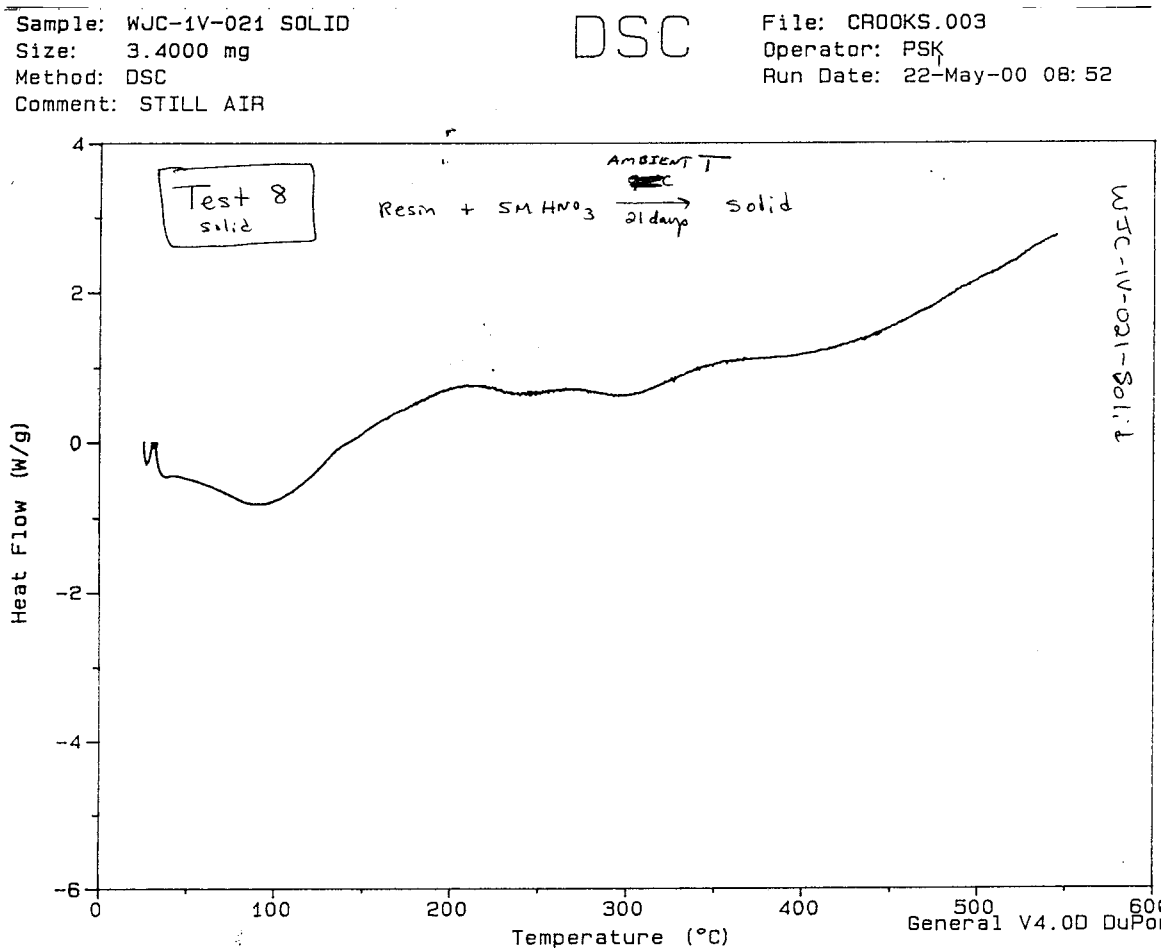


Figure 38. DSC of Test 9: Resin + H₂O at 60 °C for 7 days → Liquid Residue

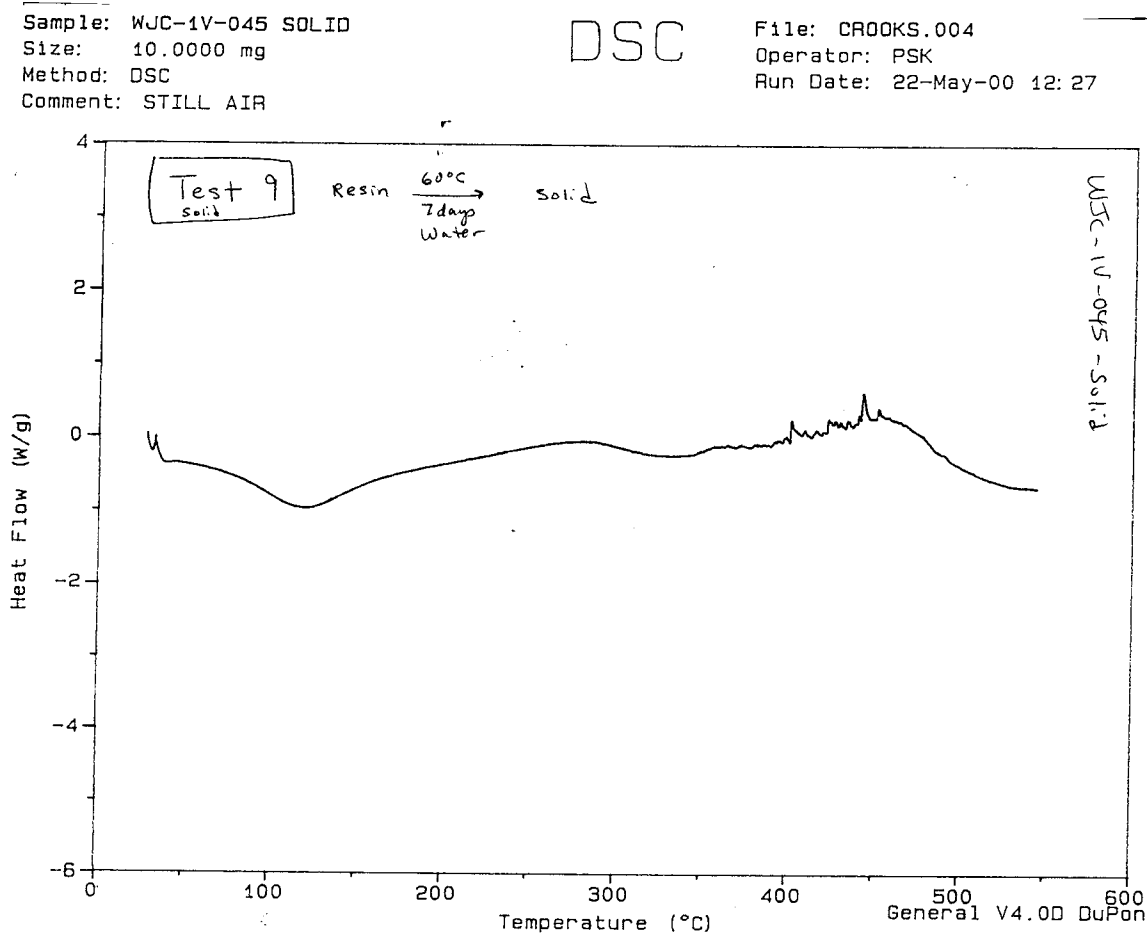
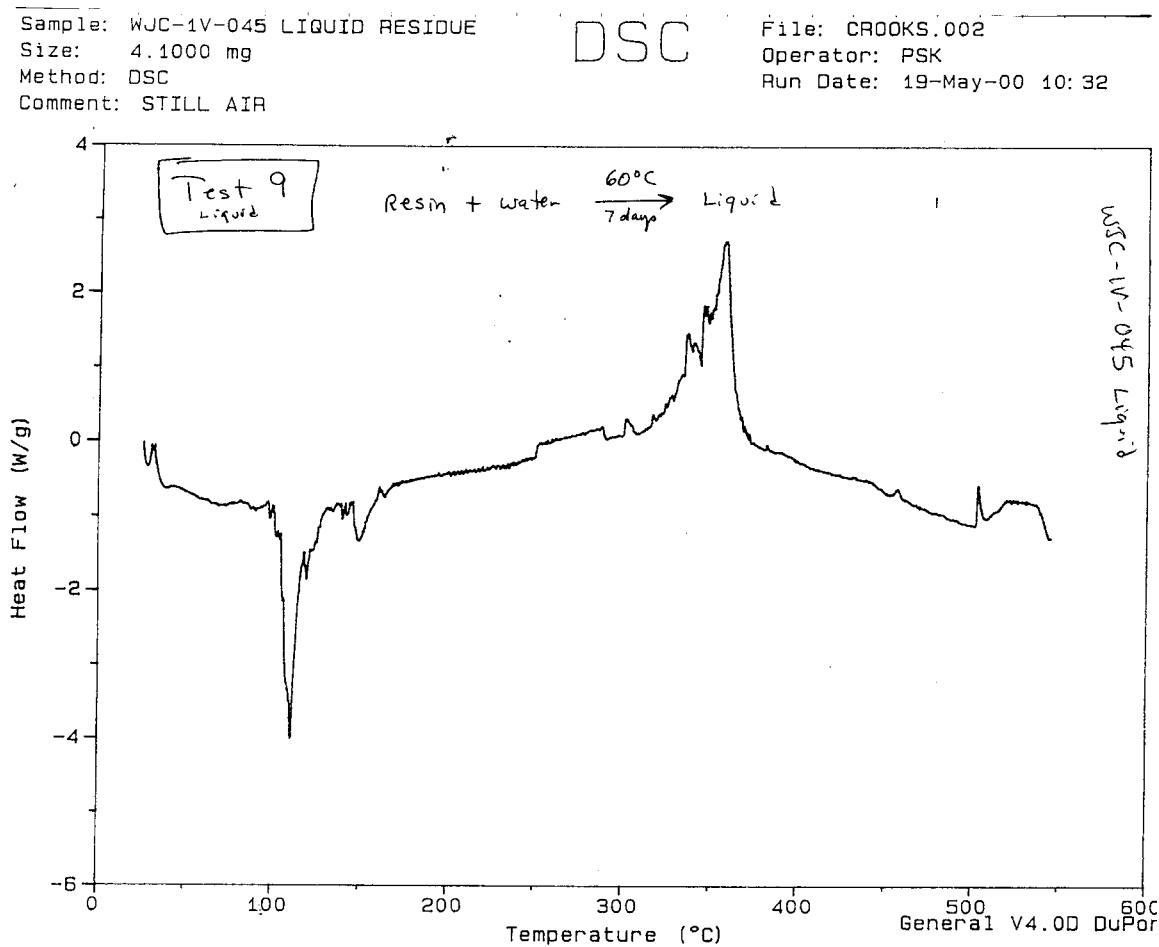


Figure 39. DSC of Test 9: Resin + H₂O at 60 °C for 7 days → Solid Residue



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