FINAL REPORT

Shelf Stable Epoxy Repair Adhesive

SERDP Project WP-1763

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14. ABSTRACT
The objective of this SERDP-funded research and development effort was to address the shelf-life shortcomings of currently available composite repair film adhesives. At military repair depots, currently used one-part adhesives have such a short shelf life that significant amounts expire and need to be discarded, contributing to a financial and environmentally costly waste stream. This effort aims to develop a one-part epoxy film adhesive that is stable at ambient temperatures for up to one year or at freezer temperatures for two years or longer.

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List of Acronyms

ACN	Acetonitrile
ASTM	American Society for Testing and Materials
BPA	Bisphenol A
DCDA	Dicyanamide
DETA	Diethylenetriamine
DMA	Dimethylamine
DOD	Department of Defense
DSC	Differential scanning calorimetry
ECH	Epichlorohydrin
FTIR	Fourier transform infrared
IC	Iota carrageenan
ID	Inner diameter
IST	Infoscitex Corporation
KC	Kappa carrageenan
MET	Minimal exposure time
NCO	Isocyanate
NS	Not soluble
PDDACL	Poly(dimethyldiallylammonium chloride)
PD	Partially decomposed
PS	Partially soluble
R&D	Research and development
S	Soluble
SAE	Society of Automotive Engineers
SERDP	Strategic Environmental Research and Development Program
SSA	Shelf Stable Adhesive
SWRI	Southwest Research Institute
TDU	2,4-toluene bis dimethyl urea

Keywords

Shelf-stable, epoxy, microencapsulated, repair adhesive

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1. Abstract

The objective of this SERDP-funded research and development effort was to address the shelf-life shortcomings of currently available composite repair film adhesives. At military repair depots, currently used one-part adhesives have such a short shelf life that significant amounts expire and need to be discarded, contributing to a financial and environmentally costly waste stream. This effort aims to develop a one-part epoxy film adhesive that is stable at ambient temperatures for up to one year or at freezer temperatures for two years or longer.

The technical program focused on enhancement of current epoxy resin-based film adhesives through modification of the accelerator package. This was accomplished through formulation of controlled release encapsulated accelerators into a one-part epoxy resin. Henkel Aerospace film adhesive product Hysol EA 9696 was chosen as the model epoxy resin system against which to develop accelerator modifications. A baseline was formulated using the primary components of the Hysol EA 9696 resin system matrix and fenuron accelerator. The experimental shelf-stable adhesive (SSA) was formulated in a manner identical to the baseline; the only differentiating factor was that the fenuron accelerator was encapsulated in a controlled release shell.

Two sets of tests were performed to establish the feasibility of encapsulating the accelerant in a one-part epoxy adhesive system. The first set of tests focused on evaluating the long-term shelf stability of the SSA adhesive. The second set of tests focused on assessing the mechanical performance of the SSA adhesive. The tests performed were based upon both standard AMS-A-25463 [1] as well as input from technical advisors. This standard provides all requisite guidance for the chemical and physical performance of film adhesives. The model epoxy resin system selected is classified as a Type I adhesive. Accordingly, performance metrics for the SSA film was tested based on Type I requirements. The long-term stability of each adhesive was assessed at a sustained elevated temperature (32°C/90°F). The SSA and baseline formulations were compared at each time interval using single lap shear testing (ASTM D1002) [10] of cured specimens. Each sample set consisted of at least nine specimens.

A matrix of five different mechanical tests was performed on the baseline and candidate SSAs under six test conditions. All of this testing was performed on samples that were prepared with new adhesive that was meticulously stored at 10°F. A summary of the test results are shown in Table 3. All operational mechanical tests were performed at third-party facilities.

The combination of the results from both the operational mechanical and shelf stability tests show that the one-part SSA has potential as a viable upgrade for the currently available one-part adhesive systems. The SSA has been demonstrated to retain at least 75% of its adhesive strength when stored at 90°F for a year. When compared to the baseline formulated with an unencapsulated accelerant, the stability of the SSA formulation offers a significant advantage. If stored in a freezer, it is anticipated that the SSA will be stable for well over two years. The data confirms that the capsules are not having a deleterious effect on the performance of the adhesive. In every test, the mechanical properties of the conditioned SSA samples were comparable or better than those of the baseline formulation. The test program has shown that the SSA formulation with encapsulated accelerator provides a significant shelf stability advantage over the baseline without sacrificing mechanical performance.

2. Objective

The objective of this SERDP funded research and development effort was to address the shortcomings of the currently available composite repair film adhesives, specifically with regards to the room temperature shelf life. Achieving this objective will contribute to SERDP's charter to facilitate the development of environmentally-beneficial technology, as it promises to aid in the reduction of hazardous waste associated with aircraft repair processes. Specifically, this effort aimed to develop a one-part epoxy film adhesive that is stable at ambient temperatures for up to one year and meets the mechanical performance requirements for aerospace adhesives.

3. Background

Conventional repair of composites used in military applications results in the generation of significant amounts of solid waste. In a SERDP funded research effort to quantify the environmental impact of composite repair operations, it was determined that millions of pounds of hazardous waste were generated. Hazardous waste disposal estimates for adhesives alone were estimated at 22 million pounds at a cost of over $100M [ARL-TR-2139, 1999]. Since the issuance of the report, the composite structures have proliferated in aerospace applications. Improvement of composite adhesive technologies is clearly needed to reduce environmental burdens and abatement costs. Repair facilities use one-part epoxy based adhesives extensively for metal to metal and honeycomb repairs. The costs associated with these processes are limiting the use of composite materials within the industry. One-part adhesive systems are primarily used within industry as they provide better control, reliability and take much less time to prepare than two-part systems. The major drawback, however, is that these systems slowly cure during storage and as a result have an extremely short shelf life (2-12 months), even when stored in freezers. Repair depots are required to employ extensive inventory and quality control to ensure that repair adhesives are not expired. Currently supplied film adhesives are only available in minimum quantities that are greater than the demands of the repair depot. Due to the combination of diverse quantity, poor shelf stability and large minimum quantity procurement requirements of military aerospace composite repair operations, much of the epoxy film adhesive is expiring before it can be used.

According to Ogden ALC at Hill AFB, more than 50% of procured film repair adhesive is ultimately discarded because it has expired or was exposed to ambient temperatures for too long. No improvements have been achieved to remedy this inefficiency. A one-part epoxy film adhesive system with improved shelf stability would greatly reduce the environmental impact of repair operations and reduce materiel procurement costs across the DoD. Applications of this adhesive technology could be utilized at DoD facilities such as Ogden and Warner-Robbins ALC and at commercial repair facilities such as would be used by OEM companies like Boeing.

Boeing uses one-part film adhesives for a wide variety of applications. Some manufacturing operations are more efficient, discarding less expired film. Commercial and military aircraft repair operations at Boeing experience very similar problems with film expiration and hazardous waste. At Ogden ALC over 720 pounds of EA 9696 film adhesive are purchased on contract every year. Individual small purchases that are less than $3,000 may be purchased via a government purchase card (GPC) and are not easily collected, so the total yearly purchase could be significantly higher. Robins ALC has purchased over 960 pounds of EA 9696 this year.

4. Materials and Methods

The technical program focused on enhancement of current epoxy resin-based film adhesives through modification of the accelerator package. This was accomplished through formulation of controlled release encapsulated accelerators into a one-part epoxy resin. Henkel Aerospace film adhesive product Hysol EA 9696 was chosen as the model epoxy resin system against which to develop accelerator modifications. A baseline was formulated using the primary components of the Hysol EA 9696 resin system matrix and accelerator (fenuron). The experimental SSA material was formulated in a manner identical to the baseline; however, the only differentiating factor was that the fenuron accelerator was encapsulated in a controlled release shell (see *Section 4.2.1* for details on this material). There were three primary thrusts to the research and development effort:

1. Adhesive Formulation
2. Film Processing
3. Performance Validation

The interrelation of these thrusts is shown schematically in **Figure 1**. Microencapsulant formulation was the foundation of adhesive development. Efforts during the first year of this project focused heavily on evaluating samples with various encapsulant shell materials produced via both complex coacervation and via in-situ polymerization. Activities during subsequent years focused on down-selecting the best candidate for validation activities, which included scaled-up batch production, shelf-stability testing, film production and mechanical testing. Film processing activities during the project involved laboratory-scale quantities. These films were used for the purpose of assessing the impact of candidate shell materials on resultant film mechanical properties. One key accomplishment was the demonstrated reproduction of scaled-up batches with consistent thermal and mechanical properties.

A preliminary requirements document was drafted to ensure the technical program yielded meaningful results that could be effectively compared to the current state-of-the-art and guiding military specifications. Requirements were based upon AMS-A-25463 [1], an SAE standard that replaced MIL-A-25463B, Notice 1 [2] to provide guidance on adhesive films used in aerospace structural construction. Key property requirements incorporated into the document included: cure time, shelf-life, peel strength, flatwise tensile strength, flexural strength and resistance to heat, fuel and humidity. In addition, non-functional requirements for the adhesive were defined to include environmental impact, cost and domestic supply. The final Requirements Document is provided in **Appendix 1**.

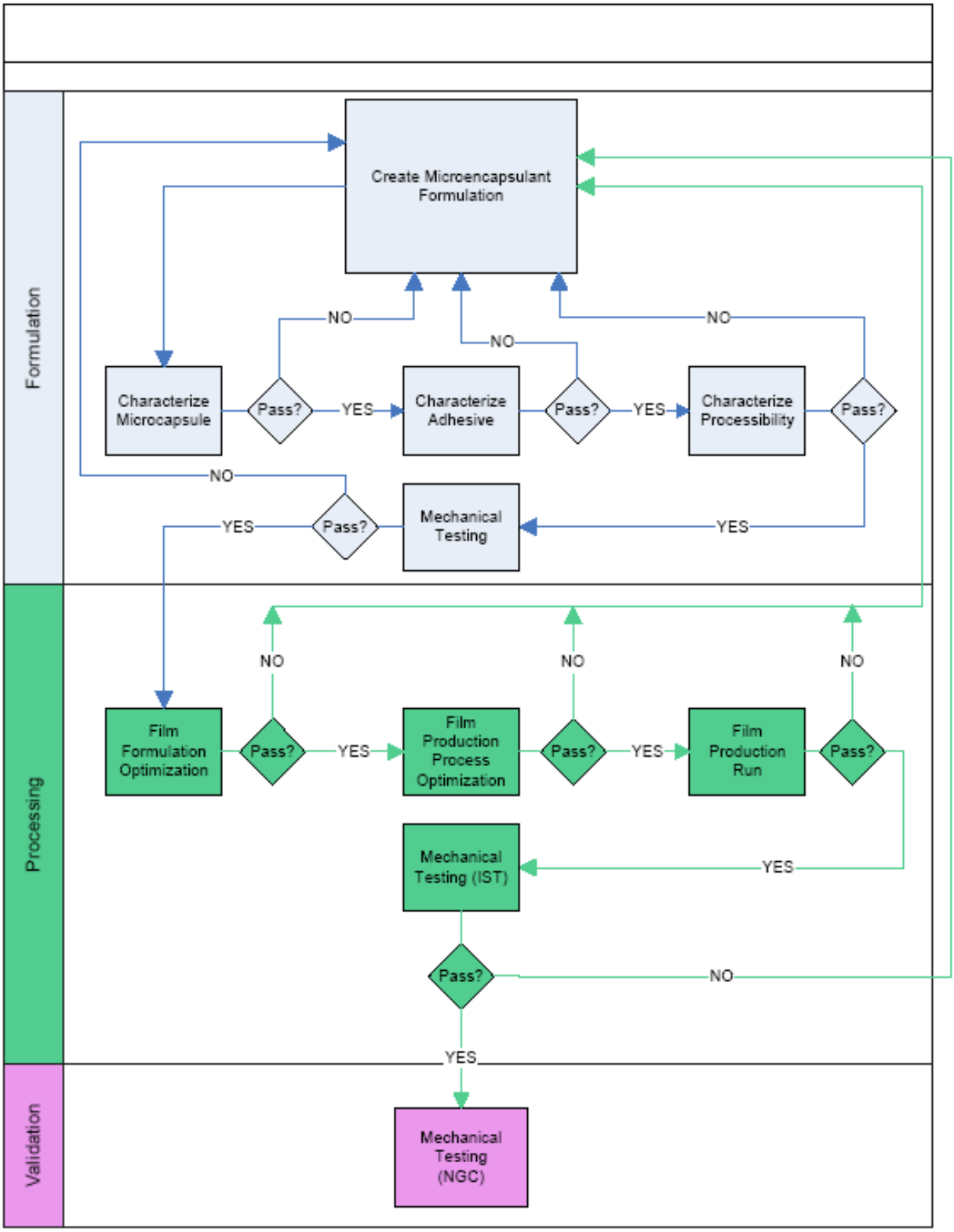


Figure 1. Flow diagram depicting interrelation of research and development threads

4.1 Model Adhesive System

A commercially available product was selected as the model epoxy resin system to ensure the technical program yielded meaningful results that could be effectively compared to the current state-of-the-art and guiding military specifications. Henkel's epoxy resin system, Hysol EA 9696 was selected as the model epoxy resin system. Hysol EA 9696 is a modified epoxy film adhesive designed for use at temperatures up to 250°F (121°C) in the joining of various structures, including metal sheet and honeycomb sandwich composite structures. Key features of this material include:

- Cure from 225°F to 265°F (107°C to 129°C)
- 12-month shelf-life at 0°F (-18°C)
- 60-day shelf-life at 77°F (25°C)
- 30-day shelf-life at 90°F (32°C)

Based on AMS-A-25463 [1], this material would be classified as a Type I adhesive, having an intended long-term use at -67°F (-55°C) to 180°F (82°C).

4.2 Adhesive Formulation

This project aimed to develop an epoxy resin film adhesive with improved shelf-stability. Focus was placed on a standard resin for the purposes of the development effort. Specifically, DOW D.E.R. 331 was selected as the primary epoxy resin. D.E.R. 331 is a liquid epoxy resin derived from the reaction of epichlorohydrin (ECH) and bisphenol A (BPA) (**Figure 2**). In addition to D.E.R. 331, IST identified DOW D.E.R. 661, a solid resin comparable to D.E.R. 331 in chemistry and performance, for use in controlling resin viscosity during film processing.

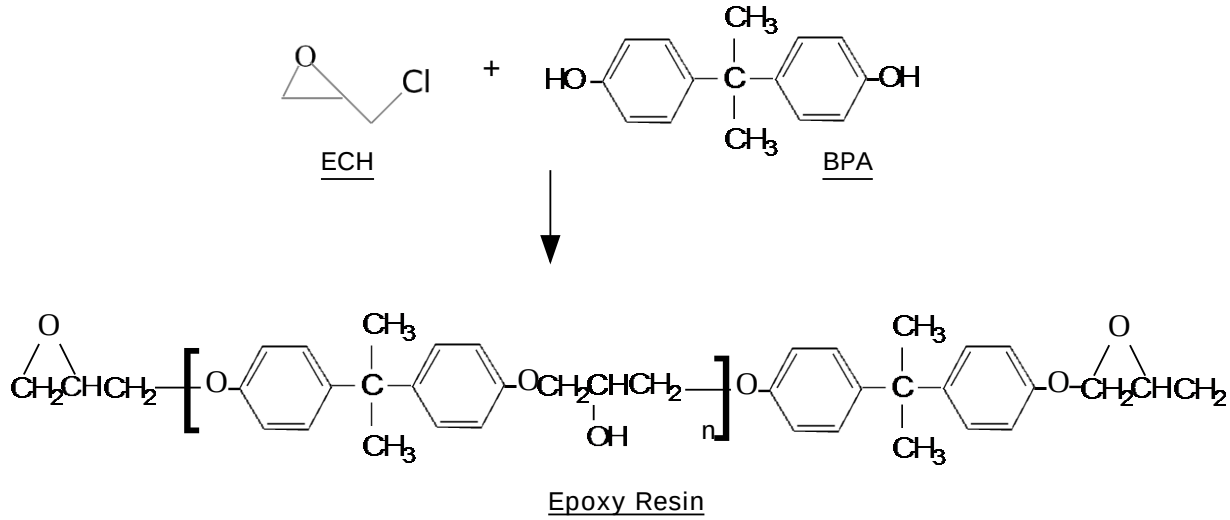
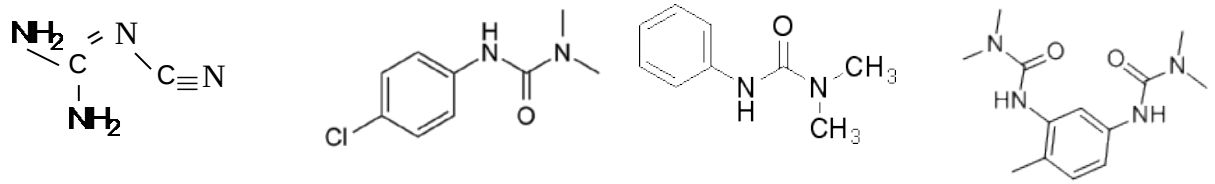


Figure 2. General reaction scheme for the production of BPA derived epoxy resins such as DER 331

The resin, catalyst, accelerator and other additives are machine mixed and stored (typically refrigerated or frozen) until use for standard one-part epoxy resin systems. In the interest of reducing the potential for confounding of results due to too many variables, it was decided that catalyst chemistry be held constant. Dicyanamide (DCDA) was selected for this function because it is an established curative. Its chemical structure is provided in **Figure 3**.



DCDA **Monuron** **Fenuron** **TDU**

Figure 3. Chemical structures of DCDA, monuron, fenuron, and TDU

Three urone accelerators were identified for use in the project. Monuron (N-(4-chlorophenyl)-N,N-dimethyl urea), a legacy accelerator, served as the baseline accelerator. Data pertaining to adhesives featuring this accelerator provided valuable data for the project, however, it will not be directly useful for downstream commercialization and transition since monuron has been phased out of new product development. Two additional accelerators were encapsulated: fenuron (phenyl dimethyl urea) and 2,4-toluene bis dimethyl urea (TDU). Chemical structures of these selected accelerators can be seen in **Figure 3.** After initial microencapsulation studies with monuron, described in the next section, IST concentrated the rest of the development on fenuron containing formulations. Fenuron is a de-chlorinated version of monuron, thus representing a safer product. According to manufacturers, it has a higher activity than monuron. TDU was also recommended for use by Henkel based on its anticipated high activity.

4.2.1 Microencapsulation

The materials and processes selected to microencapsulate the accelerant played a critical role in the efficacy of the encapsulation approach. During previous efforts, candidate encapsulant materials were evaluated in a system that used monuron as the accelerator. To make use of this benchmark performance data on the current project, IST screened encapsulant materials using monuron as well. This approach was deemed a viable path because monuron is chemically similar to fenuron.

Encapsulation efforts focused on complex coacervation (performed by Thies Technologies) and in-situ polymerization (performed by Infoscitex). Complex coacervation efforts during the first year of the project focused primarily on using mixtures of kappa carrageenan (KC) or iota carrageenan (IC) with poly (dimethyldiallylammonium chloride) (PDDACL) while efforts during subsequent years focused on utilizing gelatin. KC and IC are polysaccharides extracted from seaweeds that form gels at room temperature. They were chosen to provide a more thermally stable barrier to premature migration or leakage of accelerant into the epoxy resin [3]. Monuron was successfully encapsulated at concentrations greater than 50 wt% within four

formulations chosen: KC-PDDACL, IC-PDDACL, gelatin-KC-PDDACL and gelatin-gum arabic. The capsules were 10-25 µm in size. Analysis via differential scanning calorimetry (DSC) showed that the monuron within the capsules behaved very similarly to neat monuron, as evidenced by a sharp melting peak at its melting temperature of 350°F (178°C). The weight fraction of the microcapsules corresponding to monuron can be calculated by assuming that the enthalpy of melting (ΔH) is proportional to the amount of accelerator.

4.2.1.1 Prior Work Background

Microencapsulation is used in a wide variety of applications to isolate materials from their surroundings. There are a multitude of techniques that have been developed to encapsulate solids, liquids and gases. Prior research has attempted to develop one-part, shelf-stable epoxy resin systems using microencapsulation of accelerators or latent curing agents. Southwest Research Institute (SWRI) developed a monuron encapsulate using a paraffinic wax shell material. This microencapsulated material was intended for use in an epoxy film adhesive [4]. The product suffered from two shortcomings:

1. Diffusion of epoxy resin through the shell limited the shelf-life – as storage temperature increased, the rate of reaction increased and thus decreased the shelf-life of the adhesive.

2. Monuron was released from the microcapsule by melting the wax encapsulant – the wax became a contaminant in the adhesive and tended to migrate to the adhesive/substrate bond line thus adversely affecting adhesion strength.

Previous experience with film adhesive research [5, 6] indicated that the microcapsule shell must be capable of withstanding brief high temperature excursions (up to 250°F/121°C) for almost one minute to be processed into a hot-melt product on prototype film adhesive manufacturing lines. The microcapsule shell must have outstanding barrier properties and must completely cover the surface of the active material. Curing reactions will occur if as little as 1% of the accelerant escapes from the microcapsule. A highly cross-linked polymer is best for this application. It should be noted that these materials will not be amenable to release using a simple thermal trigger, such as the curing heat cycle itself. The microcapsule must be small enough to reside within an adhesive film during the repair process and the encapsulant material that remains within the resin after curing must not adversely affect the mechanical properties of the finished composite. Work from previous efforts found that particles with sizes of 20-50 µm, loaded into test paste adhesive specimens at a loading of 6 wt%, did not adversely impact the tensile lap shear and floating roller peel strength properties of the film adhesive [5, 6]. Since these particles are very small, they can be easily opened using a brief and temporary thermal spike to rupture the capsules and release the accelerant before the curing reaction begins.

4.2.1.2 In-Situ Polymerization

The objective of this approach was to encapsulate accelerant particles in a controlled, reproducible manner in a uniform epoxy polymer shell compatible with the bulk of adhesive. Monuron heated in absence of DCDA can contain up to 0.8 % DMA [7]. It was hypothesized that this small amount of DMA could be used for in-situ formation of a protective capsule on the

surface of urone crystals. As shown in **Figure 4**, upon moderate heating of urone, DMA forms within a urone crystal and migrates to it surface via molecular diffusion. On the urone crystal surface, DMA reacts with an epoxy monomer, such as epichlorohydrin (ECH, 1-chloro-2,3-epoxypropane) or an epoxy resin. This triggers the formation and growth of epoxy polymer chains on the urone crystal surface. The in-situ polymerization process can be stopped via:

- Quenching/termination of the reaction (addition of reactive amine, such as diethylenetriamine (DETA));
- Exhaustion of available polymer precursor (monomer, resin) and quenching;
- Removal of the polymer precursor (evaporation or draining) and quenching.

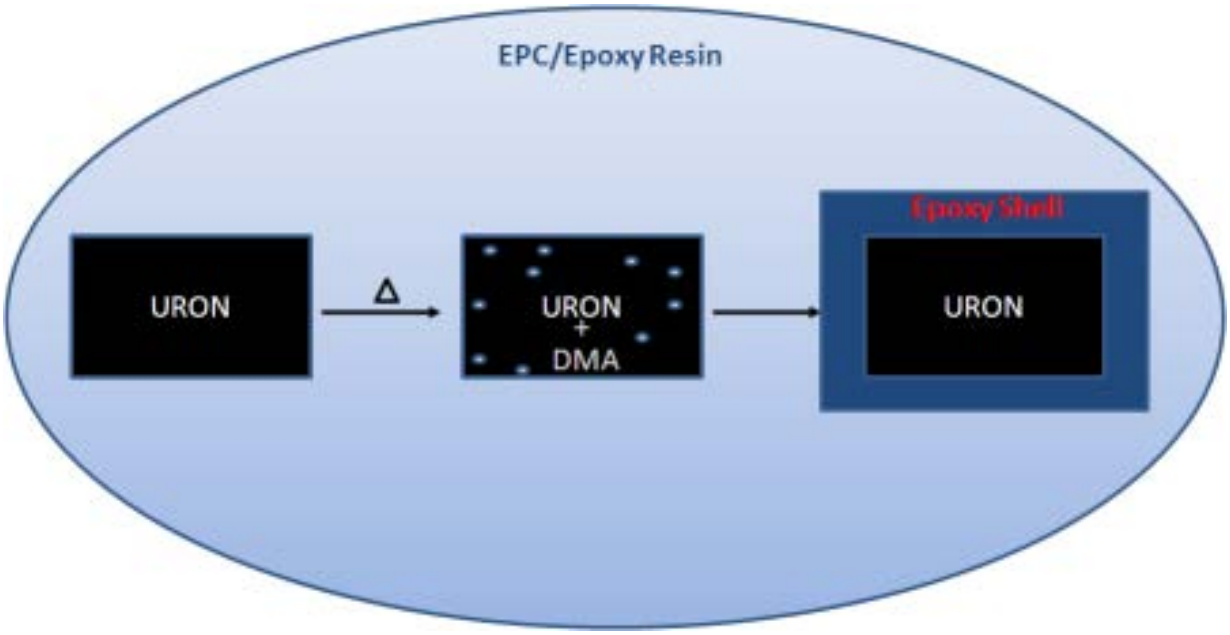


Figure 4. Urone Encapsulation by In-situ Polymerization

To verify the proposed approach, three experiments were performed. First, determine the optimal processing parameters for the in-situ generation of DMA. Second, determine the feasibility of in-situ polymerization of an epoxy shell on the urone crystal surface. And finally, determine the feasibility of generating encapsulated urone within the body of an adhesive formulation.

4.2.1.2.1 Investigation of Processing Parameters for In-Situ Generation of DMA

The in-situ method required the generation of a sufficient amount of DMA while preserving the bulk of the urone from thermal decomposition. Three urones were investigated during this experiment: monuron, fenuron and TDU. It was determined that the raw urone materials contained 5 to 10 wt% of insoluble organic matter, potentially proprietary stabilizers.

Based on the literature available regarding monuron thermal decomposition, the optimal reaction temperature should be 122°F to 158°F (50°C to 70°C), with a minimal exposure time (MET) of less than one hour. Due to the similarity of each of the urone compounds, it was anticipated that

these reaction parameters would be comparable for all three of the urones investigated. For each of the reaction temperatures studied, urone samples weighing approximately 0.1 g were produced in triplicate at three different exposure times (30 minutes, 45 minutes, and 60 minutes). Preliminary analysis of the neat urone materials showed that they each contained residues that were insoluble in organic solvents. Additionally, Fourier transfer infrared spectroscopy (FTIR) analysis showed that the neat starting urones contained some small amounts of isocyanates. Therefore, it was necessary to treat the powders. Small samples (10-12 g) of each urone powder (monuron, fenuron and TDU) were weighed into borosilicate capped vials. The vials were tightly closed and incubated in a thermo-controlled convection oven for desired periods of time and under preset temperatures. Upon incubation, the vials were removed from the oven and immediately cooled to room temperature. Approximately 1.0 mL of acetonitrile (ACN) was added to each vial using a glass syringe. The bottles were then re-capped and gently shaken to dissolve the urones and isocyanates that were present in the neat material. The resultant suspensions were centrifuged at 21,000g, and their clear supernatants were collected. The decomposition of each urone was verified using FTIR spectroscopy by observing the stoichiometric decomposition of the ureas into DMA and nonvolatile aromatic isocyanate (NCO).

4.2.1.2.2 In-Situ Polymerization of Epoxy Shell on Urone Crystal Surface

Upon defining the process parameters, it was necessary to determine the feasibility of in-situ polymerization of an epoxy shell on the urone crystal surface. The in-situ polymerization reaction was performed in a 300 mL, temperature-controlled round-bottom reaction flask equipped with stirrer and gas/vacuum inlets and outlets. Urone (15g to 30g) was added to a flask containing 270 g to 285 g of ECH under a nitrogen blanket. The contents were homogenously mixed at room temperature. The reaction was then started by slowly increasing the temperature from room temperature to the targeted reaction temperature. The reaction was exothermic; therefore, an immersion cooler was used to keep the reaction vessel at the desired temperature. Approximately 0.2 g of the reaction mixture was withdrawn from the reaction flask periodically and dispensed into 10 mL glass jars and dried under vacuum in a desiccator. Samples were drawn at three (3) time intervals: MET (as determined above), 1.5 x MET and 2.0 x MET. Dried capsules were quenched by the addition of 0.5 mL of DETA. The average size of individual encapsulated particles and shell thickness was determined using optical microscopy. The urone content in the collected samples was determined using DSC analysis.

Once the desired capsule properties were determined, three batches of the encapsulated urone were generated under conditions determined above. More specifically, the reaction was scaled-up to produce 50 g batches of the encapsulated urone. The reaction was started by mixing 450.0 g of ECH with 50.0 g of the urone. The reaction was completed by maintaining the reaction mixture at the desired temperature and time. At the end of the reaction period, ECH was removed from the reaction mixture under vacuum and collected. Dried capsules were quenched by the addition of 100 mL of DETA. The unreacted DETA was collected under vacuum. The urone content in the collected samples was determined using DSC.

4.2.1.2.3 In-Situ Encapsulation of Urones with Adhesive Formulation

Next, efforts were focused on determining the feasibility of generating encapsulated urone within the body of an adhesive formulation. As was the case for the in-situ polymerization of the epoxy monomer explained above, the driving force for this reaction was the formation of DMA from the urone particles. The shell formation process was initiated by heating the resin/urone mixture to the optimal release temperature of the DMA as previously determined. The shell formation process was terminated by directly adding the quencher into the mixture. Since the propagating epoxide polymer chain forming on the surface of the urone crystals contained the activated epoxide terminus, the quencher reacted with this epoxide group, while the bulk of the resin remained unreacted and thus available for future curing bonding applications. It was necessary to precisely control the amount of quencher added because it would have reacted with and consumed a large portion of the bulk resin if there was excess. Therefore, the optimal amount of reagents and reaction conditions were investigated. To do this, the in-situ resin polymerization reaction was carried out at room temperature in a temperature controlled Pyrex reaction round-bottom flask equipped with stirrer and gas/vacuum inlets and outlets.

Urone (3 g) was added to a flask containing 100 g of the epoxy resin. The contents were homogenously mixed at room temperature. The reaction was then started by slowly increasing the temperature from room temperature to the release temperature of DMA. The reaction was exothermic; therefore, an immersion cooler was used to keep the reaction vessel at the desired temperature. Approximately 30 g of the reaction mixture was withdrawn from the reaction flask periodically and dispensed into glass jars and dried under vacuum in a desiccator. Samples were drawn at three time intervals: MET (as determined above), 1.5 x MET and 2.0 x MET. Dried capsules were quenched by the addition of 20% (molar) excess of DETA. The required amount of DCDA was then added to each of the resin/capsule mixtures (i.e. 5 g of DCDA to 100 g of resin/capsule mixture). These samples were thoroughly mixed manually, degassed and tested for urone loading using DSC analysis.

4.2.1.3 Complex Coacervation Encapsulation

Complex coacervation was conceived in the 1930s by chemists at the National Cash Register Corporation as a means to make microcapsules for carbonless paper. A schematic of a typical complex coacervation process, using gelatin and gum arabic, can be seen in **Figure 5**. The method used during this project to encapsulate monuron in cross-linked gelatin is similar to one described by Fogle [8]. In this process, monuron is first dispersed into an aqueous solution of gelatin using mechanical agitation. For this emulsification process to be successful, the core material must be immiscible in the aqueous phase. Since monuron is insoluble in water, this approach is appropriate. A coacervating agent, such as sodium metahexaphosphate, is then added to this emulsion. After mixing, dilute acetic acid is added to adjust the pH. Addition of the acetic acid results in the phase separation of the emulsion into two immiscible liquid phases. One phase, the coacervate, has relatively high concentrations of both the accelerant and encapsulant. The second phase, the supernatant, has low concentrations. The compositions of these two phases and the pH at which phase separation occurs are governed by factors such as the ionic strength of the initial solutions, temperature, and the molecular weight of the core and shell materials. The coacervate preferentially adsorbs onto the surface of the dispersed monuron,

forming a coating that fully encapsulates the particles. The mixture is cooled and an agent such as gluteraldehyde is then added to cross-link the gelatin coating. These irregularly shaped coated particles can then be separated from the rest of the mixture. A second cross-linking step can then be performed using tannic acid. This produces a relatively hard, smooth surface to the gelatin coating and prevents agglomeration of the microcapsules.

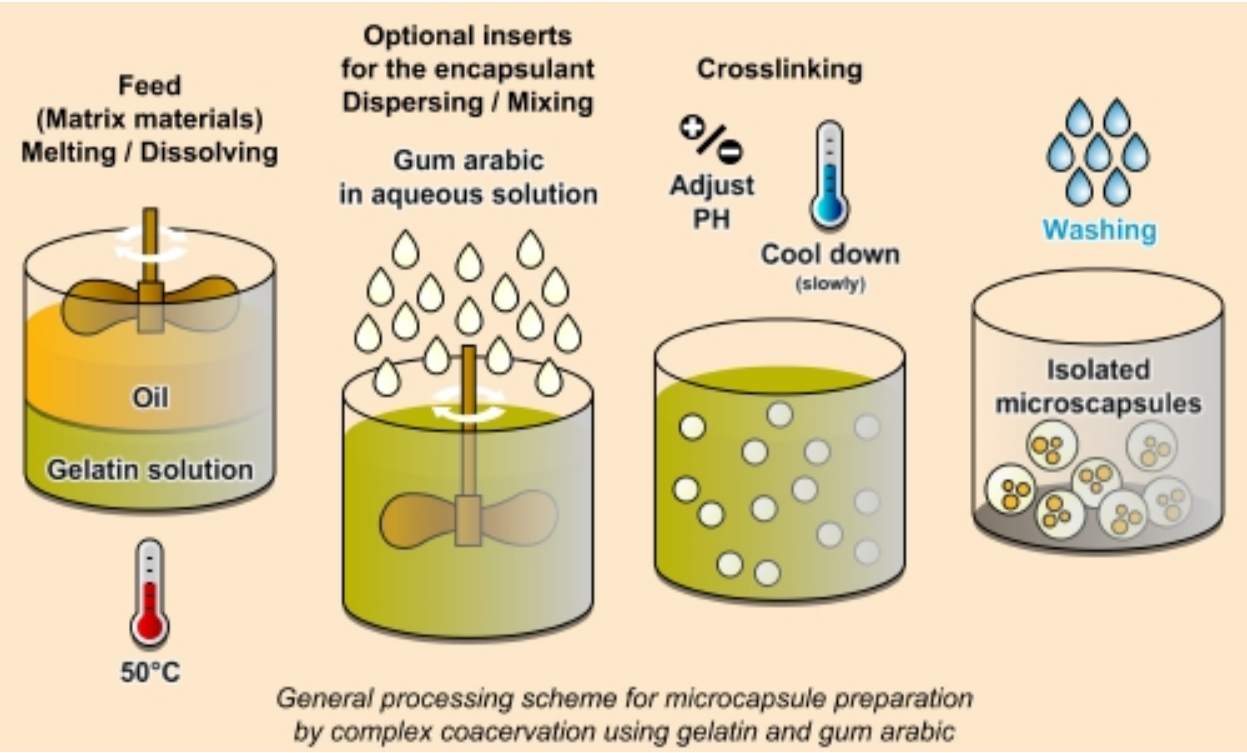


Figure 5. Schematic of a typical microencapsulation process using the complex coacervation method [9]

Complex coacervation is influenced by many highly interrelated factors. For example, the phase separation process affects the composition of the encapsulant, which in turn affects the ability of the encapsulant to wet the core phase, the barrier properties of the encapsulant and the release characteristics of the microcapsule. Due to these multifaceted interactions, it is very difficult to quantify the influence of the process parameters on the coacervation process, despite extensive research. Many existing industrial processes that rely on complex coacervation have been developed based on experience and qualitative observations of the process. **Figure 6** illustrates steps involved in microencapsulation via the complex coacervation process used in this project.

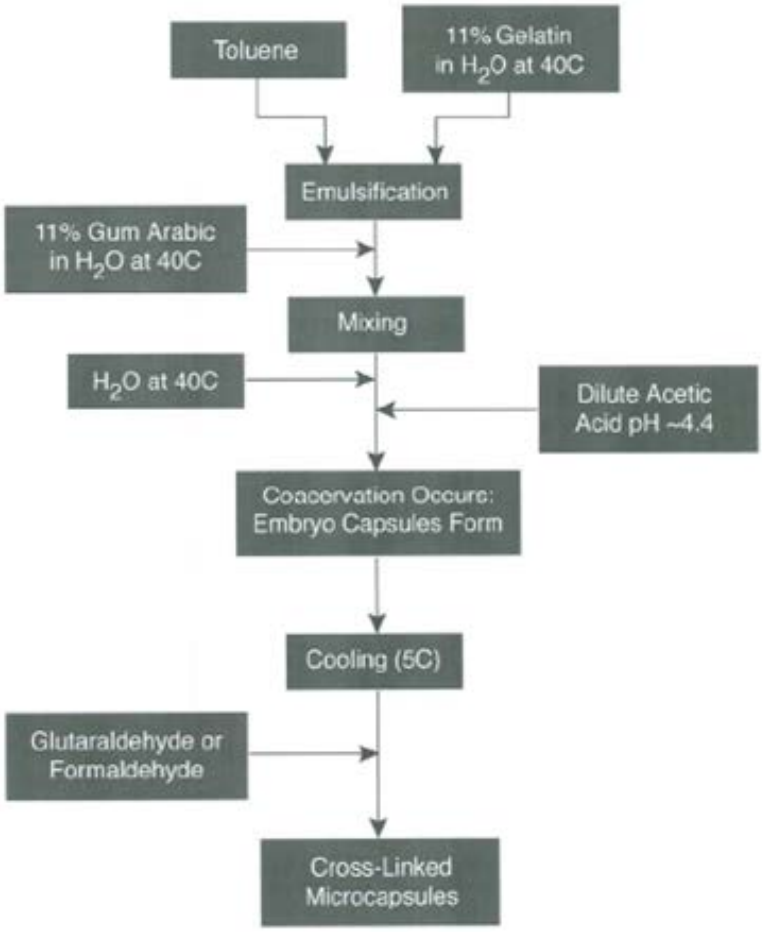


Figure 6. Complex coacervation process developed by Thies Technologies for microencapsulation of the curing accelerant in IST's shelf-stable epoxy resin adhesive

4.2.2 Adhesive Formulation

The model epoxy resin formulation is a three-part system that consists of an epoxy resin, a curative and an accelerator. Pastes were formulated according to the recipe in **Table 1**.

Table 1. Baseline epoxy resin system used for viscosity trials.

Adhesive component	Mass ratio
DER 331 liquid epoxy resin	100
Omnicure DDA5 (DCDA)	5
Accelerant	3
Fumed silica	3

A standard mixing procedure was established to ensure consistent sample production. The standard mixing procedure was developed using raw materials rather than encapsulated material to expedite the process and reduce costly waste. A small-scale (15-25 g) mixing procedure was developed first. This process was used to create paste adhesive samples for DSC analysis and

viscosity analysis. A laboratory-scale (50-100 g) mixing process was also developed. This process was used to create material for film coating.

4.2.2.1 Small-Scale Mixing

Hand mixing was performed on small-scale mixtures. The material from these small batches (15-25 g) was used for DSC analysis, long-term shelf stability testing, and viscosity analysis. These samples were made using the baseline mixing ratio of epoxy (D.E.R. 331): curative (DCDA, DDA5, Omnicure): accelerator (monuron, Sigma Aldrich): fumed silica (Cabot Corporation). The epoxy was measured out into a small glass jar and heated to 104°F (40°C) to reduce its viscosity so as to more easily mix solid particles. Each powder was added to the epoxy and then mixed via the Painter's method using a glass stir rod. Small amounts of the mixture were used for DSC analysis, as described in *Section 4.4.1*. The remainder of the mixture was poured into cylinders for viscosity testing and allowed to de-gas overnight in a convection oven at 104°F (40°C). After approximately 10 hours, the samples were removed from the oven and their viscosity was measured, as described in *Section 4.4.2*, to determine the time to cure of each mixture.

4.2.2.2 Laboratory-Scale Mixing

A laboratory-scale mixing process was devised and demonstrated to yield a homogeneous dispersion of solid particles while minimizing the amount of entrained air in the fully formulated adhesive system. Preliminary mixing trials were performed using a 100:5:3 ratio of epoxy (D.E.R. 331): curative (DCDA, DDA5, Omnicure): accelerator (monuron, Sigma Aldrich). A water bath was used to maintain the samples at 35°C during mixing. The efficacy of three types of impeller blades (two blade dispersion impeller, six blade Rushton impeller and three blade left-hand axial impeller) as seen in **Figure 7**, was investigated. As described in the following subsections, several trials were performed to determine appropriate mixing parameters, such as impeller type, impeller speed and mixing duration.

Figure 7. Blades used for preliminary mixing trials: A) two blade dispersion impeller, B) six blade Rushton impeller, and C) three blade left-hand axial impeller

After impeller selection, the next step was to create samples of the fully formulated mixture. Fumed silica was added to the mixture to help to keep the accelerator from falling out of solution. To develop a standardized mixing procedure, samples were produced using a 100:5:3:3 ratio of epoxy (D.E.R. 331 and D.E.R. 661): curative (DCDA, DDA5, Omnicure): accelerator (monuron, Sigma Aldrich): fumed silica (Cabot Corporation). Due to the increased viscosity of

this mixture, it was necessary to work at an elevated temperature (194°F /90°C). 120g of D.E.R. 331 and 180 g of D.E.R. 661 were added to a 600 mL beaker, creating a 40:60 mixture of 331:661. The mixture was allowed to heat in a convection oven at 194°F (90°C) for 2 hours. Meanwhile, the water bath in the mixing apparatus was allowed to reach 194°F (90°C) and the solid components (DCDA, monuron and fumed silica) were weighed and set aside. The beaker with the epoxy mixture was then removed from the convection oven and suspended in the water bath, allowing the water to rise above the level of the mixture within the beaker, thus ensuring that the mixture is blanketed in heat while mixing. The epoxy resin was mixed at a speed of 250 RPM for 20 minutes. The speed was then increased to 1000 RPM and the sample was allowed to mix for an additional 30 minutes. The speed was then decreased and the fumed silica was added using a plastic funnel. Once the fumed silica was completely incorporated, the speed was increased to 2000 RPM and the sample was allowed to mix for an additional 10 minutes. The speed was once again decreased to 1000 RPM and the DCDA as well as the monuron was added. After they were incorporated, the speed was increased to 1500 RPM and mixed for 5 minutes. Once mixing was complete, the mixture was poured into a Teflon mold and set aside until it was needed for film processing.

4.2.2.2.1 Two Blade Dispersion Impeller

The first impeller investigated was a two-blade dispersion impeller (as seen in **Figure 7A**). This type of impeller is typically used when mixing paint or dispersions. The water bath was filled and allowed to reach 95°F (35°C). A 600 mL beaker was filled with approximately 500 g of epoxy resin. The beaker was then suspended in the water bath, allowing the water to rise above the level of epoxy in the beaker, thus ensuring that the mixture was blanketed in heat while mixing. The impeller was then lowered into the epoxy such that the head was approximately 1 inch from the bottom of the beaker. The speed of the impeller was gradually increased until a well-developed vortex was observed. This speed was determined to be 2000 RPM. After approximately 30 minutes, the impeller speed was slowed to 1000 RPM and the pre-weighed curative and accelerator agents were slowly added using a plastic funnel to ensure that all of the power would transfer directly into the epoxy resin. Once the curative was added, the speed of the impeller was increased to 2000 RPM and allowed to mix for 30 minutes. After mixing, the impeller was slowly stopped and removed from the beaker. The mixture was analyzed for homogeneity. Upon visual inspection, large particle agglomerates were evident; therefore the selected mixing parameters did not yield a well-blended mixture. To overcome this, a high shear impeller was investigated.

4.2.2.2.2 Six Blade Rushton Impeller

Rushton impellers, as seen in **Figure 7B,** are commonly used for applications that require intense mixing. Observations made during the first mixing trial indicated that an increase in impeller speed from 2000 RPM to 2500 RPM was required to maintain the desired vortex. Otherwise, the mixing process was identical to that described above. Visual inspection of the samples prepared using the Rushton impeller indicated that fewer agglomerates were present, however, the mixing was not as homogenous as desired. Additionally, it was hypothesized that the high shear imparted by the impeller on the sample was causing the capsules to burst during mixing. Therefore, an axial impeller was investigated.

4.2.2.2.3 Three Blade Left-Hand Axial Impeller

These impellers, as seen in **Figure 7C,** are used to impose bulk motion during mixing. It was hypothesized that an axial impeller would yield a homogenous mixture and not rupture any of the capsules. Several samples were prepared as described previously in *Section 4.2.2.2*. Visual inspection of the samples prepared using the axial impeller showed the best mixing out of the three blades that were examined.

4.3 Film Processing

Adhesive formulations prepared as described in previous sections were used to create adhesive films. Films typically have fiberglass scrim with appropriate sizing and are 0.005-0.080" in thickness. The adhesive films must cure according to the prescribed profile to reach full strength. One of the primary reasons to make adhesive films during this effort was to determine if the adhesive strength of the films was degraded in any way due to the inclusion of encapsulated catalyst. Thus it was important to create very consistent films to minimize variability in the strength. Complete scrim wetting and impregnation was imperative. To achieve the adhesive film objectives stated above, IST devised a custom hot-melt film processing setup and method. The following sections describe the setup and method and are followed by a description of established inspection methods.

4.3.1 Lab-Scale Film Line Setup

A film casting setup was constructed with the purpose of producing consistent small batches of composite adhesives films. The primary film drawing components and heaters were available as a pre-packaged system from ChemInstruments. The hot melt coater system consisted of a feed spool holder, heated polymer trough, high-tolerance milled steel rollers and a heater/controller unit. As delivered, the system was designed expressly for manual, batch operation without a feed spool for release ply. It was desired to have mechanized film rolling capabilities to improve film consistency. As a result several modifications and additional hardware components were integrated:

- Two tensioned 1-¾" ID feed rolls: necessary to integrate a release ply with the adhesive film for ease of handling
- Variable speed motor and gearbox: provided mechanized consistent rolling at slow speeds
- Conveyer line stand: maintained requisite -10° pitch of film drawn from rollers

A schematic diagram of the setup can be seen in **Figure 8**.

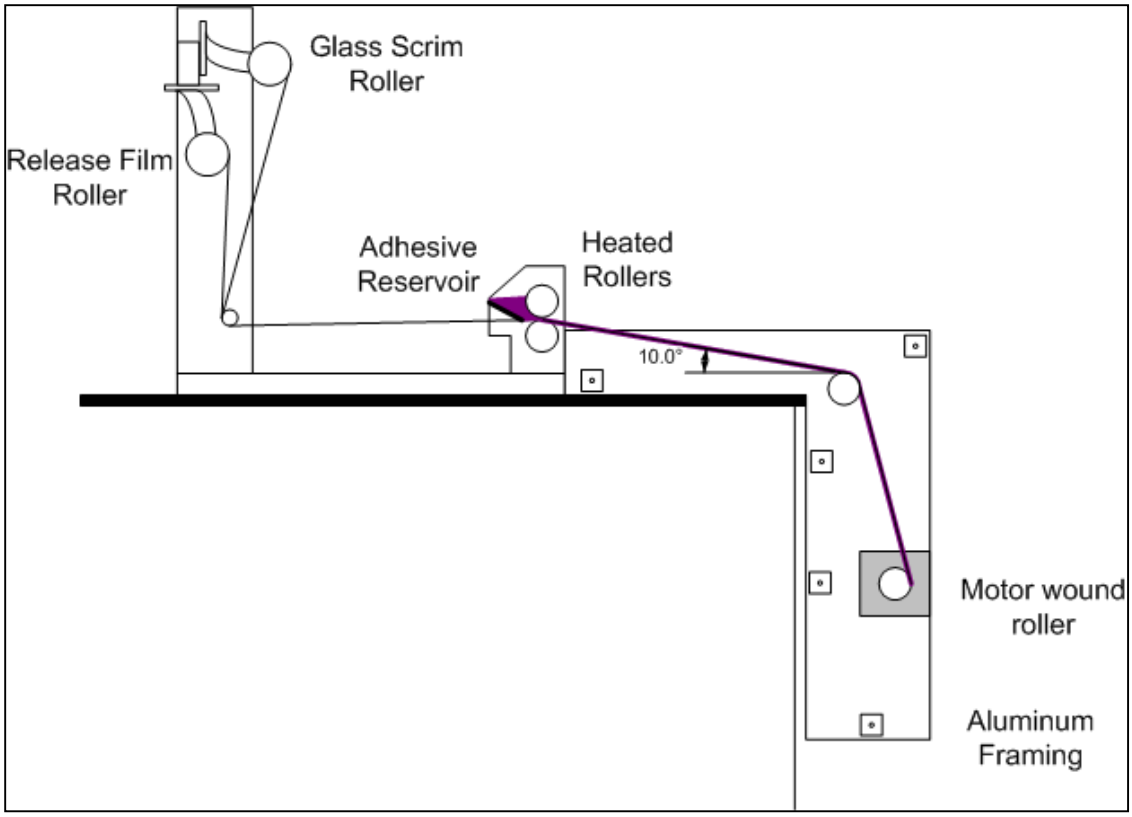


Figure 8. Schematic of the hot melt adhesive processing setup

4.3.1.1 Method

To keep the film products consistent, established processing methods were followed for each run. Run preparation, processing and clean-up methods are listed below.

4.3.1.1.1 Run Preparation

The rollers and trough were cleaned using a single wipe of an acetone-soaked cloth to ensure any residual material from previous runs was removed. Once clean, the rollers, trough, and top roller tensioner were inserted. When inserting the top roller tensioner, special attention was paid to ensure they were level with mounting brackets and that standoff screws were in place. In most instances, Teflon flow constriction blocks were required.

Next, the rolls of scrim (Type E glass scrim with a weight 4oz/sq. yd. from Fibreglast) and Teflon release ply were mounted. Cutting tape (from Fibreglast) used to make the roll was left in place. The scrim roll was mounted above the release layer. The roll of scrim was about 8 inches wide, two inches less wide than the release film. The 1 inch excess release film on either side of the scrim kept the melter clean and allowed the Teflon blocks to keep the melted resin on the part in process. **Figure 9A** shows the rollers mounted on the setup.

The scrim and release film were pulled past the directional roller and between the two hot melt rollers. Approximately four (4) inches of extra material was allowed to hang out past the rollers.

This material was taped to a wooden dowel for pulling film to the mechanical winder. It was important to have the scrim layer, as opposed to the Teflon, in tension when pulling the film, otherwise wrinkling and bunching may occur. Two (2) metal shims were inserted over the scrim layer and under the top heated roller to space the rollers and specify the desired film thickness. It was important to make sure there are no bunches or folds in the scrim under the shims and to space the shims on the far ends of the scrim cloth. Typical shim thicknesses were 8-20 mils. With the shims still in place, the four roller set screws and two tensioner bar set screws were tightened. The heating elements were inserted into the trough and two rollers to allow the system to preheat. The heaters were set to 195°F (91°C). The heaters were typically allowed to heat the system for approximately 15 minutes before the addition of the adhesive formulation. The melter and stand were aligned with the roller frame to make sure the film will wind in the center of the mechanized roller at the bottom of the setup. A picture of the aligned system is provided **Figure 9B.**

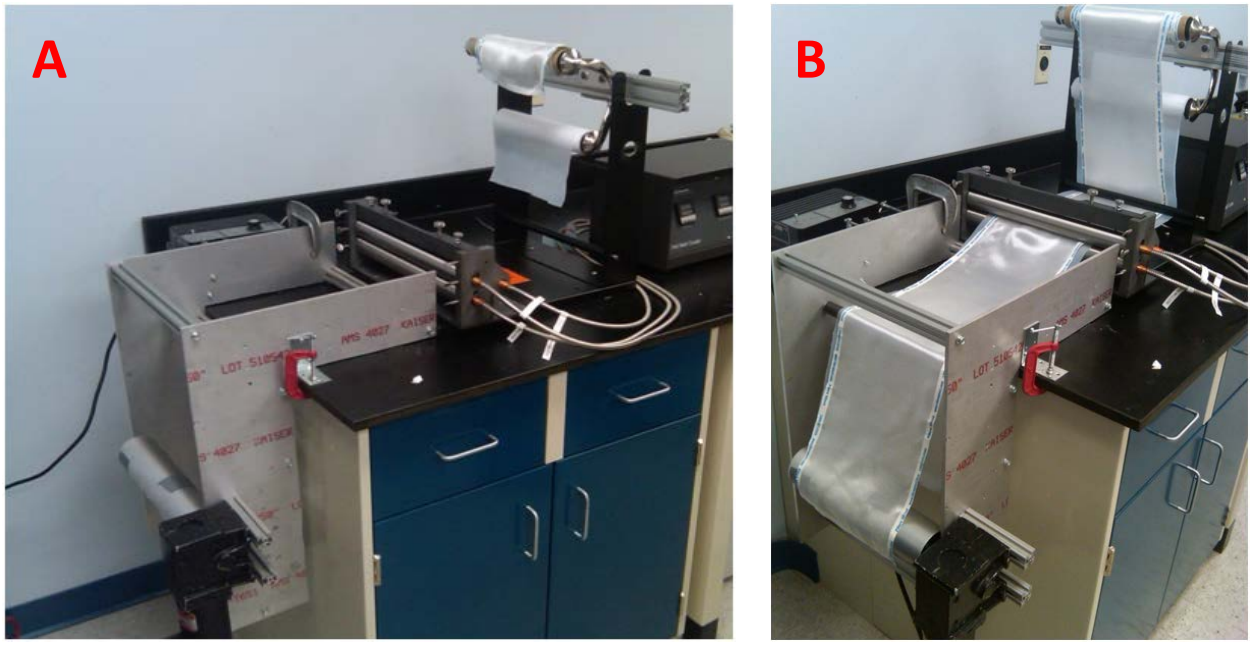


Figure 9. A) Hot melt processing set-up with scrim and release film feed rolls, B) Processing set-up with aligned scrim and release film

4.3.1.1.2 Processing

The epoxy resin in the trough was brought to the processing temperature (in most cases 195°F/91°C). Effort was made to start processing runs as soon as the batch formulation was at temperature so as to reduce additional thermal history witnessed by the capsules when heating from ambient to processing temperature. Depending on film thickness, 100-200 g of the adhesive paste was melted per film run. A picture of fully liquefied and heated resin in the melting trough can be seen in **Figure 10.**

Once the resin was ready to be applied, the wooden dowel taped to the leading edge of the scrim was drawn very slowly in such a way to maintain a less than a -10° angle out of the heated rollers. The dowel was brought over the conveyer roller and attached to the motorized roller.

Once attached to the roller, the motor was set to 1-2 RPM. The slow speed was necessary to ensure that the film resin cools by the time it reached the rollers and to facilitate better impregnation of the scrim fiber during coating. To clean the apparatus, parts were wiped down with acetone or soaked overnight if particularly gummed up with resin. Cleaning with acetone immediately after the last run prevented the resin from curing on the hardware.

Figure 10. Hot melt trough with liquefied resin in reservoir (indicated by arrow) during processing

4.3.1.1.3 Inspection

Film quality was determined by visual inspection, which targeted the following defects:

- Large scrim weave gaps
- Poor fiber wetting indicated by highly visible scrim fiber
- Full impregnation of the scrim on both sides
- Voids or particulate contamination

Microscopy was used as appropriate to determine the degree of wetting of the scrim fibers. A sample with good impregnation and moderate fiber wetting can be seen in **Figure 11**.

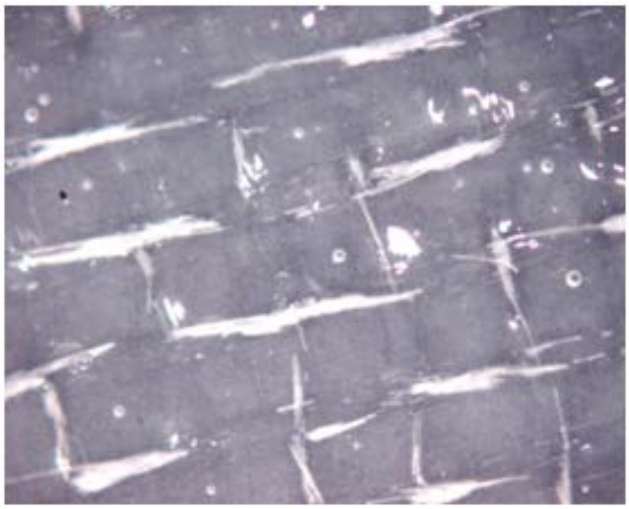


Figure 11. Micrograph of film prepreg with visible voids, good impregnation and only moderate fiber wetting. Good fiber wetting is exemplified by low-to-no visibility of the scrim fiber.

4.3.2 Development of Single Lap Joint Cure Protocol

Initial samples were cured in accordance to manufacturer's recommendations; however, single-lap-joint test results were not as expected. Therefore, an experimental test matrix was devised to determine the optimal cure conditions for the adhesive films that would ensure that samples made from both the model formulation and the SSA formulation were properly cured prior to testing while not degrading their mechanical performance. Examining uncured or partially cured samples could lead to faulty results and inaccurate conclusions. Mechanical samples of both the baseline and experimental SSA films were prepared in accordance with ASTM D1002 [10]. Samples were fabricated and subsequently heated at 41°F/hr (5°C/hr) and held at various elevated temperatures (248^{0}F (120^{0}C), 266^{0}F (130^{0}C), 275^{0}F (135^{0}C) and 284^{0}F (140^{0}C)) for two (2) hours. This range was selected based upon the epoxy resin matrix. The lower limit of the temperature range selected for investigation was based upon the manufacturer's curing protocol (hold at 248°F (120^{0}C) for two hours). The upper limit was selected based upon thermogravimetric analysis (TGA) which suggested 284^{0}F (140^{0}C) was the highest temperature that the parts could be safely cured without danger of thermal decomposition. Upon completion of the cure cycle, the samples were removed from the oven and their shear strength was tested. The results were graphically compared, as seen in **Figure 12**. A temperature of 284^{0}F (140^{0}C) was designated as the curing temperature because samples cured at this temperature from both the baseline (neat fenuron) as well as the encapsulated fenuron (CT012612A1) demonstrated the highest shear strength results. The baseline neat fenuron sample appeared to be completely cured at 266°F (130^{0}C) and maintained mechanical integrity from 130^{0}C (266°F) to 284°F (140^{0}C) with no apparent loss in strength. The encapsulated sample did not show appreciable strength until cured at 284°F (140^{0}C), thus indicated that curing at lower temperatures only allowed for a partial cure. It was hypothesized that the additional thermal energy was required for the DCDA to breach the shell wall of the encapsulated accelerator particles. A cure temperature of 284°F (140^{0}C) was used as the standard cure temperature for all future sample preparation.

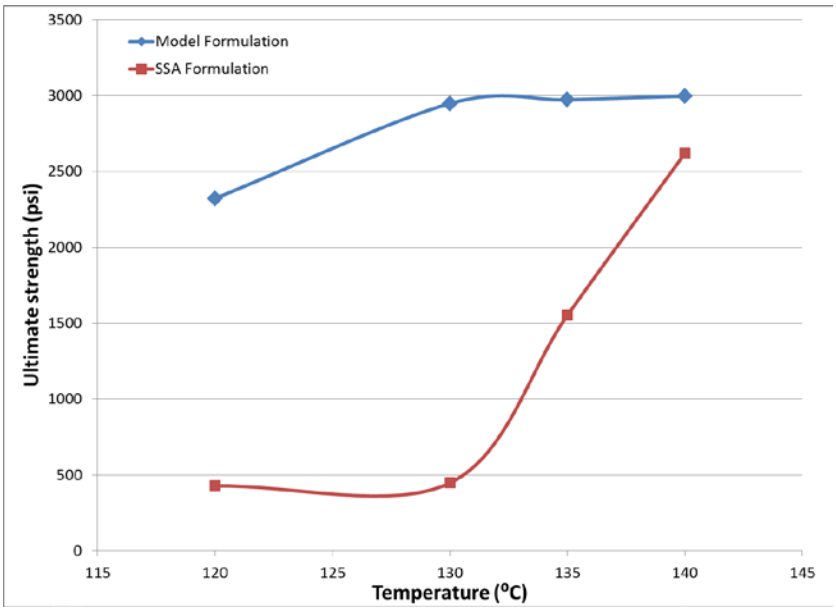


Figure 12. Data used to determine best cure protocol for mechanical samples.

Samples that were previously tested for single-lap-joint strength that were constructed using the old cure protocol (cured at 248°F (120^{0}C)) were remade and cured according to the new cure protocol at 284°F (140^{0}C). Results from both tests were then compared to one another to determine if a sample was fully cured or if curing at an elevated temperature had resulted in a decrease in mechanical performance. As seen in **Figure 13**, the ultimate strength of both formulations increased as the cure temperature increased. The increase in strength is much more drastic for the SSA specimens, increasing nearly six (6) fold.

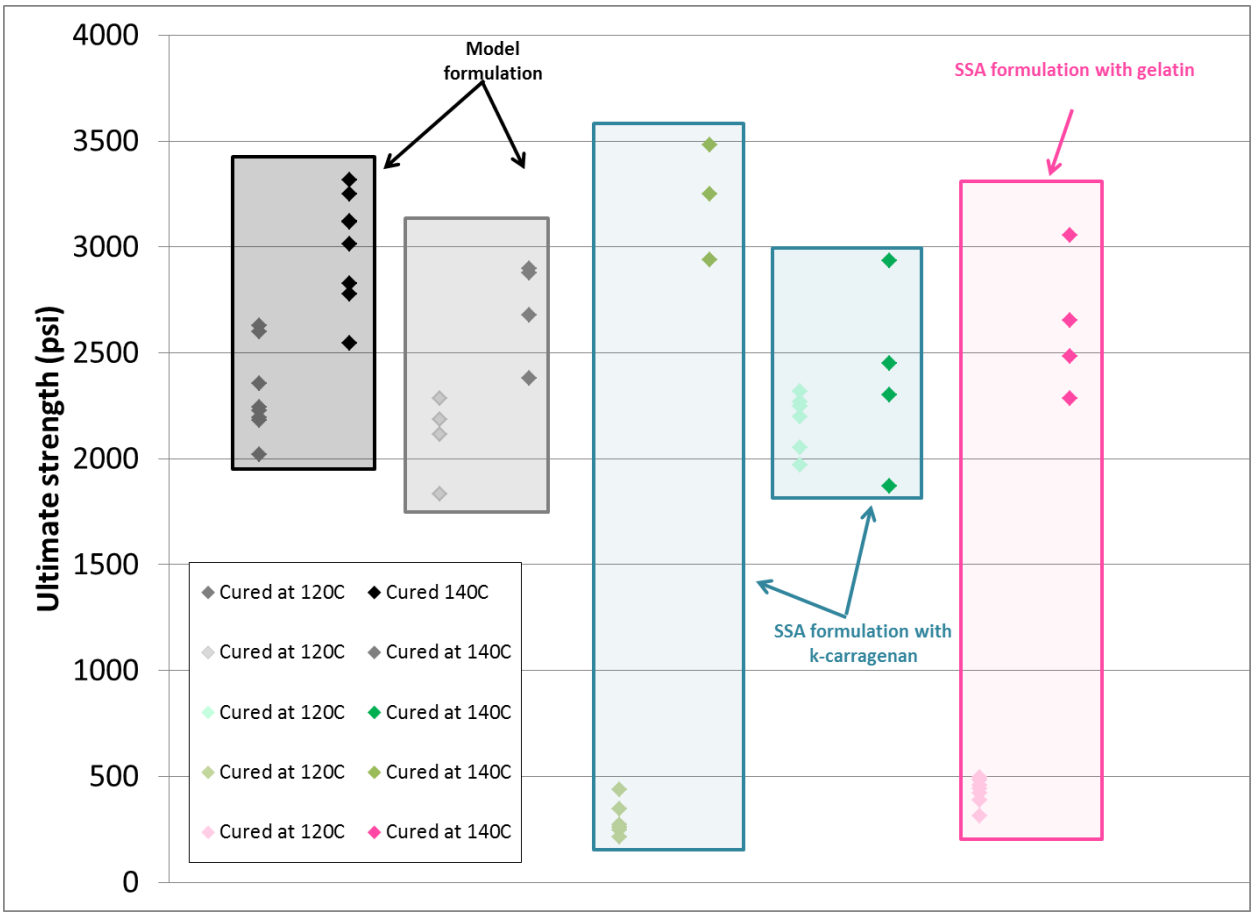


Figure 13. Ultimate strength for specimens cured at 248^{0}F (120^{0}C) as compared to specimens from the same formulation cured at 284^{0}F (140^{0}C).

4.4 Adhesive Characterization

Accurate and relevant characterization is vital to any materials development effort. Characterization activities focused on the following:

- Assessment of urone content of encapsulated accelerators
- Assessment of encapsulation material/process on activity of accelerator in terms of both shelf life and time to cure
- Assessment of impact of encapsulated accelerator on resultant film adhesive properties

Three primary characterization tools have been used to characterize materials:

- Differential scanning calorimetry (*Section 2.4.1*)
- Viscometry (*Section 2.4.2*)
- Mechanical testing (*Section 2.4.3*)

4.4.1 Differential Scanning Calorimetry (DSC) Analysis

A TA Q200 Differential Scanning Calorimeter (DSC) was utilized to analyze the temperatures and heats required to melt encapsulated urone materials. Each material was scanned in triplicate from 257^{0}F (125^{0}C) to 482^{0}F (250°C) at a constant ramp rate of 41°F/minute (5°C/minute). An example DSC curve can be found in **Figure 14**. Once the urone content of the encapsulated materials was calculated, the materials were incorporated into adhesive pastes and analyzed for cure temperature and cure energy. Each adhesive paste was mixed thoroughly and then scanned on the DSC in triplicate from 122°F (50°C) to 482°F (250^{0}C) at a constant ramp rate of 41°F/minute (5°C/minute).

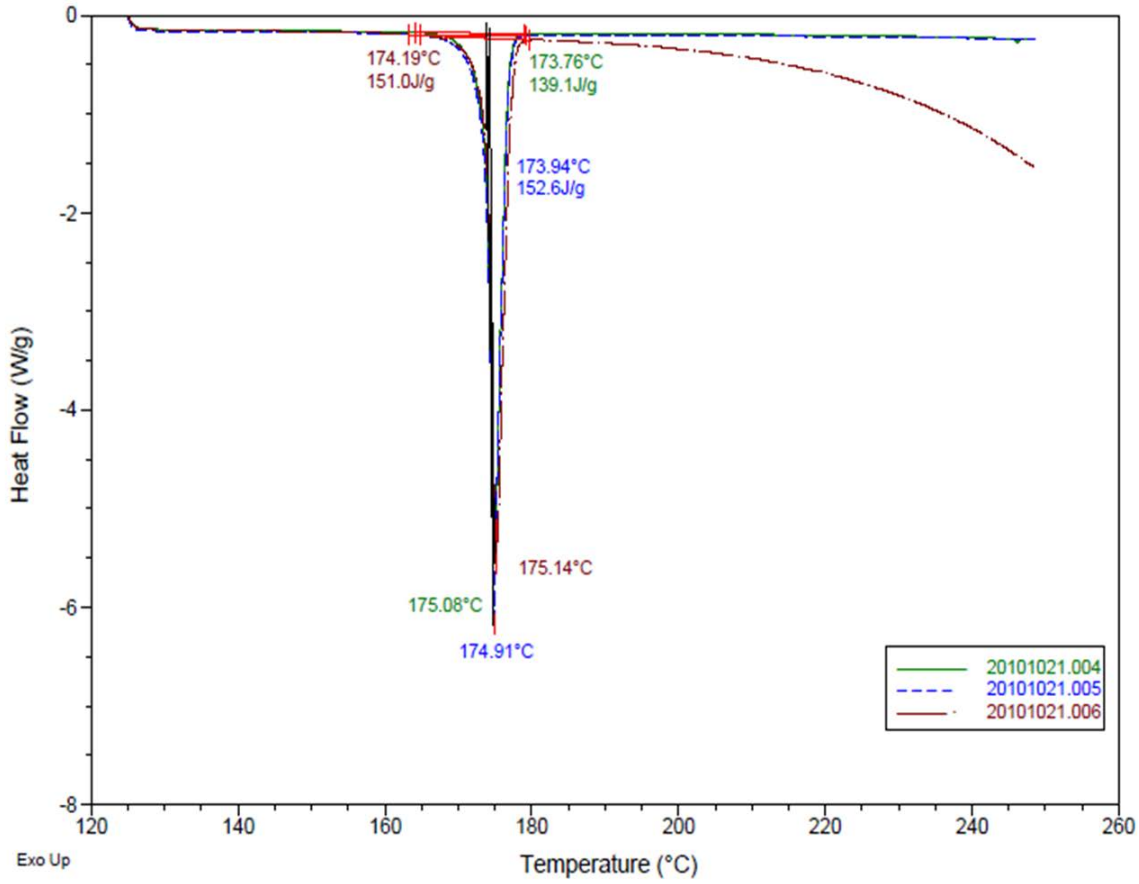


Figure 14. Sample DSC curve for neat monuron.

4.4.2 Viscosity Analysis

Curves for viscosity as a function of temperature (104°F (40°C), 140°F (60°C) and 176°F (80°C)) were generated for baseline paste adhesive formulations using neat monuron. This information was used to determine appropriate test parameters to evaluate the time to initiate cure and to identify benefits imparted by the encapsulated accelerators. Two Brookfield DV-II+ PRO Digital Viscometers, as seen in **Figure 15**, were used to simultaneously gather data. The models used allow for continuous sensing, temperature measurement and data output to a computer. A spindle speed of 0.3 RPM was selected so minimal mechanical mixing would be imparted on the samples. Measurements were taken at 5 minute intervals.

Figure 15. Viscometer setup used to determine the time to cure of two samples simultaneously

The test was concluded once the measured percent torque on the motor reached 99%. Samples tested at 176°F (80°C) reached this value after 4 hours and 25 minutes while samples tested at 140°F (60°C) reached this value after 38 hours and 30 minutes. The tests that were conducted at 104°F (40°C) were concluded after 95 hours and 20 minutes, well before this value was reached. The results of a typical test can be seen in **Figure 16**. Viscosity testing was standardized at 140°F (60°C) because it is an intermediate temperature that results in curing over days, as opposed to weeks at 104°F (40°C) (too long for practical down-selection) or hours at 176°F (80°C) (potentially too short to ensure discernable differences).

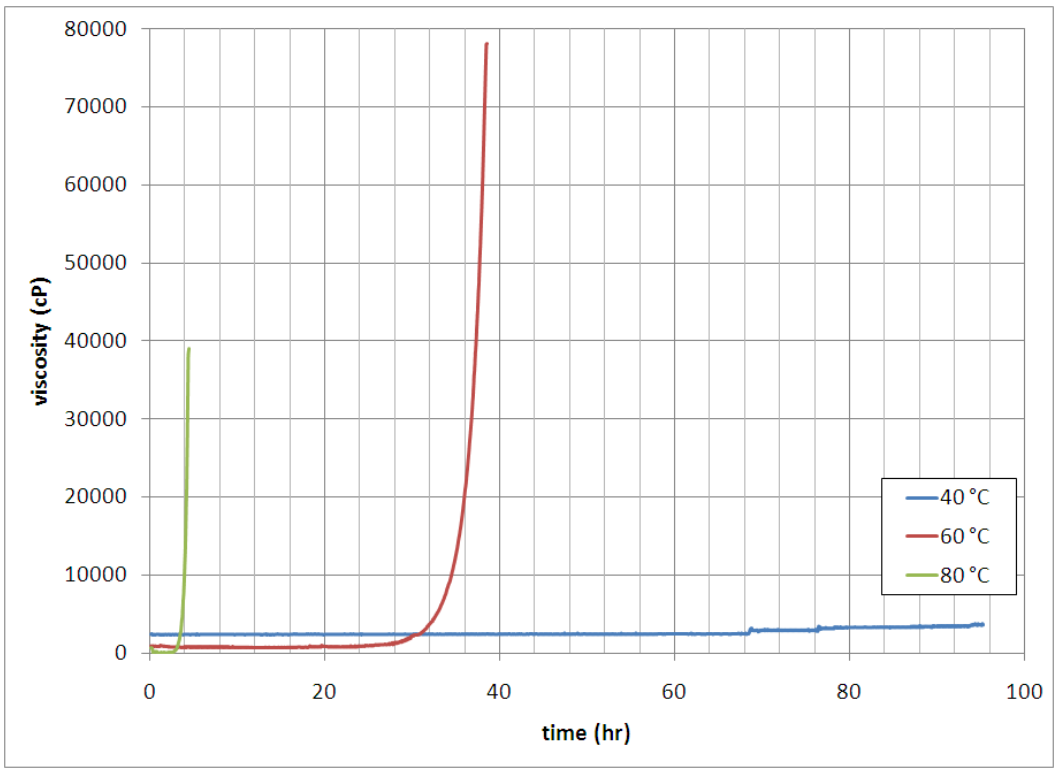


Figure 16. Viscosity curves of baseline paste adhesive measured at 104^{0}F (40^{0}C), 140^{0}F (60^{0}C) and 176^{0}F (80^{0}C). Tests were concluded once the percent torque of the motor reached 99%

4.4.3 Storage Analysis

The initial project plan specified that viscometry would be the primary tool utilized to analyze shelf-stability; however, during viscosity tests, which were conducted at 140°F (60°C), it was noted that the due to the inviscid nature of the epoxy resin system at the conditions, the fumed silica settled at the bottom of samples and may have impacted the curing mechanism. Thus, stability tests via viscosity analysis were only used as a means to screen a large number of samples with relatively low confidence. High confidence results were determined by mechanical testing films stored under controlled conditions for extended periods as described below.

4.4.3.1 Short-term Storage Stability Analysis

Epoxy films were produced and held in an oven for predetermined amounts of time to validate the results obtained from viscosity measurements. The films, which were held at 140°F (60°C), were formulated with a solid epoxy component, ensuring that there was no settling of the additives, as seen in **Table 2**.

This experiment was performed with one baseline epoxy film and other SSA films utilizing gelatin encapsulated fenuron. SSA films were made with two different batches of capsules (CT012612A3 and 1-0013-115). Single lap shear film samples (0.5” by 1” film strips) were prepared for each of the two (2) formulations to be removed from the oven every 20 hours for up

to 100 hours. In total, 27 film samples were prepared for each formulation. Upon removal from the oven at defined time intervals, the film samples were laid out on Q Panel aluminum panels for lap shear testing. The samples were cured at 284°F (140°C) for two hours and their single lap shear strength was determined using a mechanical tester. The samples were tested in accordance with ASTM D1002 [10] and the load at break was recorded to determine if exposure to elevated temperature lead to any degradation in mechanical strength within the first 100 hours of storage at 140°F (60°C).

Table 2. Baseline epoxy resin system used for film production.

Adhesive component	Parts per resin
DER 331 liquid epoxy resin	60
DER 661 solid epoxy resin	40
Omnicure DDA5 (DCDA)	5
Accelerant	3
Fumed silica	3

4.4.3.2 Long-term Storage Stability Analysis

The long-term stability test was performed both at room temperature and at a minimally elevated temperature (32°C (90°F)). Due to limited supply of material, capsules from batch number CT012612A1 were used in this test. These capsules had similar thermal and mechanical properties as capsules from the CT012612A3 batch used to make the SSA film in the short-term storage stability analysis testing described above. Single lap shear film samples (0.5” by 1” film strips) were prepared for both the baseline formulation and the SSA formulation. These films were removed from the oven at three month intervals up to one year. A final set of film samples was removed from the oven and tested after two years of exposure.

4.5 Performance Testing

Mechanical screening tests were performed to help rank the relative strength and performance of each of the candidate capsule batches. Two sets of specimens were fabricated using the baseline adhesive formulation and the most promising experimental formulation was sent to a third-party vendor (Intertek, Pittsfield, MA).

4.5.1 Mechanical Screening

Initially, samples were prepared from 0.008” thick prepreg under vacuum, with no compression or immobilization. The specimens produced in this manner had large voids and poor adhesion to the aluminum substrate, as seen in **Figure 17**. The mechanical data from baseline neat accelerator specimens such as these was typically very scattered and had poor maximum load at break values. A typical baseline load at break average was about 1300 psi using unrefined processing conditions. The optimized baseline processing method yielded much more consistent data with higher yield at break values. A typical baseline load at break average was about 2400 psi using optimized processing conditions.

The optimized method used the following parameters:

- Resin solid to liquid ratio (D.E.R. 331 and D.E.R. 661, respectively): 40/60
- Film thickness: 0.016”
- Film post processing: additional heat was applied to prepreg increase fiber wetting
- Cure atmosphere: cured under ambient pressure
- Specimen compression: a 100 g steel slug was placed on each sample
- Specimen immobilization: a fixture was devised to keep the specimens in alignment (shown in **Figure 18**)

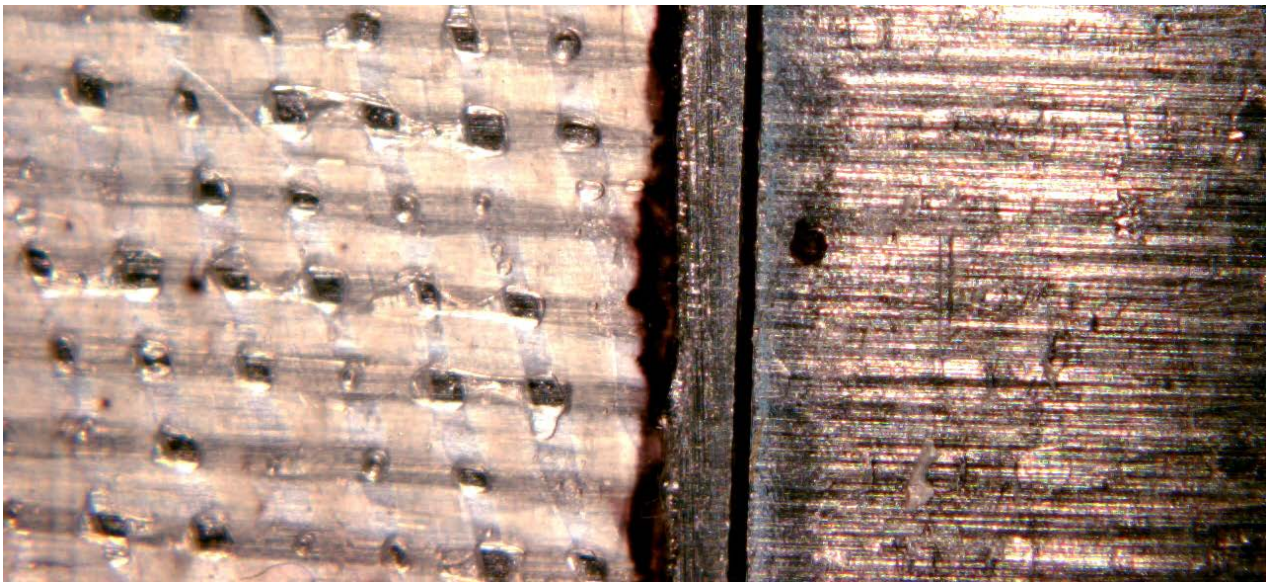


Figure 17. Close up of a lap shear sample after testing. The plate on the left retained nearly all of the adhesive, while the plate on the right had virtually no prepreg material. Also, significant number of voids can be in the remaining adhesive on the left.

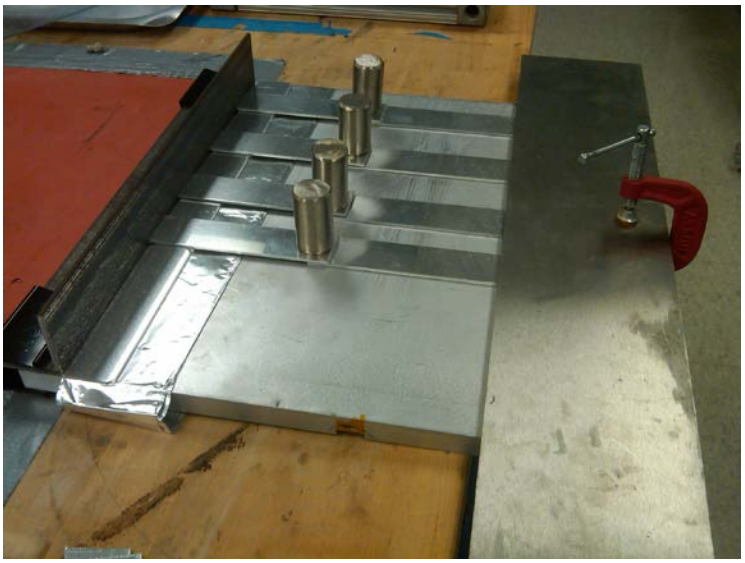


Figure 18. Lap shear specimen alignment fixture and compression slugs

Adhesive film samples were tested for shear strength of single-lap-joints in accordance with ASTM D 1002 [10]. Type 2024 T3 aluminum samples were purchased from Q-Lab. Each panel had the dimensions 4.0 ± 0.005 in. x 1 ± 0.005 in x 0.064 ± 0.005 in (101.6 ± 0.25 mm x 25.4 ± 0.25 mm x 1.62 ± 0.125 mm). Before applying the film, the panel was cleaned with acetone. Samples were prepared as illustrated in **Figure 19**. A 1 in x 0.5 in strip of adhesive film is applied to one end of the panel and a second panel is adhered. Samples were then placed in a programmable oven and cured according to the protocol discussed in *Section 2.3.3.* Samples were removed from the oven at the end of the cure cycle and then the shear strength of the film is measured using an Instron Testing Machine. The panels were secured into the Instron Testing Machine and pulled at a rate of 0.1 in/min until the adhesive breaks.

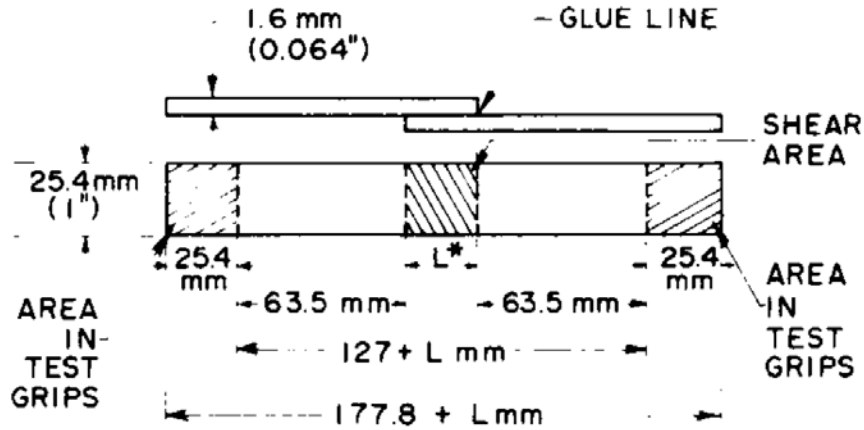


Figure 19. Form and dimensions of Shear Strength of single-lap-joint test specimens [10]

4.5.2 Validation Testing

Five (5) separate mechanical tests were performed under six (6) post-cure aging conditions, as outlined in **Table 3.** Nine (9) of each specimen, baseline and SSA, were submitted for each test indicated below (except the climbing drum peel, only three (3) SSA samples were submitted for each set of test conditions). Each test is described in further detail in the following subsections.

4.5.2.1 Shear Strength of Single-Lap-Joint

This test was performed to validate results obtained during screening tests as well as to quantify the shear strength of the SSA in austere conditions. Samples were prepared and tested as described in *Section 2.5.1.1*. Samples were placed in a programmable oven, cured according to the protocol discussed in *Section 2.3.3,* and then removed from the oven at the end of the cure cycle. Samples of both the baseline adhesive material and the SSA material were sent to Intertek where they were conditioned as outlined in **Table 3** and subsequently tested at ambient temperature, -67^{0}F (-55^{0}C) and 180^{0}F (82.2^{0}C). The shear strength of single-lap-joint samples, as measured by Intertek, was compared to those obtained by Infoscitex to confirm that they were comparable prior to performing additional validation tests. This was done to determine reliability in data obtained from Intertek and to help determine consistency in batches of capsules made.

Table 3. Summary of validation mechanical testing and post-curing conditions.

Objective	Conditioning		ASTM D1002	ASTM D3528	ASTM D1781	ASTM C393	ASTM C297
	Post-Cure Aging	Test Conditions	Single Lap Shear	Double Lap Shear	Climbing Drum Peel	3-Point Sandwich Flex	Flatwise Tensile
Control	none	ambient	x	x	x	x	x
Dry Extreme Temperature	none	-67°F	x	--	--	--	x
Dry Extreme Temperature	none	180°F	x	--	--	--	x
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	x	--	--	--	--
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	x	--	x	--	x
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	x	x	x	x	x

4.5.2.2 Strength Properties of Double Lap Shear Joints by Tension Loading

Strength properties of double lap shear joint by tension loading were evaluated in accordance with ASTM D 3528 [11]. Type 2024 T3 aluminum samples were purchased from Q-Lab. Each panel had the dimensions 4.25± 0.01 in x 1.0 ± 0.01 in x 0.125 in ± 0.005 in (108 ± 0.25 mm x 25.4 ± 0.25 mm x 3.24 mm ± 0.125 mm). Before applying the film, each panel was cleaned with acetone. Samples were then prepared as illustrated in **Figure 20**. Two strips of adhesive film measuring 1" x 0.5" were applied to either side of one end of a panel. Two additional panels were then adhered to the first panel. Samples were then placed in a programmable oven and cured according to the protocol discussed in *Section 2.3.3*. Samples were removed from the oven at the end of the cure cycle. Samples of both the baseline adhesive material as well as the SSA material were sent to Intertek where they were conditioned as outlined in **Table 3** and subsequently tested at ambient temperature. The strength of the film by tension loading was then measured using an Instron Testing Machine. The panels were secured into the Instron Testing Machine and pulled at a rate of 0.1 in/min until the adhesive broke.

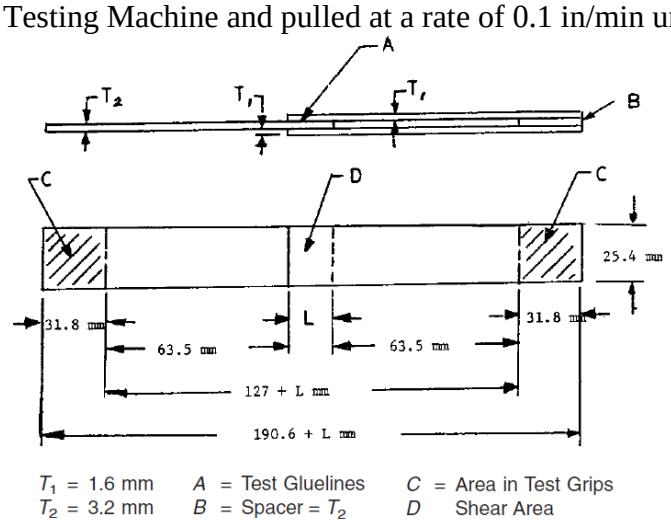


Figure 20. Form and dimensions of Shear Strength of single-lap-joint test specimens [11]

4.5.2.3 Climbing Drum Peel for Adhesives

Samples were tested for peel resistance of adhesive bonds between an aerospace-grade foam core and metal substrate in accordance with ASTM D 1781 [12]. Type 2024 T3 aluminum samples were purchased from Q-Lab. Each panel had the dimensions 12.0 ± 0.005 in x 3.0 ± 0.005 in x 0.020 ± 0.005 in (304.8 ± 0.25 mm x 76.2 ± 0.25 mm x 0.50 ± 0.125 mm). Before applying the film, each panel was cleaned with acetone. Samples were then prepared as illustrated in **Figure 21A**. Two strips of adhesive film measuring 12" x 3" were applied to two separate cleaned aluminum substrates. The aluminum/film was then applied to the foam core and the samples were then placed into a specially constructed fixture to ensure proper alignment during curing. Samples were then placed in a programmable oven to be cured according to the protocol discussed in *Section 2.3.3* and then removed from the oven at the end of the cure cycle. Cured samples were sent to Intertek for conditioning as outlined in **Table 3** above. The peel resistance of the film by tension loading was then measured using an Instron Testing machine at ambient temperature and 180^{0}F (82.2^{0}C) as seen in **Figure 21B**. The sandwiches were pulled at a rate of 1.00 ± 0.10 in/min (25.40 ± 2.54 mm/min) until failure was observed.

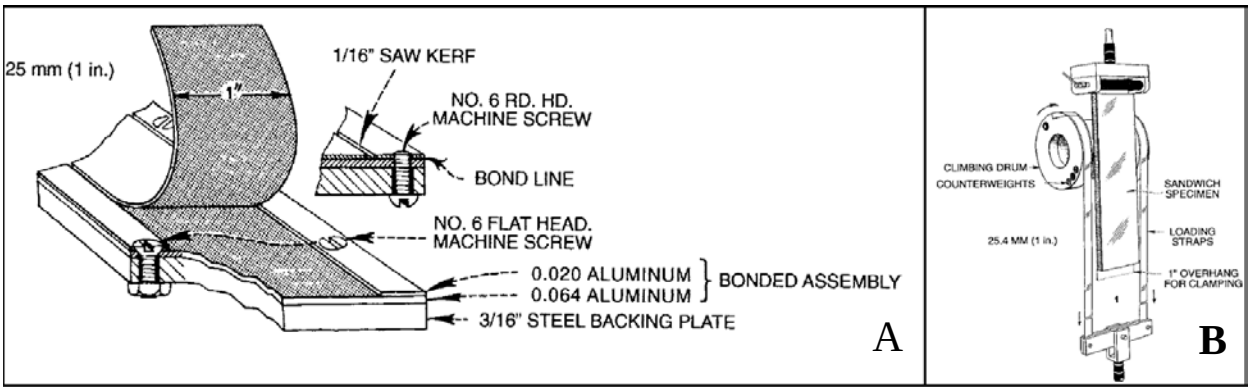


Figure 21. A) Diagram of laminated test specimen assembly, B) Assembly of peeling apparatus [12]

4.5.2.4 Core Shear Properties of Sandwich Constructions by Beam Flexure

Samples were tested for core shear properties of sandwich construction by being subjected to flexure in such a manner that the applied moments produce curvature of the sandwich facing planes in accordance with ASTM C393 [13]. This test method was used to determine the core shear properties of flat sandwich constructions. Type 2024 T3 aluminum samples were purchased from Q-Lab. Each panel had the dimensions 8.0 ± 0.005 in x 3.0 ± 0.005 in x 0.064 ± 0.005 in (200 ±0.25 mm x 75 ± 0.25 mm x 1.62 ± 0.125 mm). According to ASTM C393, permitted core materials include those with continuous bonding surfaces (such as balsa wood and foams) as well as those with discontinuous bonding surfaces (such as honeycomb). The material that was selected for this evaluation was aerospace-grade closed cell foam. Before applying the film, each panel was cleaned with acetone. Samples were then prepared as illustrated in **Figure 22A**. Two strips of adhesive film measuring 8" x 3" were applied to two separate cleaned aluminum substrates. The aluminum/film was applied to the foam core and the samples were then placed into a specially constructed fixture to ensure proper alignment during curing. Samples were then placed in a programmable oven to be cured according to the protocol

discussed in *Section 2.3.3* and then removed from the oven at the end of the cure cycle. Cured samples were sent to Intertek for conditioning as outlined in **Table 3** above. The core shear properties of the sandwich due to beam flexure was measured using an Instron mechanical testing machine at ambient temperature. The sandwiches were then subjected to a bending moment normal to the plane of the sample, as seen in **Figure 22B**. The only acceptable failure modes, according to ASTM C393, are core shear or core-to-facing bond: failure of the sandwich facing preceding failure of the core or core-to-facing bond is not an acceptable failure mode

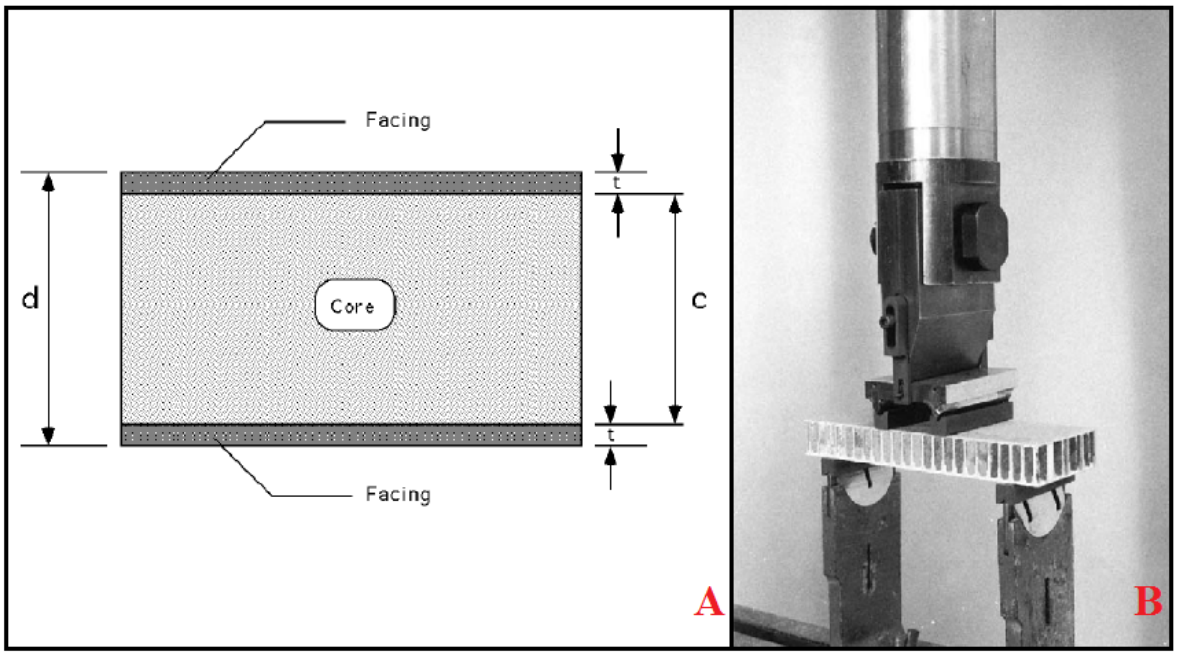


Figure 22. A) diagram of construction of samples, B) image of test rig used to test samples.

4.5.2.5 Flatwise Tensile Strength of Sandwich Constructions

Samples were tested for flatwise tensile strength of the core-to-facing bond of the adhesive in accordance with ASTM C 297 [14]. Type 2024 T3 aluminum samples were purchased from Q-Lab. Each panel had the dimensions 1.0 ± 0.005 in x 1.0 ± 0.005 in x 0.020 ± 0.005 in (25.4 ± 0.25 mm x 25.4 ± 0.25 mm x 0.50 ± 0.125 mm). The foam selected was aerospace-grade closed cell foam. Before applying the film, each panel was cleaned with acetone. Samples were then prepared as illustrated in **Figure 23A**. Two strips of adhesive film measuring 1" x 1" were applied to two separate cleaned aluminum substrates. The aluminum/film was applied to the foam core and the samples were then placed into a specially constructed fixture to ensure proper alignment during curing. Samples were then placed in a programmable oven to be cured according to the protocol discussed in *Section 2.3.3* and then removed from the oven at the end of the cure cycle. Cured samples were sent to Intertek for conditioning as outlined in **Table 3** above. The peel resistance of the film by tension loading was measured using an Instron mechanical testing machine at ambient temperature, -67^{0}F (-55^{0}C) and 180^{0}F (82.2^{0}C). The sandwich construction samples were then subjected to a uniaxial tensile force normal to the plane of the sandwich which was transmitted to the sample through thick loading blocks which are bonded to the sandwich, as seen in **Figure 23B**. The sandwiches were pulled at a head

displacement rate of 0.020 in/min (0.50 mm/min) until failure is observed. The only acceptable failure modes for flatwise tensile strength, according to ASTM C297, are those which are internal to the sandwich construction: failure of the loading block-to-sandwich bond is not an acceptable failure mode.

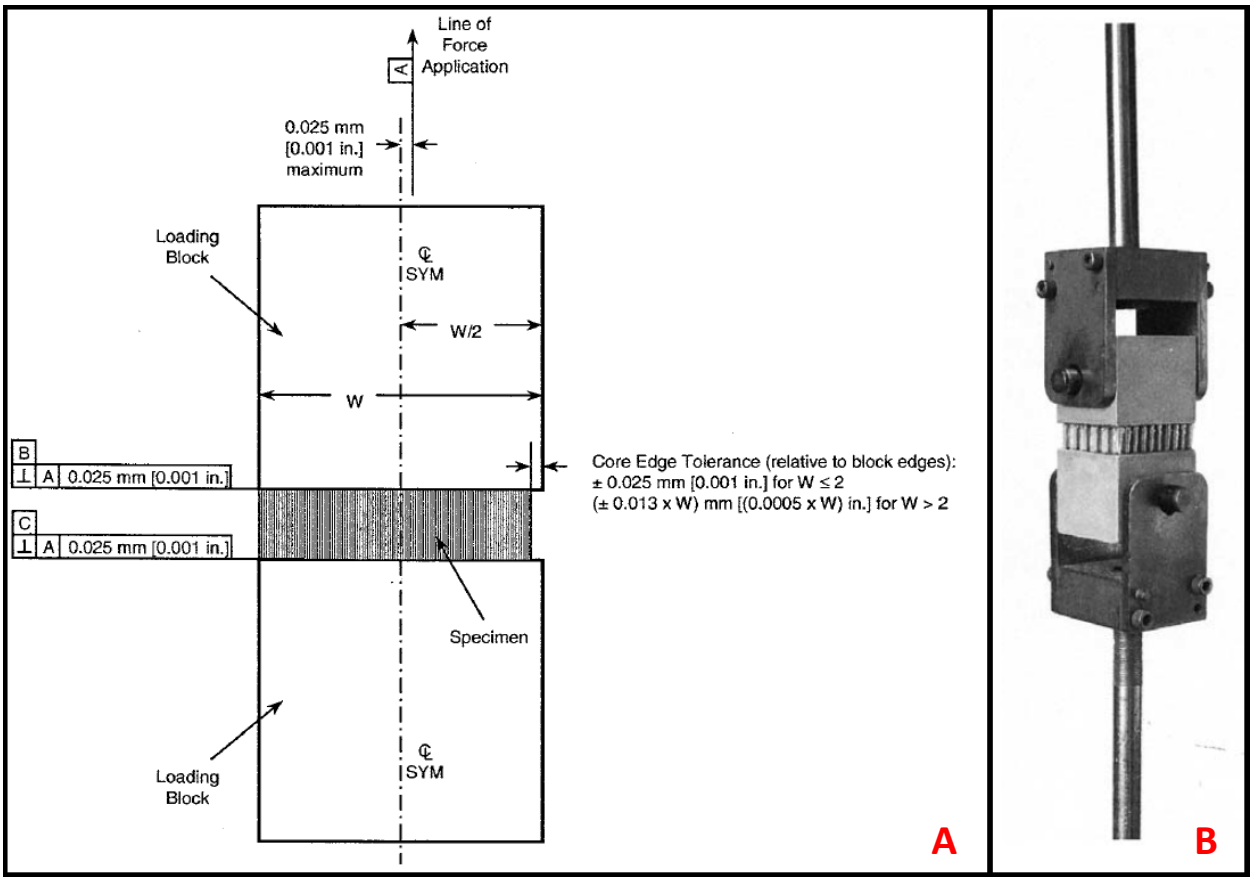


Figure 23. A) Maximum permissible dimensions of flatwise tension samples B) Flatwise tension setup [14]

5. Results and Discussion

This effort has served to establish an isolated fenuron accelerator for use in a stable one-part epoxy film. Specifically, results are summarized as follows:

- Two (2) methods of encapsulating urone accelerators were investigated, in-situ polymerization and complex coacervation. Results strongly indicated that complex coacervation yielded encapsulated accelerators with targeted stability and activity.
- Three (3) urone accelerators were investigated: monuron, fenuron and TDU. Ultimately, due to environmental safety concerns, fenuron was selected for incorporation in the final formulation.
- Three types of shell materials were investigated: KC- PDDACL, IC- PDDACL and gelatin. Gelatin was selected due to observed batch-to-batch consistency, ease of scalability, and excellent formulation stability.
- The ability to produce adhesive films featuring encapsulated accelerants was demonstrated.
- The initial scale-up of the encapsulation process was demonstrated from 25g batches to 75g batches.
- Shelf stability of the formulation was demonstrated to be greater than one year at 90°F.
- *Mechanical performance tests have demonstrated that the candidate SSA formulation has strength similar to or greater than the baseline after aging and hot/wet testing.*

5.1 Microencapsulation Results

5.1.1 In-Situ Encapsulation

5.1.1.1 In-Situ Generation of DMA

The production of DMA and isocyanate as a function of temperature, incubation time and urone type was determined using FTIR analysis. Samples were compared to each other using a ratio of the area beneath the absorbance peaks for NCO (2290 to 2230 cm^{-1}) to the area beneath the absorbance peaks for CH bonds (3000 to 2800 cm^{-1}) as described in literature [15]. For monuron samples, there was no change after incubation at 158°F (70°C). However, as seen in **Figure 24**, the monuron samples showed an increase in the NCO content after incubation at 212°F (100°C) for 30 minutes. These conditions led to the most statistically significant increase in the NCO content. At longer incubation periods and high incubation there was samples exhibited a loss of NCO content. This can be explained by gradual loss of isocyanate due to its further decomposition.

The data obtained with fenuron did not show a distinctive pattern within the explored condition ranges, as seen in **Figure 25**.

The samples of TDU demonstrated an increase in the NCO content after incubation at elevated temperatures, however, the statistical significance of the change was less pronounced than for the monuron based samples, as seen in **Figure 26**. Similar to monuron, there was a loss of NCO content with longer incubation times and higher incubation temperatures. This can be explained by gradual loss of isocyanate due to its further decomposition.

From these experiments, it was determined that it was possible to perform the in-situ encapsulation of monuron (at 212°F (100°C) for 30 minutes) and TDU (at 158°F (70°C) to 185°F (85°C) for 60 minutes). However, the in-situ encapsulation of fenuron could not be performed at temperatures below 221°F (105°C) and incubation periods under 4 hours.

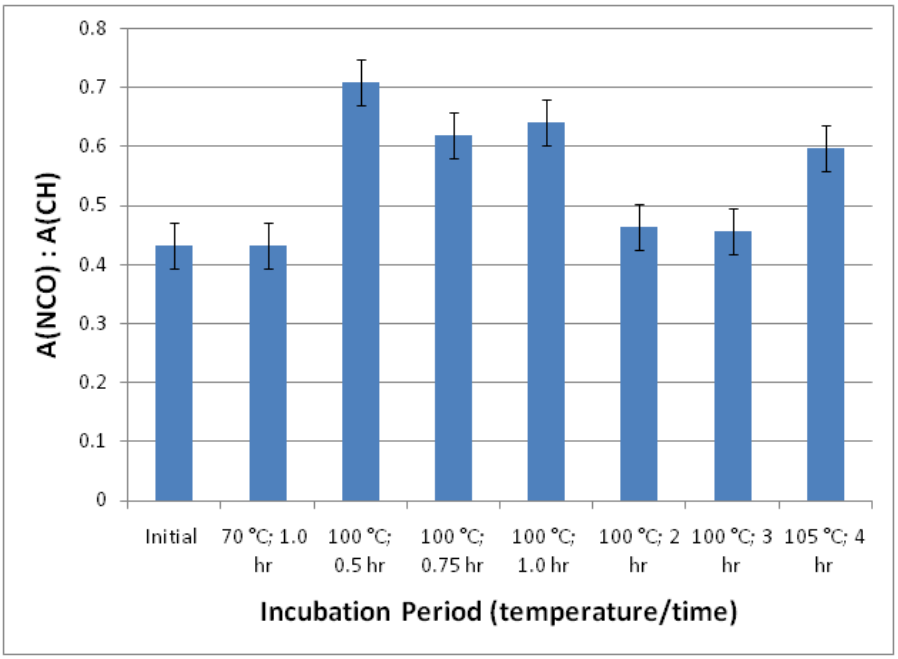


Figure 24. NCO production in monuron samples

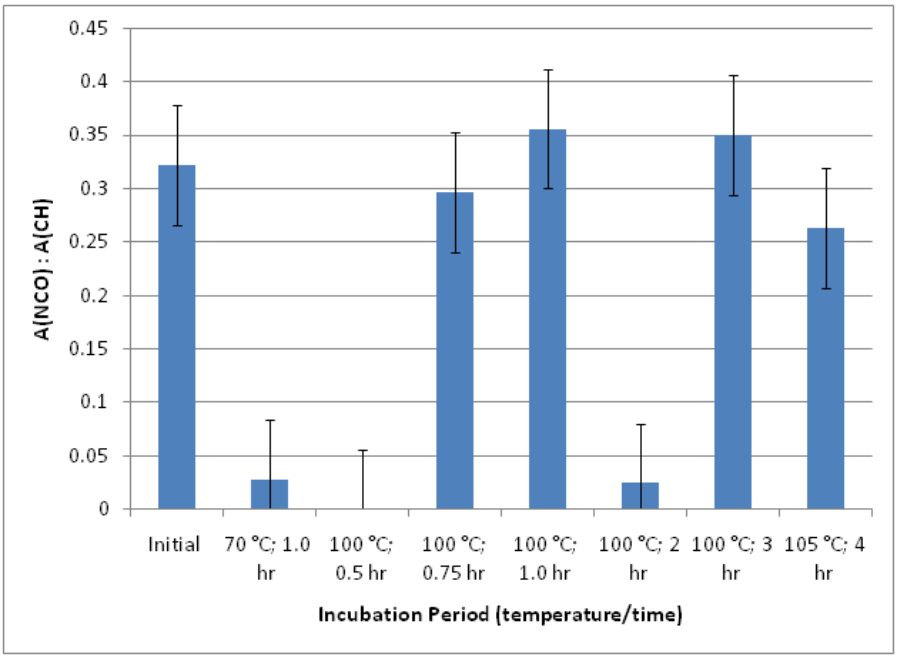


Figure 25. NCO production in fenuron samples

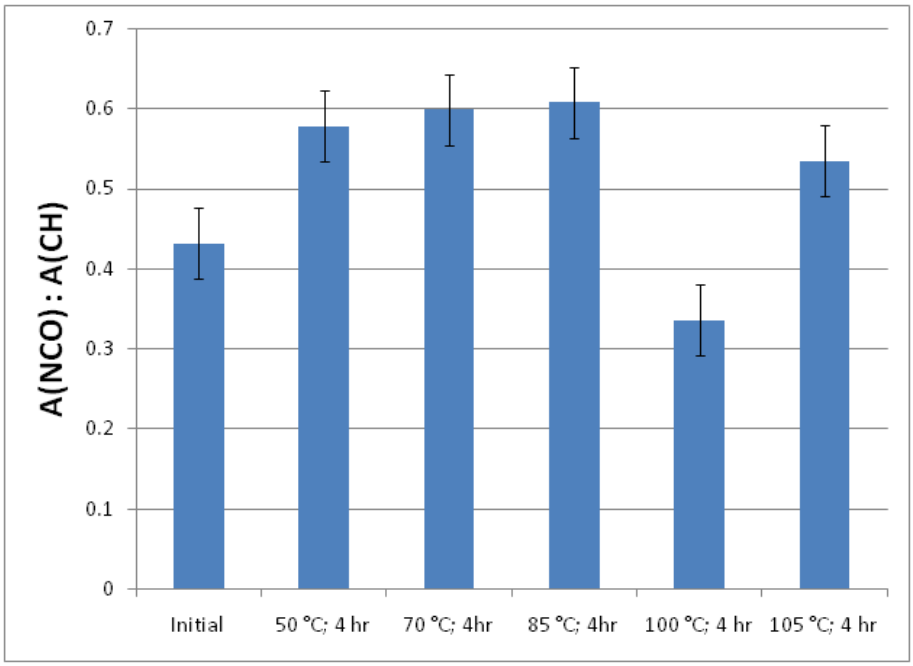


Figure 26. NCO production in samples of TDU

5.1.1.2 In-situ Polymerization of Epoxy Shell on Urone Crystal Surface

The first in-situ polymerization attempt was performed in ECH, as described in *Section 2.2.1.2*. The reaction was started by rising the temperature to reach the target 212°F (100°C) (the optimal monuron decomposition temperature). Samples of the reaction mixture were withdrawn from the flask prior to heating and once the mixture reached 104°F (40°C). It was observed that almost complete dissolution of monuron in the solvent occurred when the temperature of the reaction mixture reached 158°F (70^0C) and heating was discontinued. The reaction mixture was collected and stored in a flask. No analysis of the product was performed because no monuron crystals were present during incubation at elevated temperature.

To decrease solubility of monuron in the heated reaction mixture, several different solvents were identified, such as toluene, cyclohexane, and decane [16]. The solubility of monuron at room temperature in each of these solvents is: 0.001155, 0.00005088, and 0.00010076 mole fraction, respectively. Toluene was selected as the first solvent for investigation. The in-situ polymerization reaction in excess toluene was performed as outlined in *Section 2.2.1.2*. The initial reaction mixture sample was collected. The reaction was started by bringing the temperature to the 212°F (100^0C) (the optimal monuron decomposition temperature). Samples of the reaction mixture were withdrawn from the flask prior to heating and once the mixture reached 104°F (40^0C). It was observed that some dissolution of monuron in the co-solvent occurred when the temperature of the reaction mixture reached 212°F (100^0C). The incubation was continued for 2 hours. Samples of the reaction mixture were collected when the temperature reached 212°F (100^0C) and every 30 minutes after. The reaction samples were dried under vacuum overnight and quenched in 0.5 mL DETA. The quenched samples were kept for potential investigation of the reaction course.

After the 2 hour incubation period, heating was discontinued. It was observed that an abundant amount of newly formed monuron crystals was produced during cooling. The precipitate was collected by vacuum filtration, washed twice with toluene and mixed with DETA to quench the in-situ epoxy and dissolve unreacted monuron. A sample of the end product was further washed once with DETA and twice with toluene, dried and subjected to DSC analysis for monuron content. The DSC analysis, as seen in **Figure 27**, of the encapsulated material (IST Run-2) when compared with raw monuron showed 110% content of monuron in the final product. It was concluded that possible chemical modification of monuron could have occurred in the toluene-ECH solution. Based on the observed dissolution and re-crystallization of monuron, as well as its potential chemical modification, it was concluded that the in-situ encapsulation reaction should be performed at lower temperatures to maintain monuron non-dissolved and to avoid undesired chemical modification of monuron.

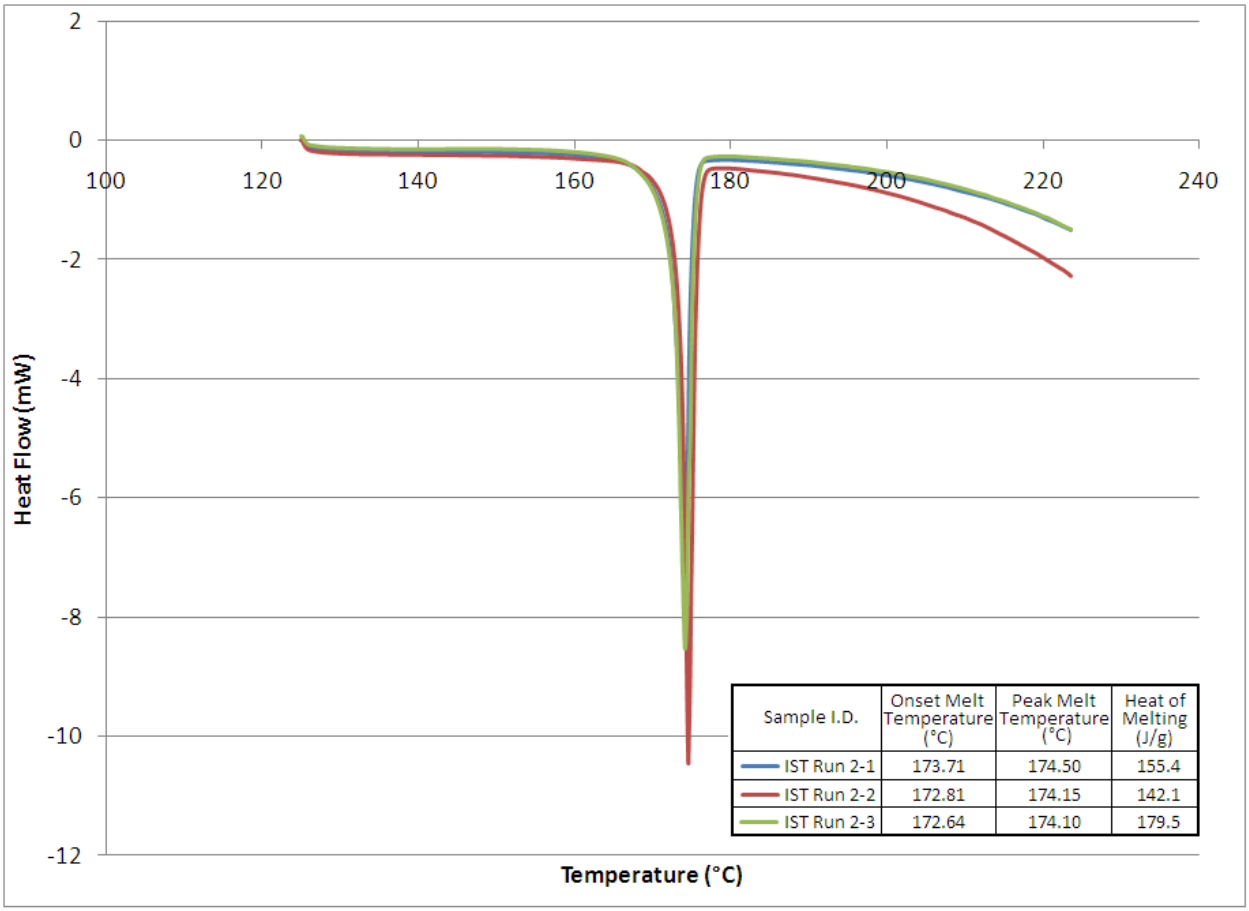


Figure 27. DSC data for IST Run-2, epoxy encapsulated monuron. Average onset of melt temperature = 173.05^{0}C, average peak melt temperature = 174.25^{0}C, average heat of melting = 159.00 J/g. Active monuron in sample (comparison of heat of melting of IST Run-2 to raw monuron) = 109.68%

To further decrease solubility of monuron in the heated reaction mixture, the reaction temperature was decreased to 176°F (80^{0}C). It was determined that moderate decomposition of dry monuron at 176°F (80^{0}C) started after approximately 19 hours of incubation. The in-situ

polymerization reaction in excess toluene was performed as outlined in *Section 2.2.1.2*. A violent reaction occurred and an exotherm was observed when the temperature in the reaction mass reached 167°F (75^{0}C), thus heating was discontinued. The reaction mass temperature reached 181°F (83^{0}C) and a color change was observed (bisque color). When the reaction mass cooled to 176°F (80^{0}C), heating was resumed. Reaction mixture samples were collected when the mixture temperature reached 176°F (80^{0}C) and after 6, 24, 32 and 39 hours of reaction. These samples were dried, quenched with DETA, and kept for further investigation. After the 39 hour incubation period, the heat was discontinued. Once the reaction mass reached room temperature, the precipitate was collected by vacuum filtration, washed twice with toluene, collected and mixed with DETA to quench the in-situ epoxy polymerization and dissolve unreacted monuron. The end product was further washed once with DETA and twice with toluene, dried and subjected to DSC analysis for the monuron content. A total of 5.00 g of the reaction product was obtained. The DSC analysis, as seen in **Figure 28**, of the encapsulated material (IST Run-3) when compared with raw monuron showed 98% content of monuron in the final product. Optical microcopy showed a thin encapsulated layer on the surface of the product, as seen in **Figure 29**.

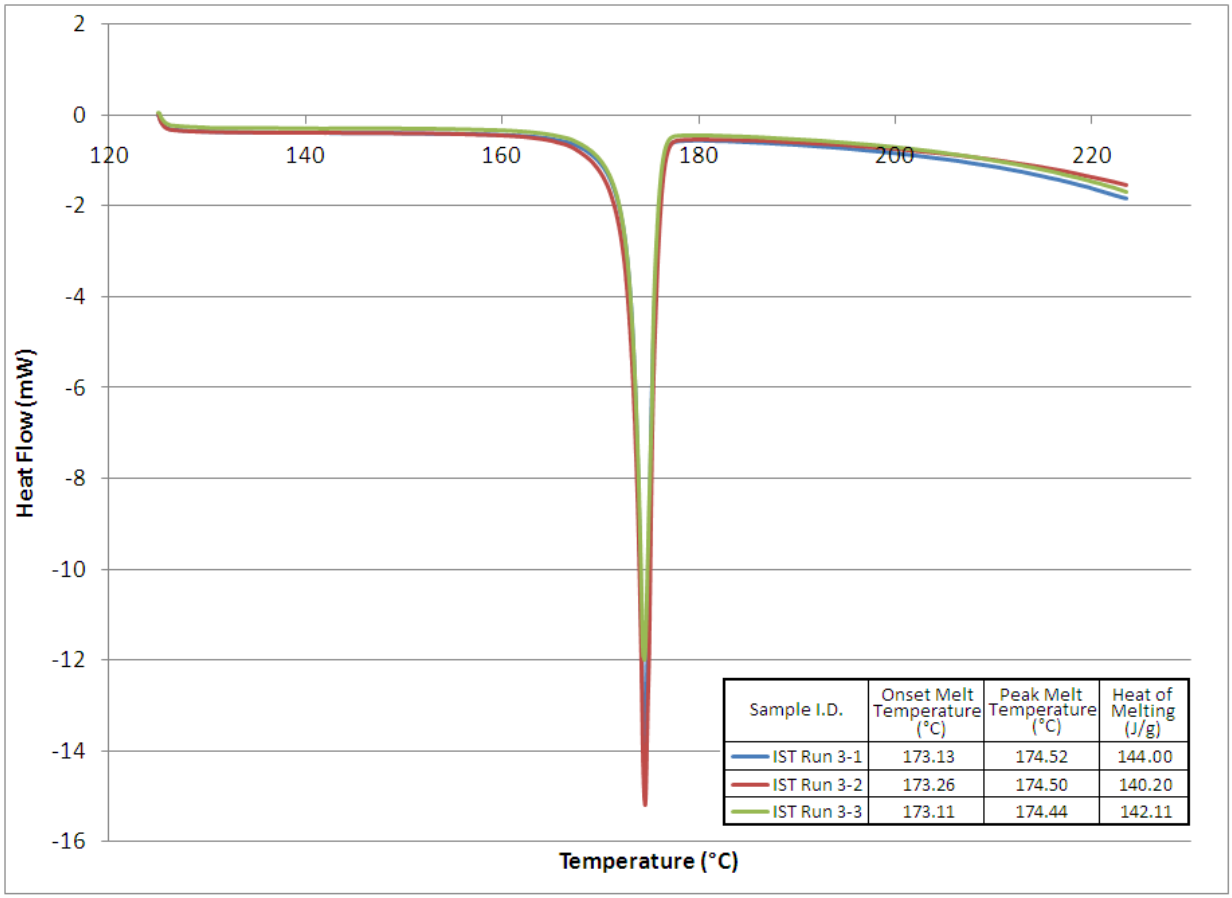


Figure 28. DSC data for IST Run-3, epoxy encapsulated monuron. Average onset of melt temperature = 173.17^{0}C, average peak melt temperature = 174.49^{0}C, average heat of melting = 142.10 J/g. Active monuron in sample (comparison of heat of melting of IST Run-3 to raw monuron) = 98.02%

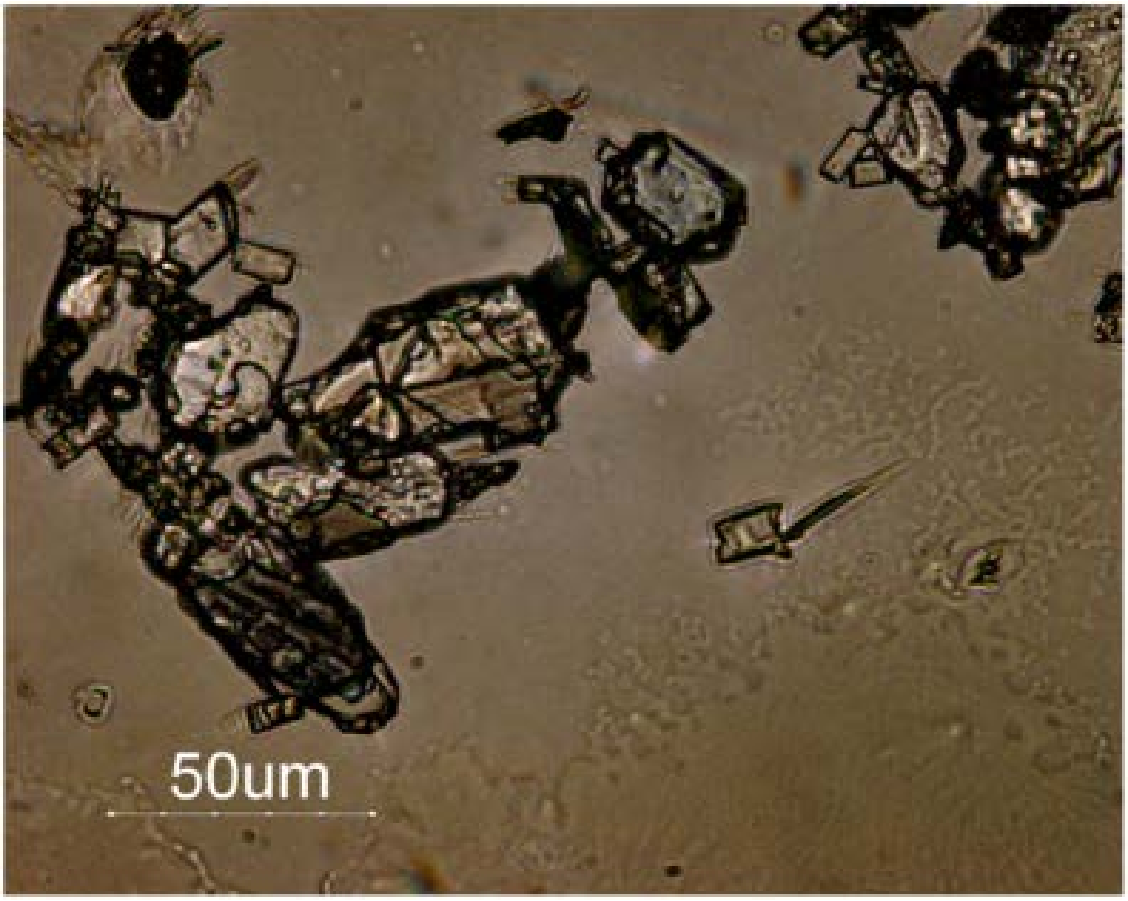


Figure 29. Micrograph of monuron encapsulated via in-situ polymerization

5.1.1.3 Encapsulation of Urones within the Adhesive Formulation

The encapsulation of monuron crystals in an epoxy shell formed by D.E.R. 331 and DETA was attempted. The reaction mixture containing monuron and D.E.R. 331 in toluene was prepared as previously described (*Section 2.2.1.2*); however, the mixture was not heated. The mixture was stirred to allow D.E.R. 331 adsorption on the surface of monuron crystals for one (1) hour. Then 0.3 g of DETA was added to the mixture. The mixture was then stirred for 72 hours. The sediment was collected by vacuum filtration and washed 3 times with toluene. This resulted in collection of 16.6 g of wet product. The product was dried under vacuum and yielded 12.7 g of product. The DSC analysis, as seen in **Figure 30,** of the encapsulated material (IST Run-6) when compared with raw monuron showed 80% content of monuron in the final product.

Similarly to the encapsulation of monuron using D.E.R. 331, the in-situ polymerization was to be conducted in excess of a solvent incapable of TDU solubilization while maintaining D.E.R. 331 in solution. Information regarding the solubility of TDU in organic solvents was not readily available. A quick visual solubility test was performed using small amounts of candidate solvents, TDU and D.E.R. 331. Mixtures were placed in sealed vials and incubated at room temperature and in a temperature-controlled bath at 70.0^{o}C for 60 min. The results of the solubility tests are shown in **Table 4**.

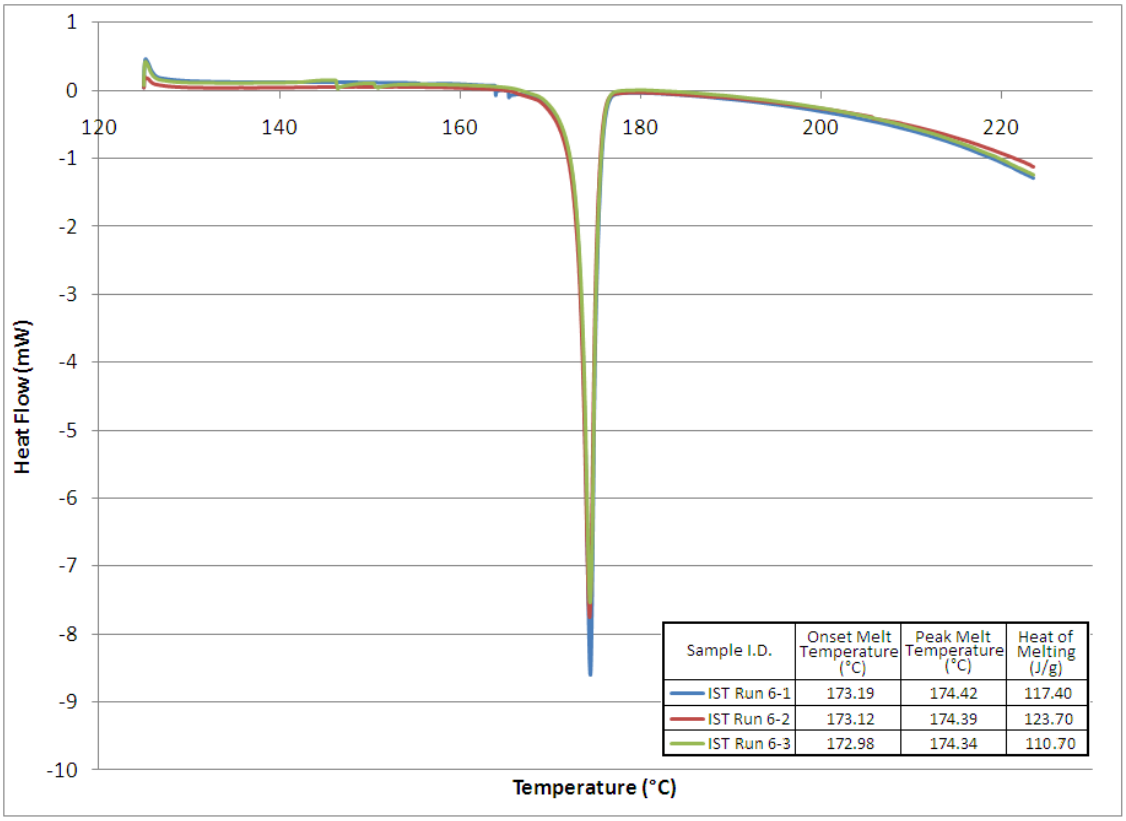


Figure 30. DSC data for IST Run-6, epoxy encapsulated monuron. Average onset of melt temperature = 173.10^{0}C, average peak melt temperature = 174.38^{0}C, average heat of melting = 117.27 J/g. Active monuron in sample (comparison of heat of melting of IST Run-6 to raw monuron) = 80.89%

As shown in **Table 4**, almost all of the solvents readily dissolved D.E.R. 331. Some solvents did not dissolve TDU; however, the powder was slightly discolored which was indicative of decomposition. Several solvents (n-decane, n-hexadecane, and xylenes) did not visibly dissolve TDU and did not cause discoloration; however, they completely dissolved D.E.R. 331 at 70 °C. Among those solvents, xylenes had the lowest boiling point. Thus, this solvent was selected for ease of its evaporation during drying of the final reaction product. In-situ encapsulation of TDU was conducted in excess xylenes at 70°C as described in *Section 2.2.1.2*. Two experiments were performed. The DSC analysis for the first trail, as seen in **Figure 31**, of the encapsulated material (IST Run-7) when compared with raw monuron showed 129% content of monuron in the final product. The DSC analysis for the first trail, as seen in **Figure 32**, of the encapsulated material (IST Run-8) when compared with raw monuron showed 97% content of monuron in the final product.

Table 4. Solubility of TDU in various solvents

Solvent	Room Temperature		70^{0}C		Boiling point (^{o}C)
	TDU	D.E.R. 331	TDU	D.E.R. 331	
1-Methyl-2-pyrrolidone	S	S	S	S	202-204
Cyclohexanone	NS	PS	S	S	155.6
Ethylacetate	NS	S	S to PS	S	77.1
Ethylacetoacetate	NS	S	PS	S	180.8
Cyclohexane	NS	PS	PD	S	80.7
n-Decane	**NS**	**NS**	**NS**	**S**	**174.1**
n-Pentadecane	NS	NS	NS	PS	268-270
n-Hexadecane	**NS**	**PS**	**NS**	**S**	**287**
Dichloromethane	NS	S	n/a	n/a	39.6
Chloroform	S	S	n/a	n/a	62.1
Dimethylsulfoxide	S	S	S	S	189
Benzene	NS	S	PD	S	80.1
Toluene	PS	S	PD	S	110.6
Xylenes	**NS**	**S**	**NS**	**S**	**137**

*S – soluble, PS – partially soluble, NS – not soluble, PD – partially decomposed.

Based upon their favorable thermal properties, capsules from IST Run-3 and IST Run-6 were used to make small paste batches, as described in *Section 2.2.2*. Samples were allowed to degas overnight and then tested for time to cure at 60^{0}C based upon viscosity measurements. Measurements were taken until the viscometer reached 100% of allowable torque. The IST Run-3 sample, which contained encapsulated monuron produced via in-situ polymerization of an epoxy shell on the crystal, cured after 28 hours and 25 minutes. The IST Run-6 sample, which in which the encapsulation of the monuron occurred within D.E.R. 331 cured after 29 hours and 5 minutes. The baseline sample, which contained neat monuron, cured after 40 hours and 5 minutes. These results can be seen in **Figure 33**.

Due to their poor thermal properties, in comparison with capsules made via the complex coacervation method and inferior stability, this procedure was deemed to be an unattractive method to encapsulate accelerators.

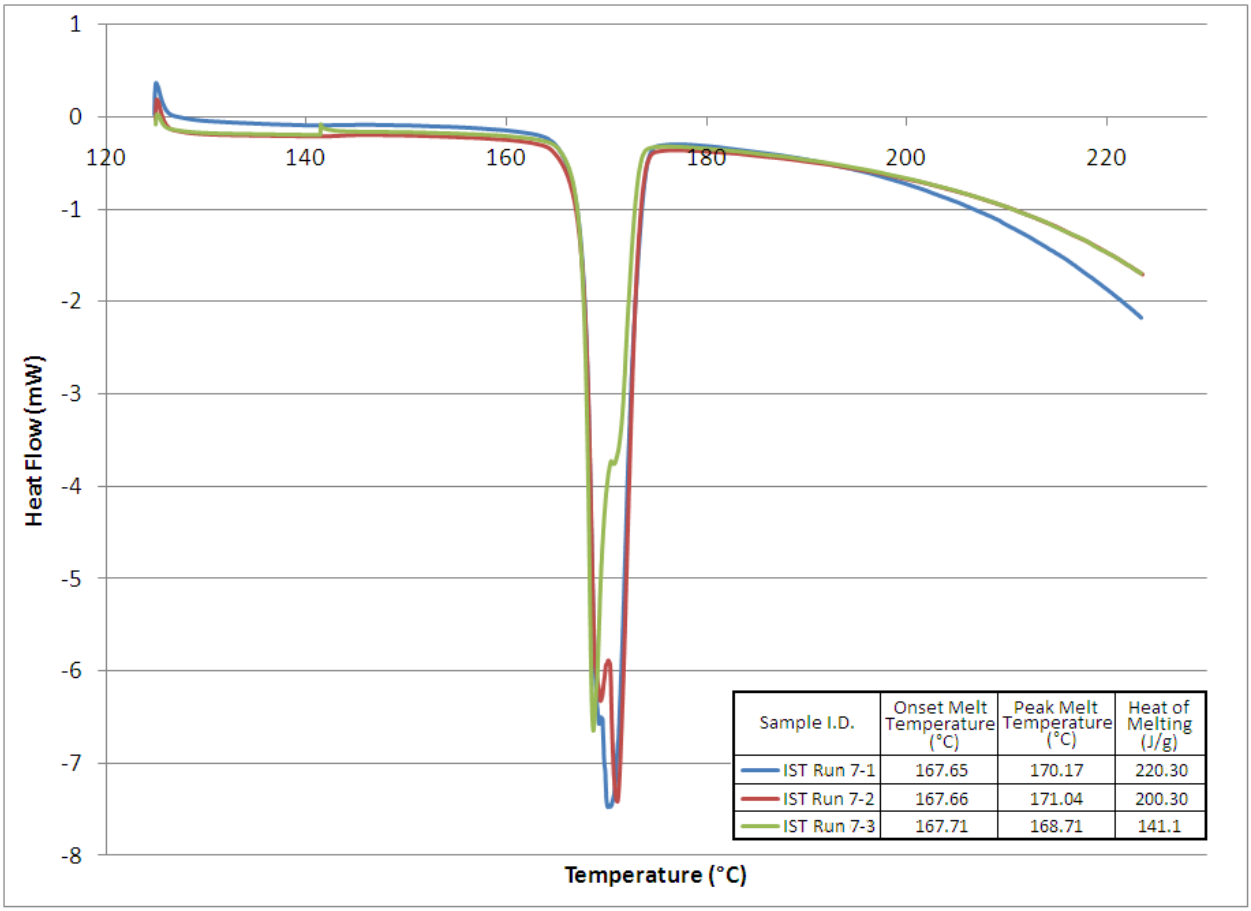


Figure 31. DSC data for IST Run-7, epoxy encapsulated monuron. Average onset of melt temperature = 167.67^{0}C, average peak melt temperature = 169.97^{0}C, average heat of melting = 187.23 J/g. Active monuron in sample (comparison of heat of melting of IST Run-7 to raw monuron) = 80.89%

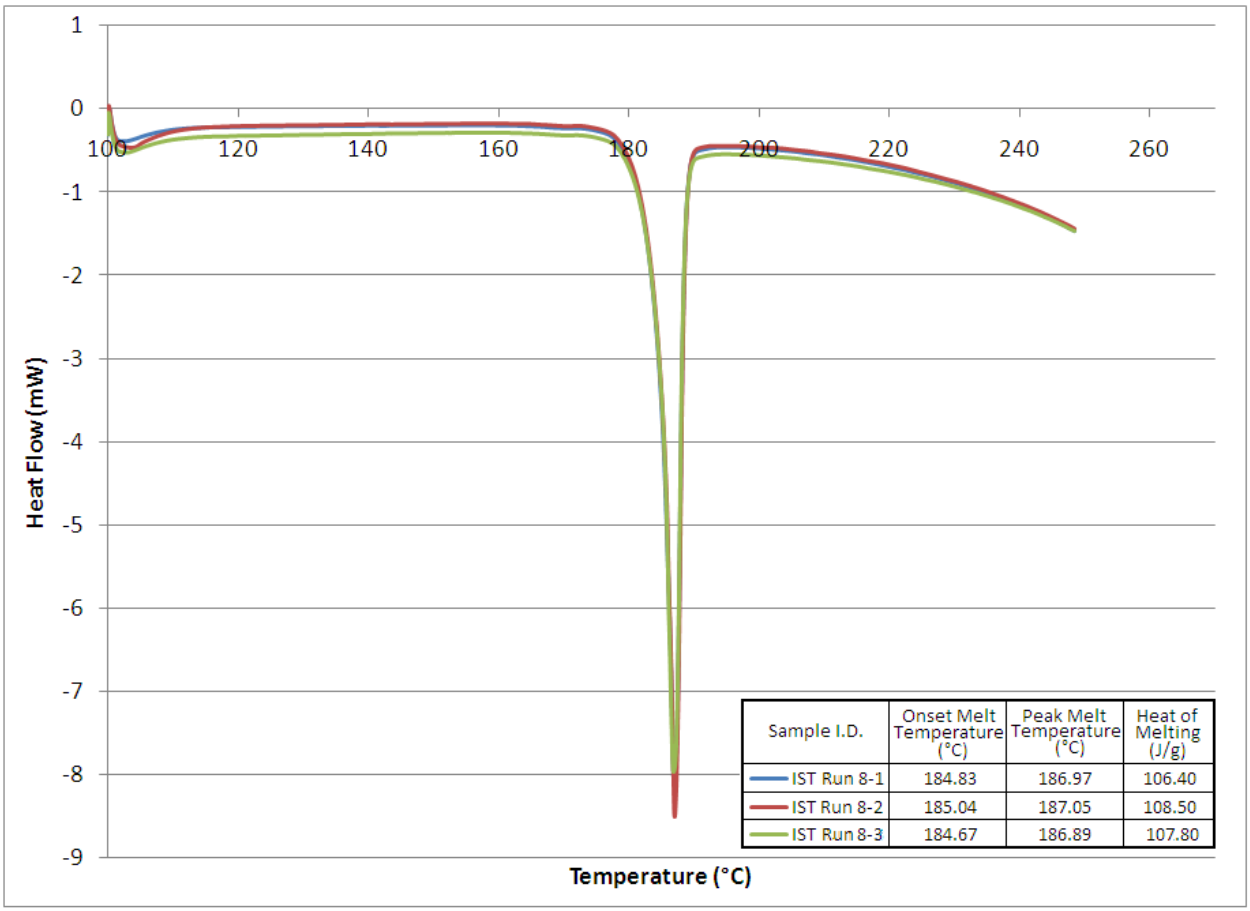


Figure 32. DSC data for IST Run-8, epoxy encapsulated monuron. Average onset of melt temperature = 184.85^{0}C, average peak melt temperature = 186.97^{0}C, average heat of melting = 107.57 J/g. Active monuron in sample (comparison of heat of melting of IST Run-7 to raw monuron) = 97.26%

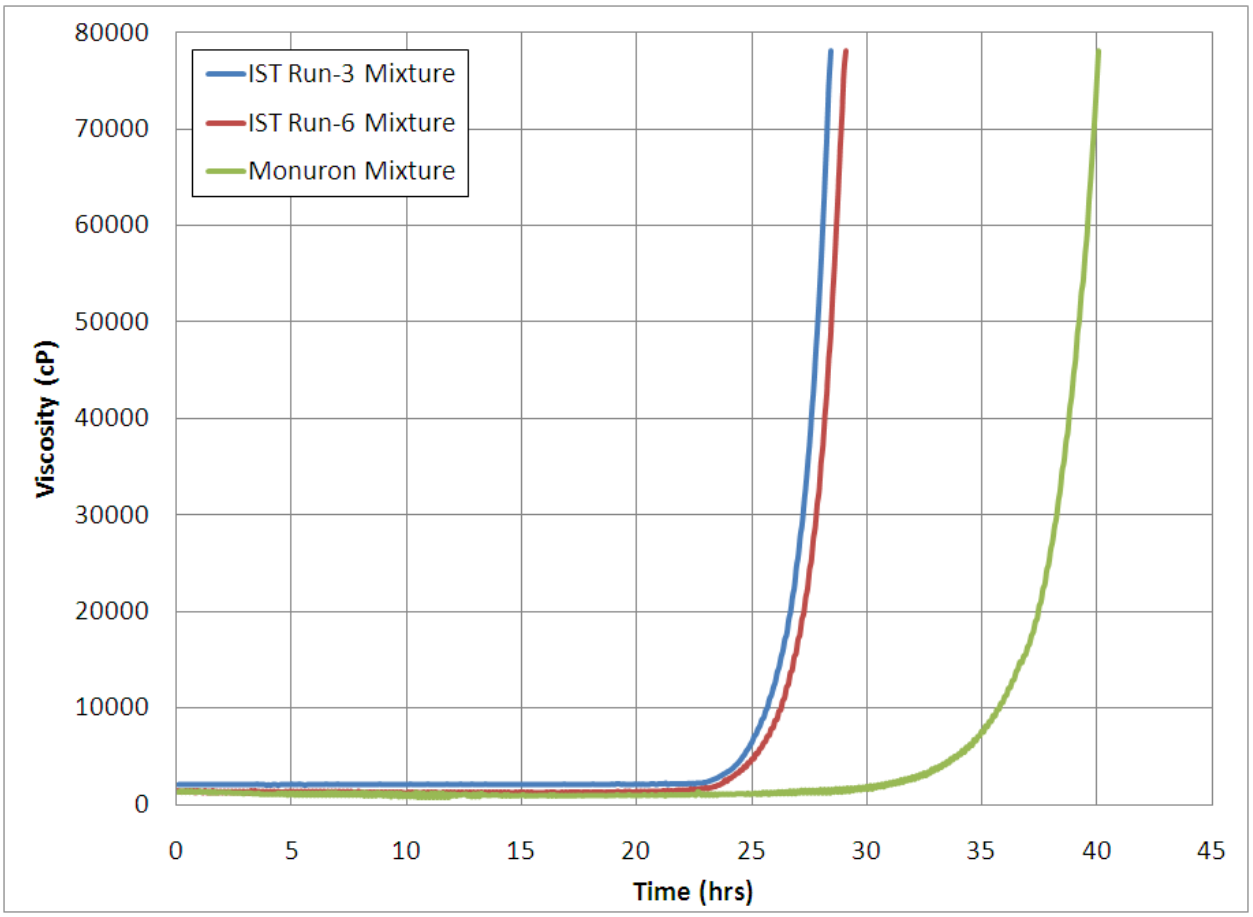


Figure 33. Viscosity measurements for IST Run-3, IST Run-6 and neat monuron baseline mixtures. The time to 100% torque for each sample was: 28 hrs 5 mins (IST Run-3), 29 hrs 5 mins (IST Run-6) and 40 hrs 5 mins (Baseline)

5.1.2 Encapsulation Using the Complex Coacervation Method

A total of 42 encapsulated samples were created using the complex coacervation method as described in *Section 2.2.1.3*. These samples are summarized in **Table 5**. There were 15 encapsulated monuron samples, of which, seven (7) had a gelatin-based shell, one sample had a gelatin + IC shell, seven (7) samples had a PDDACL + KC shell and one (1) sample had a PDDACL + IC shell. There were 27 encapsulated fenuron samples, of which two (2) had a PDDACL + KC shell and the remaining 25 had a gelatin derived shell. The thermal properties, cure properties, shelf-life and mechanical strength of samples made with these capsules are discussed in the subsequent sections.

Table 5. Summary of capsules made via complex coacervation

Accelerant	Sample I.D.	Shell Material	Yield (g)
Monuron	CT121110E	PDDACL + k-carragenan	25.9
	CT122910A	gelatin	25.0
	CT123010B	gelatin	29.4
	CT123110A	gelatin	33.6
	CT010511A	PDDACL + i-carragenan	28.3
	CT011211D	gelatin	25.3
	CT011411B	gelatin	25.4
	CT011211E repeat 1	PDDACL + k-carragenan	25.0
	CT011211E repeat 2	PDDACL + k-carragenan	25.0
	CT011211E repeat 3 - under 63 um	PDDACL + k-carragenan	22.0
	CT011211E repeat 3 - 63-106 um	PDDACL + k-carragenan	23.0
	CT011211E repeat 4 - under 63 um	PDDACL + k-carragenan	10.0
	CT011211E repeat 4 - 63-106 um	PDDACL + k-carragenan	32.4
	CT012612B	gelatin	20.0
	CT012612C	gelatin	23.0
Fenuron	CT112211B	gelatin	27.8
	CT121911A	k-carragenan	25.0
	CT121911B	k-carragenan	19.0
	CT123011B	gelatin	25.0
	CT010512A	gelatin	10.3
	CT010512B	gelatin	20.5
	CT012612A1	gelatin	25.0
	CT012612A2	gelatin	25.4
	CT012612A3	gelatin	27.0
	CT060512 - jar 1	gelatin	41.0
	CT060512 - jar 2	gelatin	34.0
	CT022612B2	gelatin	26.0
	JCT071412A	gelatin	55.0
	JCT071412B	gelatin	55.0
	JCT071412C	gelatin	20.0
	CT082612-1	gelatin	43.0
	CT082612-2	gelatin	45.0
	CT082612-3	gelatin	50.0
	CT1-0013-111	gelatin	26.0
	CT1-0013-112	gelatin	25.0
	CT1-0013-113	gelatin	24.0
	CT1-0013-114	gelatin	26.0
	CT1-0013-115	gelatin	75.0
	CT1-0013-116	gelatin	75.0
	CT1-0013-117	gelatin	75.0
	CT1-0013-118	gelatin	75.0
	CT1-0013-119	gelatin	75.0

5.2 Film Processing

Development of a film process and specimen layup procedure was required to effectively evaluate adhesive performance. Lap shear mechanical strength data was used as the primary metric to validate the consistency and reliability of the processing methods. The second phase of testing used optimized preparation methods for consistent specimens and films to compare different film compositions. The results of the process development and composition comparison are described in the following subsections.

Several different parameters were varied to determine the optimal film processing and lap shear specimen production process:

- Resin solid to liquid ratio (D.E.R. 331 and D.E.R. 661, respectively)
- Film thickness
- Film post processing
- Cure atmosphere
- Specimen compression
- Specimen immobilization

The cure conditions were not varied. Each sample was cured according to the established protocol: from room temperature the temperature was increased at 36^0F/min (2^0C/min) to 284^0F (140^0C) and held for two hours. A summary of the film samples fabricated to date is given in **Table 6**.

Table 6. Summary of films prepared

Film Designation	Resin ratio (liq/solid)	Accelerator	Thickness (inches)	Film Product Description
058_102	40/60	Neat Monuron	0.008	Excellent wetting, some bubbles, good tack
058_103	45/55	Neat Monuron	0.008	Excellent wetting, acceptable uniformity, excessive tack
058_103	45/55	Neat Monuron	0.008	Excellent wetting, acceptable uniformity, excessive tack
058_104	50/50	Neat Monuron	0.008	Excellent wetting, acceptable uniformity, excessive tack
058_105	55/45	Neat Monuron	0.008	Excellent wetting, acceptable uniformity, excessive tack
058_106	60/40	Neat Monuron	0.008	Excellent wetting, acceptable uniformity, excessive tack
058_107	40/60	CT122910A	0.008	Excellent wetting, good uniformity, good tack
058_108	50/50	CT122910A	0.008	Excellent wetting, good uniformity, good tack
005_128_1	40/60	Neat Monuron	0.012	Excellent wetting, good uniformity, good tack
005_128_2	40/60	CT011411B	0.012	Excellent wetting, good uniformity, good tack
005_128_3	40/60	Neat Monuron	0.016	Excellent wetting, good uniformity, good tack
005_128_4	40/60	CT011411B	0.016	Excellent wetting, good uniformity, good tack
005_142	40/60	Neat Fenuron	0.016	Acceptable wetting, some bubbles, good tack
005_140	40/60	CT011211E-R1	0.016	Excellent wetting, good uniformity, good tack
058_112	40/60	CT011211D	0.016	Acceptable wetting, good uniformity, good tack

As an example of the film product, film 005_142 is shown in **Figure 34**. It was determined from the level of tack and the acceptable wetting properties that the 40/60 liquid-solid epoxy blend would be used as the standard formulation. This resin ratio was used in all formulations moving forward.

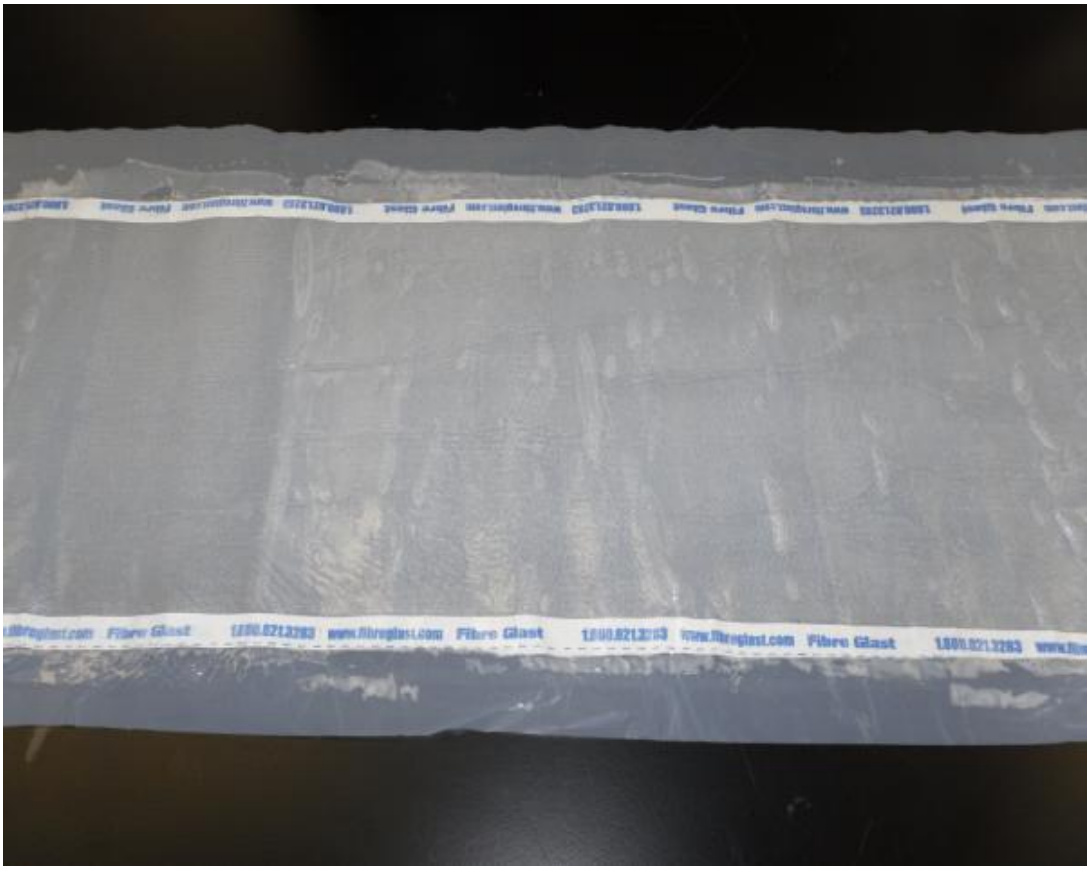


Figure 34. Photograph of film 005_142

5.3 Adhesive Characterization

5.3.1 DSC Analysis

Analysis showed that the accelerant within the capsules behaved similarly to the neat accelerant as evidenced by the sharp endothermic melt peak lying in the graphs seen in **Figure 35.** The peak melt temperature of the encapsulated materials is slightly lower than that of the neat materials but this difference was deemed to be inconsequential with regards to the cure effectiveness within an epoxy resin. The weight fraction of the microcapsules corresponding to the active accelerant content can be calculated by assuming the enthalpy of melting (ΔH) is proportional to the amount of accelerator. The results of this testing are shown in **Table 7**. Plots of the DSC results for all batches of monuron and fenuron capsules can be found in **Appendix 2A** and **Appendix 2B**, respectively. The peak temperatures did not vary drastically between the neat accelerants and the same encapsulated accelerants. Therefore, it was possible to calculate the amount of active accelerant in each batch of capsules by comparing the enthalpy value for SSA samples to baseline values.

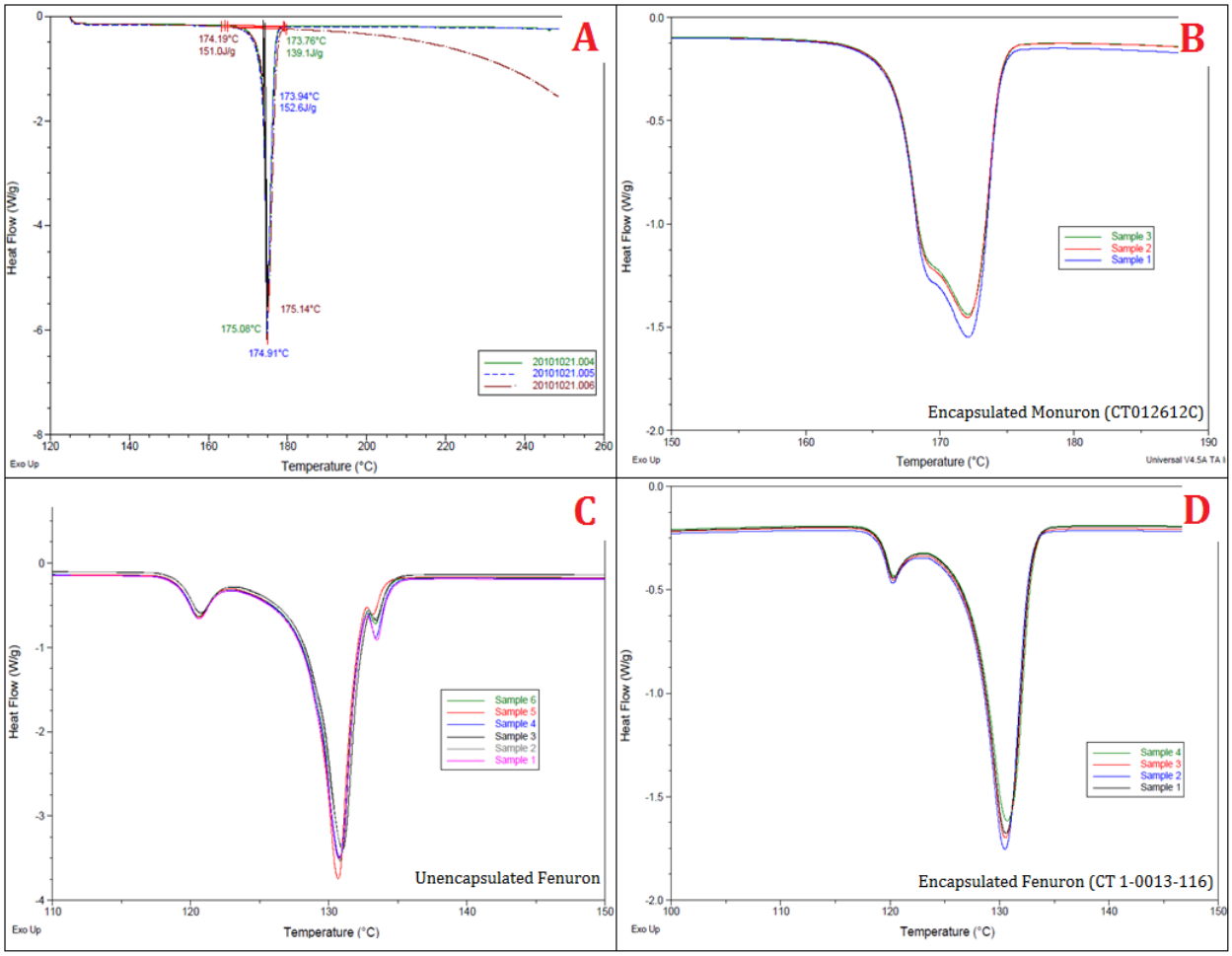


Figure 35. DSC exotherms for A) neat monuron, B) encapsulated monuron, C) neat fenuron and D) encapsulated fenuron

Table 7. Summary of encapsulated materials (complex coacervation method)

		Raw Material				Hand-mixed Paste		
		Onset Temp (ºC)	Peak Temp (ºC)	Heat (ºC)	% Active	Onset Temp (ºC)	Peak Temp (ºC)	Heat (ºC)
Monuron	Neat Monuron	167.90	175.04	144.97	100.00%	133.75	140.67	351.30
	CT121110E	165.86	169.61	93.16	64.26%	143.32	154.86	337.53
	CT122910A	166.86	172.29	98.68	68.07%	*Not tested*		
	CT123010B	167.10	169.05	92.58	63.86%	*Not tested*		
	CT123110A	167.41	168.94	98.56	67.99%	*Not tested*		
	CT010511A	166.25	168.99	90.41	62.36%	*Not tested*		
	CT011211D	167.12	169.61	89.37	61.65%	*Not tested*		
	CT011411B	165.99	169.09	95.04	65.56%	*Not tested*		
	CT011211E repeat 1	165.97	170.24	107.00	73.81%	133.68	142.09	347.90
	CT011211E repeat 2	165.88	169.16	120.30	82.98%	133.67	141.17	310.77
	CT011211E repeat 3 - under 63 um	164.86	169.10	94.45	65.15%	135.29	142.57	333.50
	CT011211E repeat 3 - 63-106 um	164.65	168.86	95.87	66.13%	137.08	143.24	357.53
	CT011211E repeat 4 - under 63 um	164.76	168.93	97.95	67.57%	137.85	146.07	325.03
	CT011211E repeat 4 - 63-106 um	164.70	168.93	99.19	68.42%	136.93	144.58	323.00
	CT012612B	166.16	172.00	91.33	63.00%	157.51	168.78	275.97
	CT012612C	166.59	172.07	95.33	65.76%	*Not tested*		
Monuron	IST Run #2	173.05	174.25	159.00	1.10	*Not tested*		
	IST Run #3	173.17	174.49	142.10	0.98	*Not tested*		
	IST Run #6 (unwashed)	173.10	174.38	117.27	0.81	*Not tested*		
	IST Run #6 (washed)	173.13	174.73	144.40	1.00	*Not tested*		
	IST Run #7	167.67	169.97	187.23	1.29	*Not tested*		
	IST Run #8	184.85	186.97	107.57	0.74	*Not tested*		
Fenuron	Neat Fenuron	127.56	129.81	151.17	100.00%	134.69	140.20	359.97
	CT112211B	126.73	130.86	82.45	54.54%	153.53	157.44	290.73
	CT120311B	125.42	129.82	75.96	50.25%	*Not Tested*		
	CT121911A	128.77	131.74	88.76	58.72%	150.36	154.14	346.33
	CT121911B	128.71	131.52	76.73	50.76%	139.68	145.77	329.87
	CT010512A	128.67	131.24	57.09	37.77%	*Not Tested*		
	CT010512B	124.55	129.56	54.55	36.09%	*Not Tested*		
	CT012612A1	126.36	130.90	93.06	61.56%	155.52	158.88	348.60
	CT012612A2	126.25	130.76	96.47	63.82%	157.70	160.96	367.03
	CT012612A3	126.47	130.67	89.07	58.92%	159.73	162.69	337.57
	CT060512 - jar 1	125.87	130.88	63.61	42.08%	*Not Tested*		
	CT060512 - jar 2	125.74	130.74	65.97	43.64%	*Not Tested*		
	CT022612B2	125.15	129.96	78.56	51.97%	*Not Tested*		
	JCT071412A	126.90	130.43	80.04	52.95%	*Not Tested*		
	JCT071412B	125.76	130.09	84.21	55.71%	*Not Tested*		
	JCT071412C	119.20	128.41	61.58	40.74%	*Not Tested*		
	CT082612-1	127.07	130.90	95.85	63.40%	*Not Tested*		
	CT082612-2	128.14	131.19	49.17	32.53%	*Not Tested*		
	CT082612-3	126.89	130.57	92.79	61.38%	*Not Tested*		
	CT1-0013-111	127.76	130.58	83.69	55.36%	*Not Tested*		
	CT1-0013-112	127.93	130.42	76.84	50.83%	*Not Tested*		
	CT1-0013-113	126.43	129.98	81.48	53.90%	*Not Tested*		
	CT1-0013-114	126.67	130.10	74.82	49.50%	*Not Tested*		
	CT1-0013-115	126.95	130.82	84.75	56.06%	*Not Tested*		
	CT1-0013-116	127.16	130.58	83.88	55.49%	*Not Tested*		
	CT1-0013-117	126.64	130.73	83.25	55.07%	*Not Tested*		
	CT1-0013-118	126.73	130.89	83.73	55.39%	*Not Tested*		

5.3.2 Viscosity Analysis Results

Based upon the accelerant content as determined via DSC analysis, small-scale paste adhesive samples of 49 different batches of samples and two baselines were analyzed for the time to initiate cure. Results for these samples can be seen in **Table 8**. Viscosity curves can be found in **Appendix 3A** (monuron based samples) and **Appendix 3B** (fenuron based samples). All samples had to have a time to cure longer than 100 hours to be considered "promising." Samples in **Table 8** marked with an asterisk (*) were deemed to be very stable and stopped before the onset of cure.

Table 8. Summary of viscosity results (time to cure at 60°C) of all paste samples prepared with Fenuron accelerant. Samples marked with an (*) were stopped before full cure was attained due to project time constraints.

Sample I.D.	Shell Material	Time to cure (hrs)	
Neat Fenuron	N/A	35.7	
CT112211B	gelatin	267.7	*
CT120311B	k-carragenan	50.5	
CT121911A	k-carragenan	336.3	
CT121911B	gelatin	54.3	
CT010512A	gelatin	47.1	
CT010512B	gelatin	37.2	
CT012612A1	gelatin	53.2	
CT012612A2	gelatin	232.8	*
CT012612A3	gelatin	166.4	
JCT071412A	gelatin	33.8	
JCT071412B	gelatin	36.3	
CT1-0013-111	gelatin	555.3	*
CT1-0013-112	gelatin	635.3	*
CT1-0013-113	gelatin	600.0	*
CT1-0013-114	gelatin	600.0	*
CT1-0013-115	gelatin	654.9	*
CT1-0013-116	gelatin	600.0	*
CT1-0013-117	gelatin	600.0	*
CT1-0013-118	gelatin	600.0	*
CT1-0013-119	gelatin	600.0	*

5.3.3 Short-term Storage Analysis

Based upon high active accelerant content, demonstrated long shelf stability as measured by viscosity measurements, and high single-lap-joint shear strength, two batches of capsules (CT012612A3 and CT-0013-115) were down-selected as the best performing SSA candidates and used for storage analysis testing. Film samples of both the baseline and the SSA films were held at 60^{0}C for various lengths of time and the residual strength was measured via single lap shear. Results for these tests can be seen in **Figure 36**. The SSA samples exhibited superior mechanical strength over the baseline sample: the baseline sample lost all mechanical strength within 20 hours of exposure while the SSA samples showed no discernable decline in mechanical strength after 100 hours of exposure.

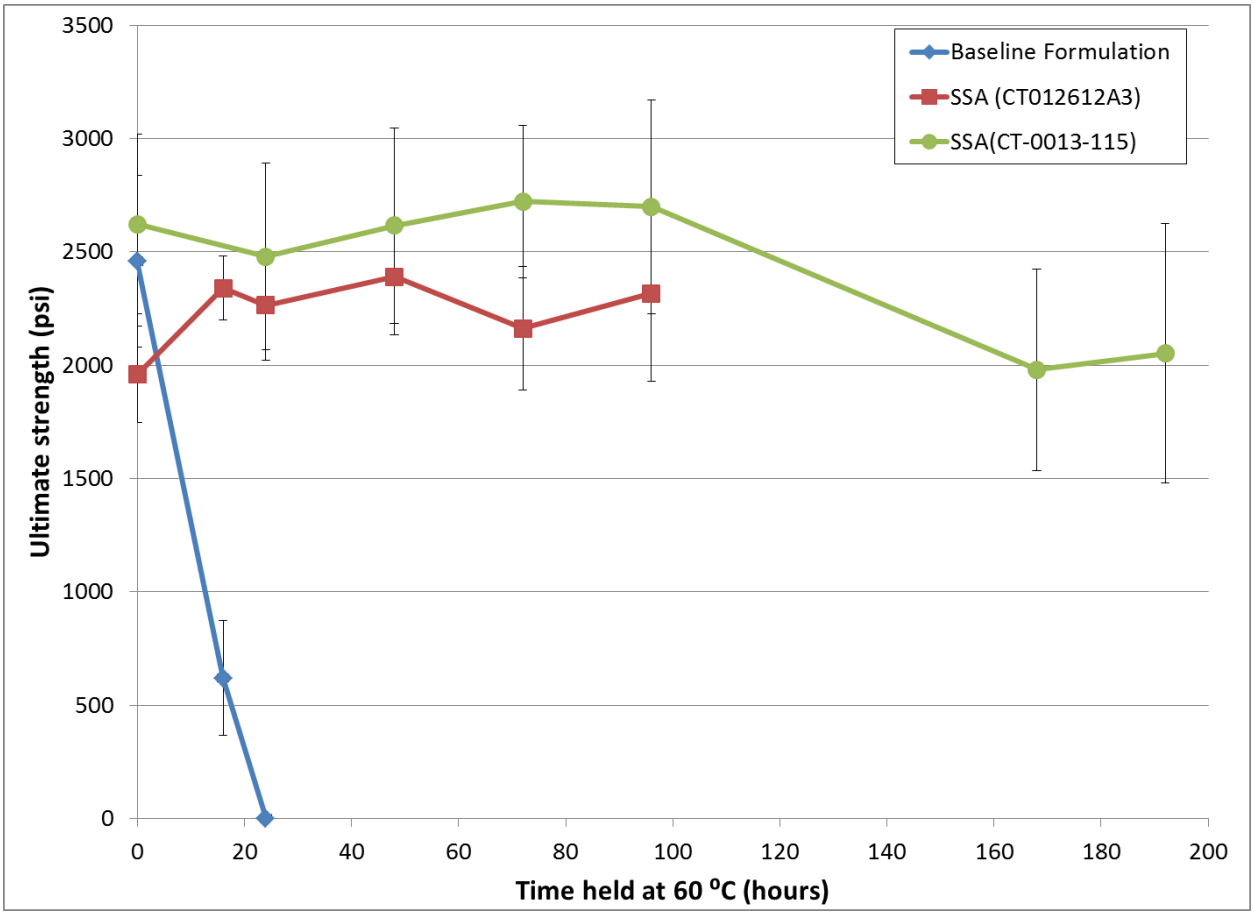


Figure 36. Residual mechanical strength after short term storage exposure to 60^{0}C for baseline and SSA film samples

5.3.4 Long-term Storage Analysis

Based upon high active accelerant content, demonstrated long shelf stability as measured by viscosity measurements, and high single-lap-joint shear strength, one batch of capsules (CT012612A3) was down-selected as the best performing SSA candidate. Due to limited quantity of this initial batch, a batch with similar physical characteristics (CT012612A1) was used for this experiment. Film samples of both the baseline and the SSA film (CT012612A1)

were stored at both ambient conditions and 90^{0}F (32^{0}F) for various lengths of time in controlled environments and the residual mechanical strength (single lap shear) was measured. Results for these tests can be seen in **Figure 37**. The SSA sample exhibited superior mechanical strength over the baseline sample at the elevated temperature. The baseline sample lost all mechanical strength after only 6 months of exposure while the SSA sample showed a slight decline in mechanical strength after one year of storage at 90^{0}F (32^{0}C). At ambient conditions, both sets of films appear to have no dramatic loss in single-lap-joint strength over the duration of the long-term storage test.

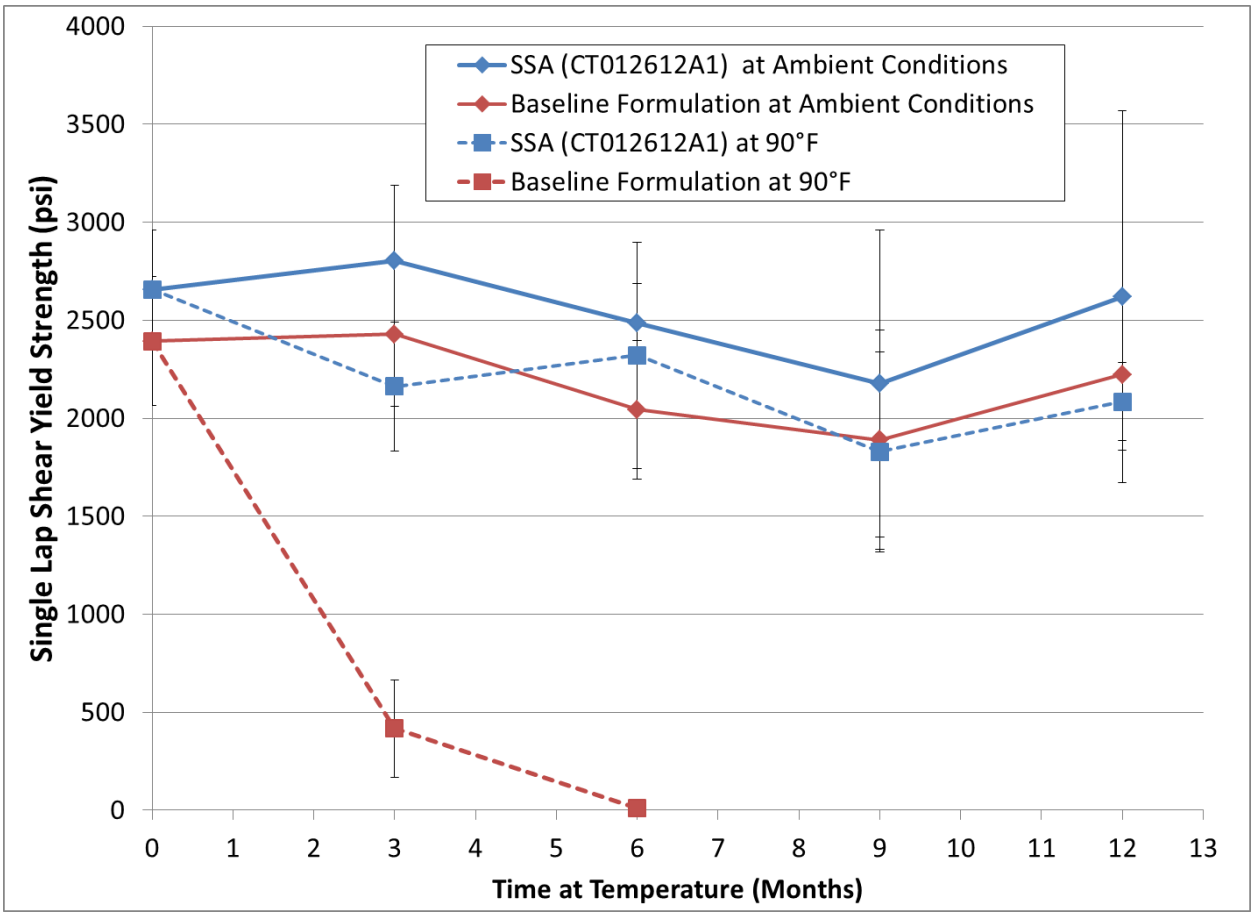


Figure 37. Residual mechanical strength after long term storage exposure to both ambient conditions and 90^{0}F (32^{0}C) for baseline and SSA film samples

5.4 Mechanical Testing

5.4.1 Mechanical Screening

Batches of capsules that were considered to have desirable shelf-stability as determined via DSC analysis (*Section 3.3.1*) and viscosity analysis (*Section 3.3.2*) were made into films for mechanical screening. The shear strength of single-lap-joint of 28 samples was evaluated over the course of the project. These results can be found in **Table 9**. Overall, the fenuron samples demonstrated higher single-lap-joint strength than the monuron samples. Additionally several

films made from the encapsulated accelerant materials showed comparable strength to those created using neat accelerants.

Table 9. Summary of single-lap-joint strength of all films tested.

		Average Load at Break (psi)	Std. Dev
Monuron	Neat Monuron	2192.50	± 388.75
	CT121110E	1320.46	± 100.22
	CT011211D	326.14	± 48.74
	CT011411B	1790.76	± 259.93
	CT011211E repeat 1	2151.87	± 97.64
	CT012612B	335.73	± 61.86
Fenuron	Neat Fenuron	2422.37	± 351.77
	CT112211B	400.97	± 144.05
	CT121911A	1585.84	± 1139.72
	CT121911B	2244.40	± 270.47
	CT012612A1	1225.07	± 1068.94
	CT012612A2	481.88	± 99.72
	CT012612A3	1662.84	± 614.49
	CT060512 - jar 1	2563.41	± 224.04
	CT022612B2	2576.34	± 242.06
	JCT071412A	2831.19	± 216.06
	JCT071412B	2412.18	± 268.36
	JCT071412C	2660.82	± 354.65
	CT082612-2	2447.83	± 301.41
	CT1-0013-111	2518.29	± 567.94
	CT1-0013-112	2653.71	± 469.14
	CT1-0013-113	2420.97	± 360.09
	CT1-0013-114	2917.18	± 311.29
	CT1-0013-115	2483.91	± 425.02
	CT1-0013-116	2572.67	± 259.71
	CT1-0013-117	2665.23	± 449.73
	CT1-0013-118	2497.48	± 272.77
	CT1-0013-119	2659.77	± 188.00

5.4.2 Mechanical Performance Testing

5.4.2.1 Selection of Shelf-Stable Adhesive Candidate

The single-lap-joint strength and shelf-stability via viscosity of all batches of encapsulated candidates were compared in **Figure 38**. A target performance zone was defined as any candidate encapsulated formulation that demonstrated both:

1. Minimum lap-joint strength of approximately 1600 psi
2. Minimum shelf-life, as determined via viscosity analysis of 250 hours

As seen on the graph, the batch of capsules that was selected for long-term storage analysis (CT0126122A3) does not fall within the target performance zone; however, at the time of the selection for that particular test, this batch of capsules was the most promising. To improve performance of the samples, capsule fabrication procedures were optimized. The first of these optimized capsule batches, sample CT-0013-111, fell in the target zone. This batch was repeated three times (CT-0013-112, CT-0013-113 and CT-0013-114) to replicate results. When tested, all fell within the target performance zone. As seen in **Figure 38** these four batches all demonstrated a smaller range of variance with regards the single-lap-joint strength.

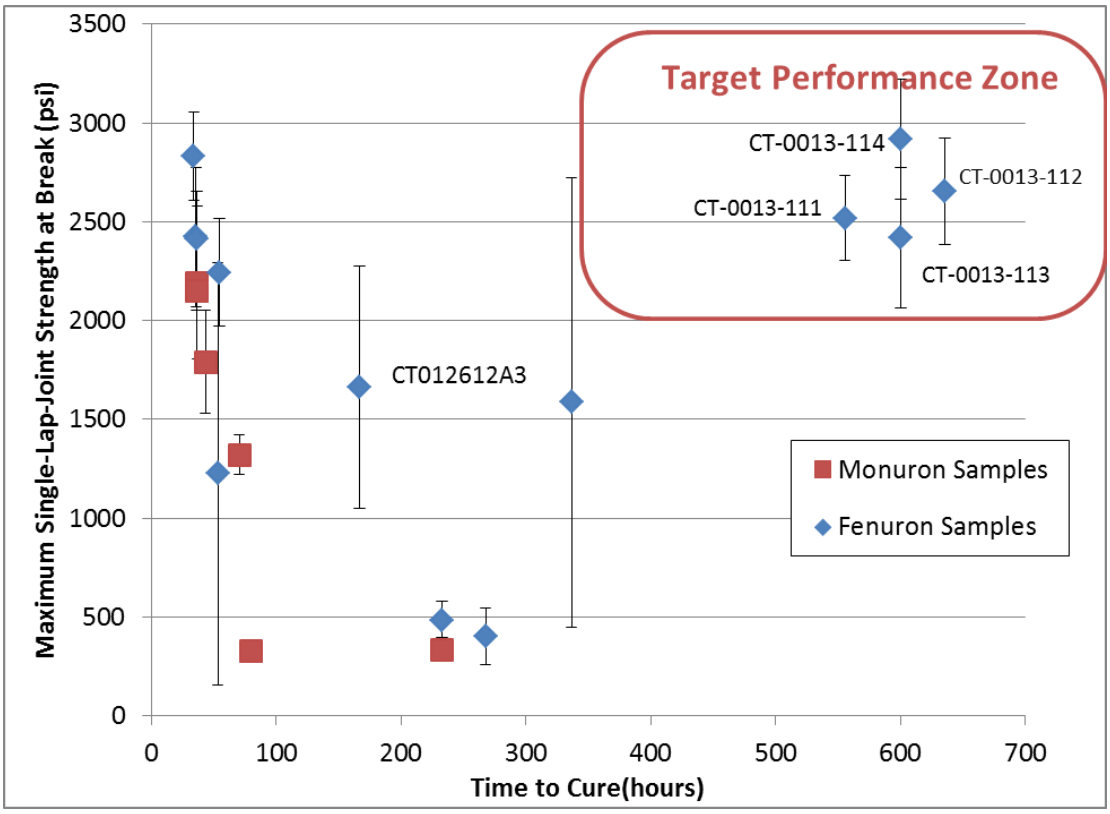


Figure 38. Comparison of performance of batches of capsules with regards to single-lap-joint strength and time to cure, as determined via viscosity analysis.

Production of these capsules was scaled-up from a batch size of roughly 25 g to 75 g. These new batches (CT-0013-115, CT-0013-116, CT-0013-117, CT-0013-118 and CT-0013-119) were then

screened for thermal properties via DSC, storage stability via DSC and single-lap-joint strength. All batches examined were within the target performance zone, as seen in **Figure 40**. Based upon the observed results, CT-0013-117 was selected as the material that would be used for validation testing. This batch of capsules demonstrated thermal and mechanical properties that were closest to the average values for the large batches of capsules.

Two (2) manufacturing goals were demonstrated with these batches: first, the ability to scale-up small (25 g) batches to larger (75 g) batches was demonstrated. Secondly, each batch of capsules demonstrated comparable performance to each other with regards to the metrics that were evaluated (thermal analysis, shelf-stability as measured via viscosity analysis and single-lap-joint strength).

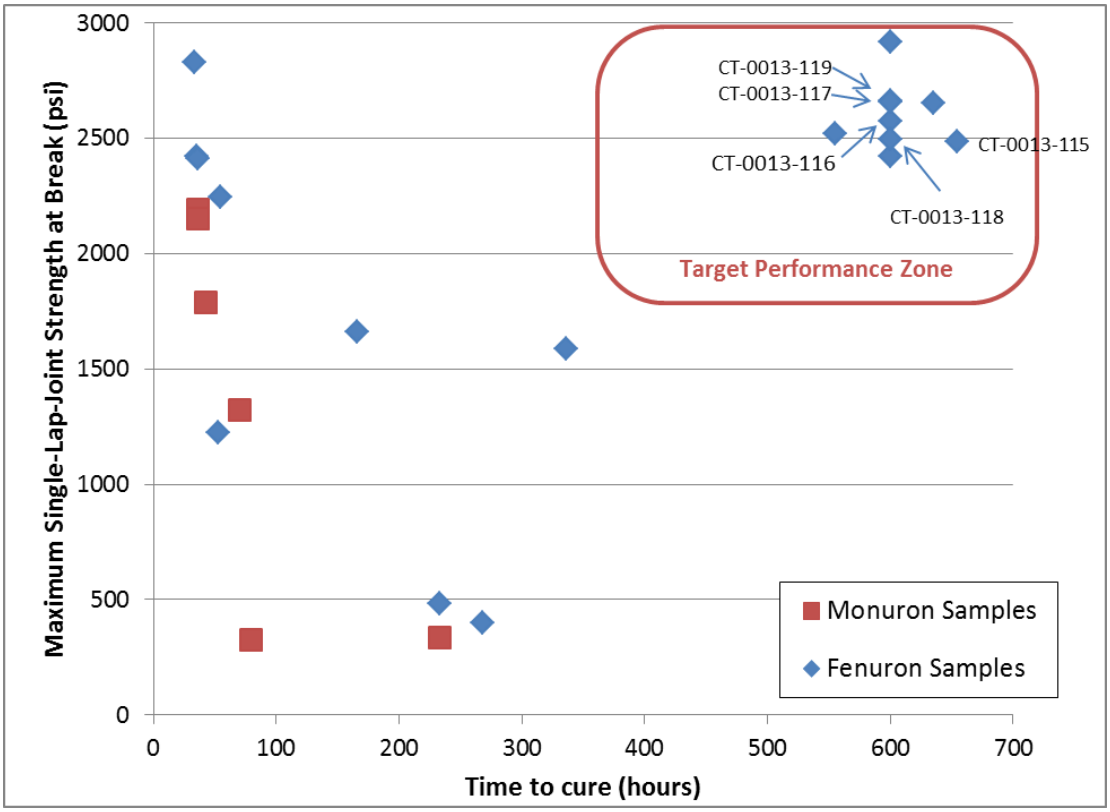


Figure 39. Comparison of performance of large batches of capsules with regards to single-lap-joint strength and time to cure, as determined via viscosity analysis.

5.4.2.2 Shear Strength of Single-Lap-Joint

Single-lap-joint samples were tested at several conditions: control, dry extreme temperature (high), dry extreme temperature (low), long term hot/wet testing, accelerated aging and high humidity accelerated aging. Results from Intertek for all samples tested can be found in **Appendix 4A**. A comparison between the average shear strength at break for both the baseline formulation and the SSA formulation (CT-0013-117) can be found in **Figure 40** as well as **Table 10**. The overwhelming mode of failure for both types of samples under all testing conditions was the adhesion of the film to the metal substrate. Overall, the SSA formulation demonstrated

higher shear strength than the baseline formulation for every testing condition. The mode of failure for all tests performed for both types of films tested was the adhesion to metal, except for the SSA films that were subjected to dry temperature extreme (high) - those samples demonstrated adhesion/cohesive failure mechanisms.

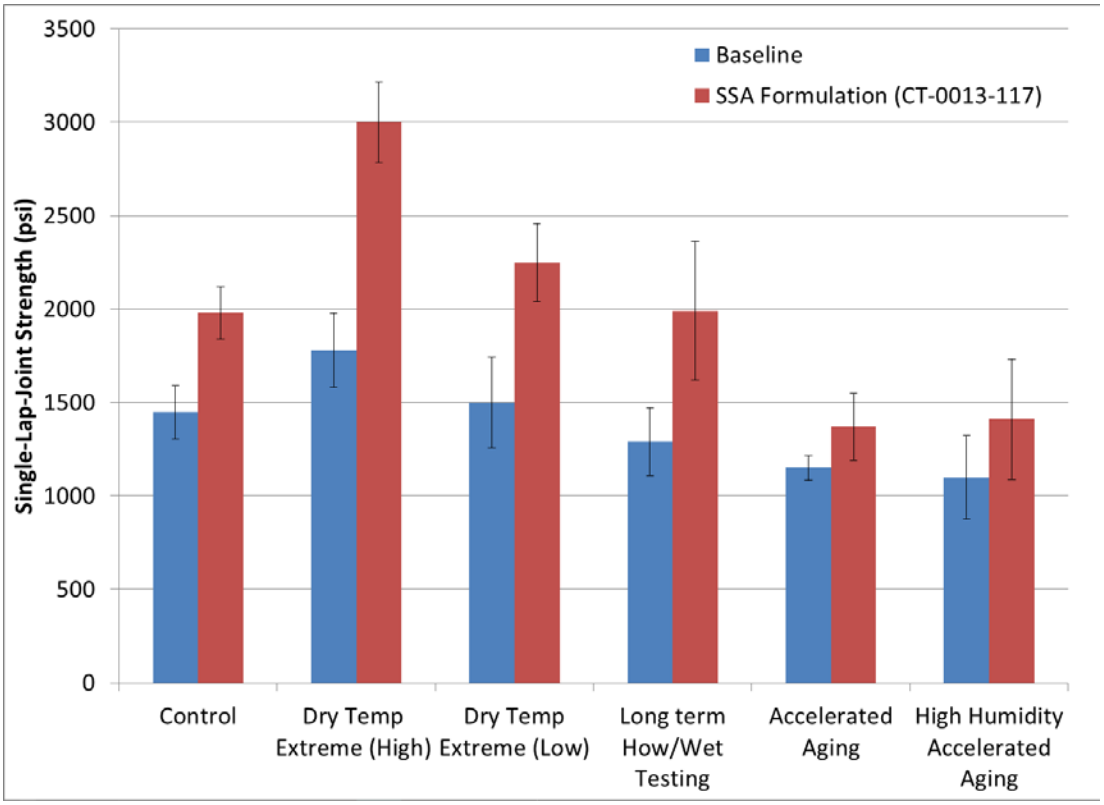


Figure 40. Comparison of the results from the single-lap-joint testing for both the baseline formulation and the SSA formulation (CT-0013-117).

Table 10. Shear strength of single-lap-shear results for both the baseline and SSA films.

Objective	Conditioning		Shear Strength of Single Lap Joint			
			ASTM D1002-05			
	Post-Cure Aging	Test Conditions	Shear Strength Reported in (psi)			
			Baseline	Std. Dev	SSA	Std. Dev
Control	none	ambient	1450 ±	141	1980 ±	140
Dry Extreme Temperature	none	-67°F	1502 ±	245	2253 ±	265
Dry Extreme Temperature	none	180°F	1784 ±	198	3001 ±	215
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	1290 ±	182	1990 ±	371
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	1150 ±	66	1370 ±	180
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	1101 ±	221	1412 ±	323

5.4.2.3 Strength Properties of Double Lap Shear Joints by Tension Loading

Double lap shear samples were tested under two conditions: control and high humidity accelerated aging. Results from Intertek for all samples tested can be found in **Appendix 4B**. A comparison between the average apparent shear strength at break for both the baseline formulation and the SSA formulation (CT-0013-117) can be found in **Figure 41** and **Table 11**.

As seen for the single-lap-joint samples, the overwhelming mode of failure for both types of samples under all testing conditions was the adhesion of the film to the metal substrate. There was no statistical difference between the baseline and the SSA formulations under control test conditions. The SSA formulation outperformed the baseline formulation under high humidity aging testing conditions, demonstrating a higher retention rate of residual strength after exposure to high temperatures (90^0F (32^0C)) and high levels of humidity (95% relative humidity) for 30 days.

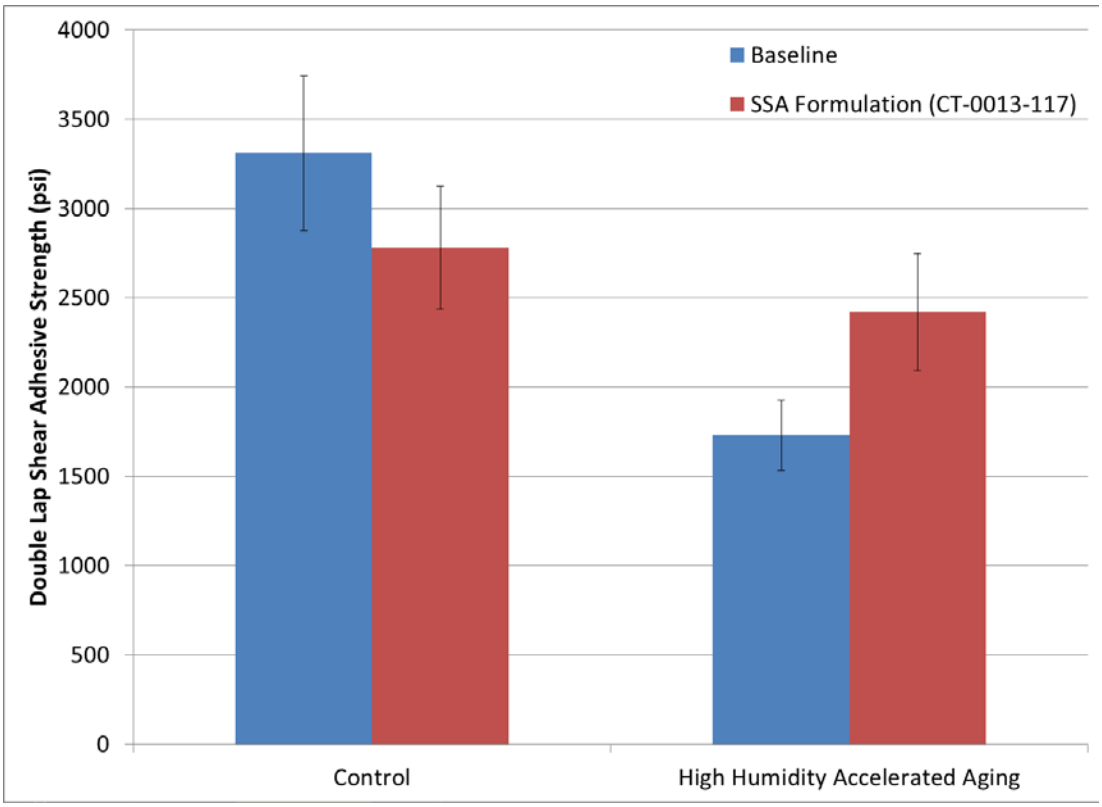


Figure 41. Comparison of the results from the double lap shear testing for both the baseline formulation and the SSA formulation (CT-0013-117).

Table 11. Strength properties of double lap shear adhesive joints by tension loading.

Objective	Conditioning		Shear Strength of Double Lap Joint ASTM D3528 Shear Strength Reported in (psi)			
	Post-Cure Aging	Test Conditions	Baseline	Std. Dev	SSA	Std. Dev
Control	none	ambient	2381 ±	407	2762 ±	323
Dry Extreme Temperature	none	-67°F	Not Evaluated			
Dry Extreme Temperature	none	180°F	Not Evaluated			
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	Not Evaluated			
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	Not Evaluated			
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	1728 ±	184	2419 ±	310

5.4.2.4 Climbing Drum Peel for Adhesives

Climbing drum peel tests were performed under three conditions: control, accelerated aging and high humidity accelerated aging. Results from Intertek for all samples tested can be found in

Appendix 4C. Due to limitation of SSA film material, only three (3) samples were submitted for each of the climbing drum peel tests, whereas nine (9) samples of the baseline formulation were submitted for each test. A comparison between the average peel torque for both the baseline and the SSA formulation (CT-0013-117) can be found in **Figure 42** and **Table 12**. The failure mode for both the baseline formulation and SSA samples tested under the control conditions was both a cohesive failure within the adhesive as well as a failure within the core material. The mode of failure for the baseline formulations samples under high humidity accelerated aging conditions was only adhesion to the facing whereas for the SSA samples, two failure modes were observed: cohesive failure within the adhesive as well as adhesion to the facing. As seen with the double lap shear results, the baseline formulation performed marginally better than the SSA formulation under control test conditions, however, the SSA formulation outperformed the baseline formulation under high humidity aging testing conditions, demonstrating a higher retention rate of residual strength after exposure to high temperatures (90^0F (32^0C)) and high levels of humidity (95% relative humidity) for 30 days.

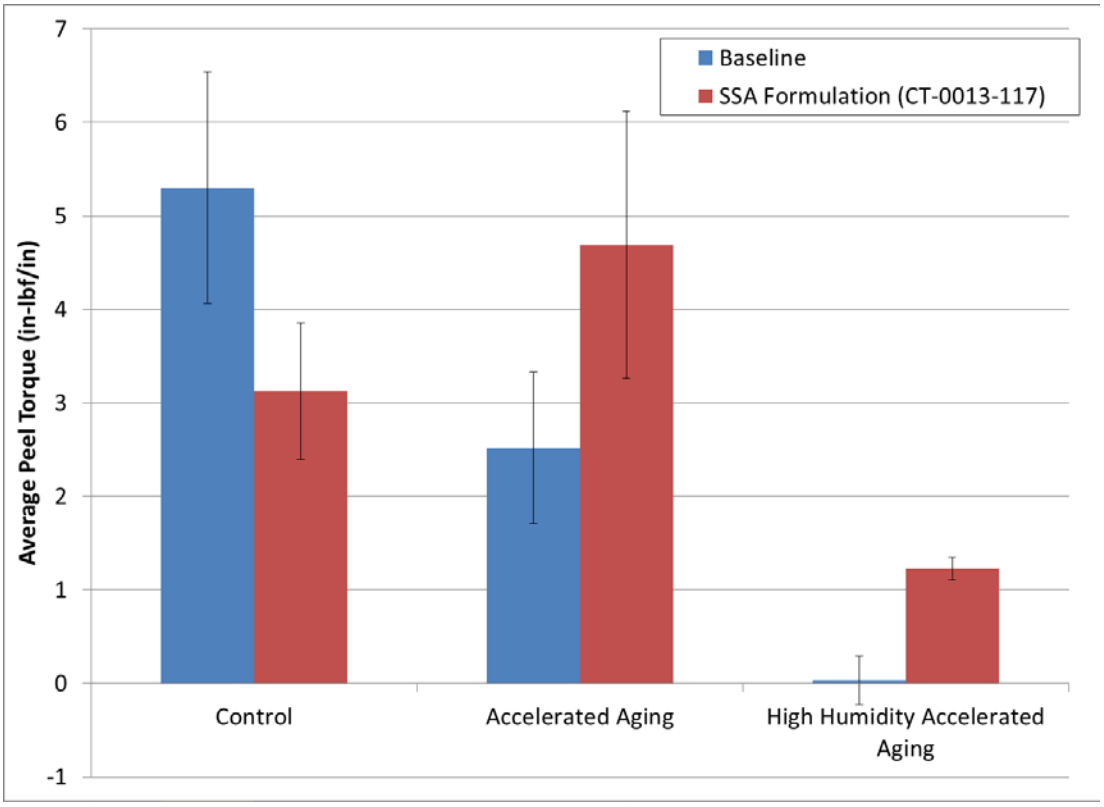


Figure 42. Comparison of the results from the climbing drum peel testing for both the baseline film and the SSA film (CT-0013-117).

Table 12. Climbing drum peel strength of both the baseline film and the SSA film (CT-0013-117)

Objective	Conditioning		Climbing Drum Peel					
			ASTM D1781					
	Post-Cure Aging	Test Conditions	Average Peel Torqure Reported in (in-lbf/in)					
			Baseline		Std. Dev	SSA		Std. Dev
Control	none	ambient	5.3	±	1.2	3.1	±	0.6
Dry Extreme Temperature	none	-67°F	Not Evaluated					
Dry Extreme Temperature	none	180°F	Not Evaluated					
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	2.4	±	0.9	1.8	±	0.1
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	2.5	±	0.8	4.7	±	1.4
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	0.0	±	0.2	1.2	±	0.1

5.4.2.5 Core Shear Properties of Sandwich Constructions

Core shear properties of the films were evaluated under three conditions: control, hot/wet and high humidity accelerated aging. Results from Intertek for all samples tested can be found in **Appendix 4D**. A comparison between the maximum facing stress for both the baseline and the SSA formulation (CT-0013-117) can be found in **Figure 43** and **Table 13**. The failure mode for both the baseline formulation and the SSA films was unknown under control testing conditions. The failure mode for both sets of films under high humidity accelerated aging conditions was due to skin to core delamination on the top facing. Overall, results showed that there was no discernable difference in performance for either film tested.

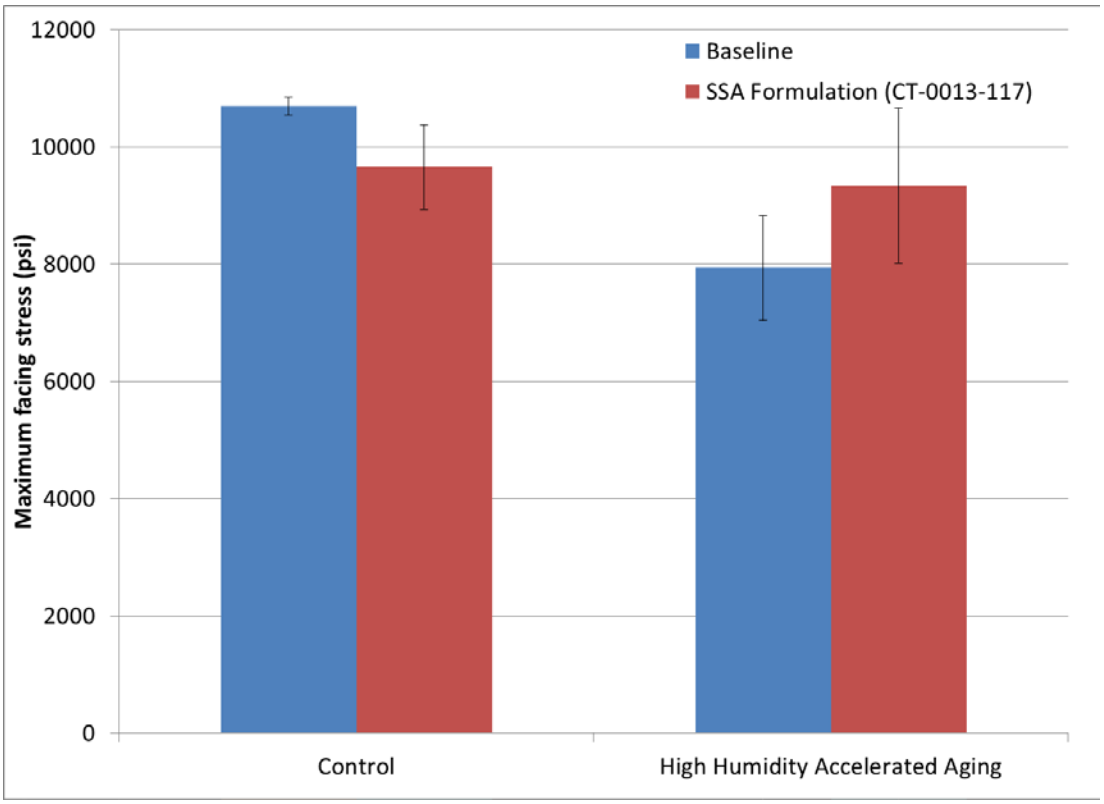


Figure 43. Comparison of the core shear maximum facing strength for both the baseline film and the SSA film (CT-0013-117).

Table 13. Core shear properties of sandwich samples for both the baseline and SSA (CT-0013-117) films.

Objective	Conditioning		Core Shear by Beam Flexure			
			ASTM C393			
	Post-Cure Aging	Test Conditions	Maximum Load Values Reported in (PSI)			
			Baseline	Std. Dev	SSA	Std. Dev
Control	none	ambient	10656	142	9963	± 674
Dry Extreme Temperature	none	-67°F	Not Evaluated			
Dry Extreme Temperature	none	180°F	Not Evaluated			
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	10478	± 162	10162	± 209
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	Not Evaluated			
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	7938	± 838	9340	± 1242

5.4.2.6 Flatwise Tensile Strength of Sandwich Constructions

Flatwise tensile samples were tested at several conditions: control, dry extreme temperature (high), dry extreme temperature (low), hot/wet testing, accelerated aging and high humidity accelerated aging. Results from Intertek for all samples tested can be found in **Appendix 4E**. A comparison between the flatwise tensile stress for both the baseline formulation and the SSA formulation (CT-0013-117) can be found in **Figure 44** and **Table 14**. Several modes of failure were noted under each of the tests performed. Both the baseline formulation and the SSA films

failed for several reasons under control testing conditions: cohesive failure, core failure and adhesive failure. The failure modes observed under the extreme temperature (low) test conditions were core/cohesive failure and bond to facing failure for both sets of films. The bond to facing failure is an observed failure of the bonded block to the facing of the sample and does not indicate a failure in the films tested, rather a failure in the strength of the commercially available adhesive that was used to bond test specimens to the block needed to be used in the testing rig. The overwhelming mode of failure for the baseline formulation under extreme temperature (high) testing conditions was bond to facing, while for the SSA film, the failure modes included bond to facing as well as core/cohesive failure. Finally, for the high humidity accelerated aging for both the baseline formulation and the SSA film, the observed mode of failure was an adhesive failure of the core-facing adhesive. According to the ASTM governing this test, all specimens tested are supposed to be tested at a speed that will produce failure in 3 to 6 minutes; however, all of the samples from the high humidity accelerated aging tests failed in less than 3 minutes.

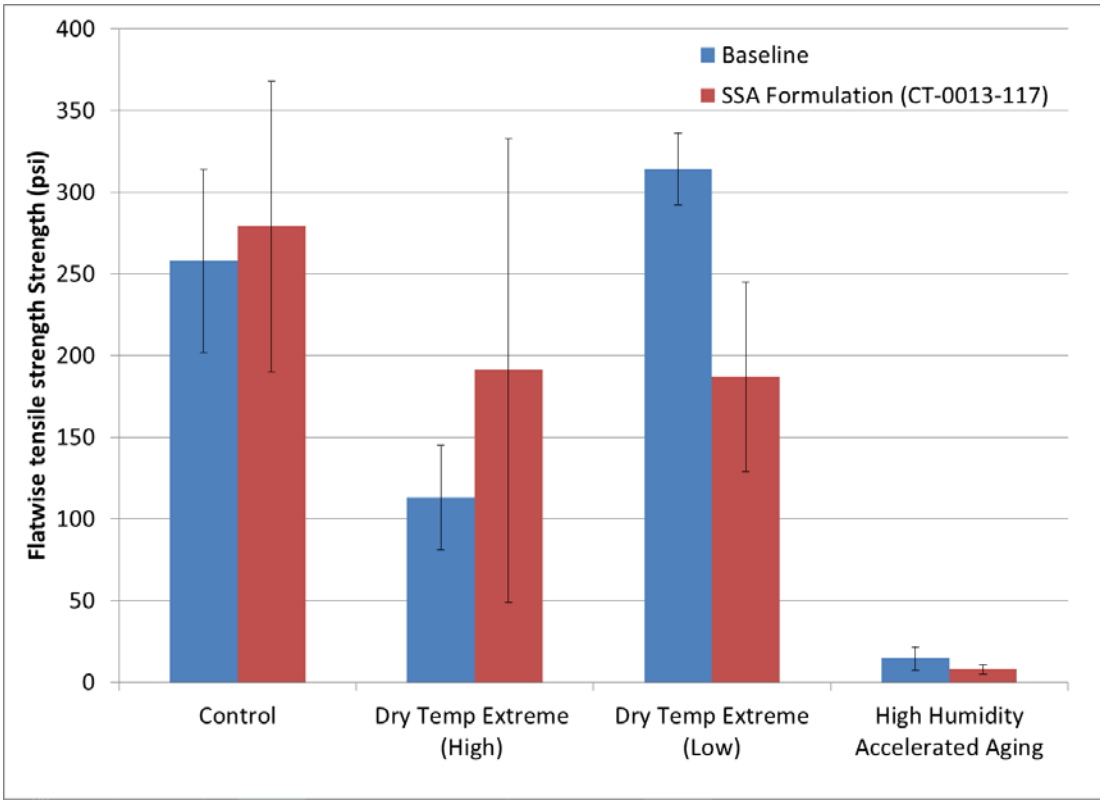


Figure 44. Comparison of the flatwise tensile strength for both the baseline formulation and the SSA formulation (CT-0013-117).

Table 14. Flatwise tensile strength of sandwich constructions of both the baseline and SSA (CT-0013-117) films.

Objective	Conditioning		Flatwise Tensile					
			ASTM C297					
	Post-Cure Aging	Test Conditions	latwise Tensile Strength Reported in (PS					
			Baseline		Std. Dev	SSA		Std. Dev
Control	none	ambient	259	±	52	274	±	80
Dry Extreme Temperature	none	-67°F	280	±	52	185	±	100
Dry Extreme Temperature	none	180°F	113	±	52	148	±	52
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	not evaluated					
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	not evaluated					
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	15	±	7	8	±	3

6. Conclusions

This program demonstrated the potential of a SSA film system for DoD repair operations. To facilitate rapid insertion into repair operations, IST worked with Henkel to develop the baseline formulation based on its Hysol EA9696 film adhesive. Incorporation of the stabilizing technology into an existing, approved product is anticipated to reduce the time to market.

The shelf stability of film adhesives was improved with the SSA system through modification of the accelerator package. This was accomplished through formulation of controlled release encapsulated accelerators into the one-part epoxy resin. The reactivity of the one-part epoxy SSA film is preserved for extended periods of time through the microencapsulation of the curing accelerant. A complex coacervation technique was developed to isolate the accelerant using an exclusive polymeric formulation for the encapsulant material, thus inhibiting the cure process until its intended use. No additional activation steps were required; the accelerant shell ruptures when exposed to the autoclave thermal energy. A baseline was formulated using the primary components of the Hysol EA 9696 resin system matrix and accelerator (fenuron). The experimental SSA material was formulated in a manner identical to the baseline; however, the only differentiating factor was that the fenuron accelerator was encapsulated in a controlled release shell.

The SSA has been demonstrated to retain at least 75% of its adhesive strength when stored at 90°F (32^{0}C) for a year. As seen in **Figure 45**, when compared to the baseline formulated with a neat accelerant, the stability of the SSA formulation offers a significant advantage. If stored in a freezer, it is anticipated that the SSA will be stable for well over two years.

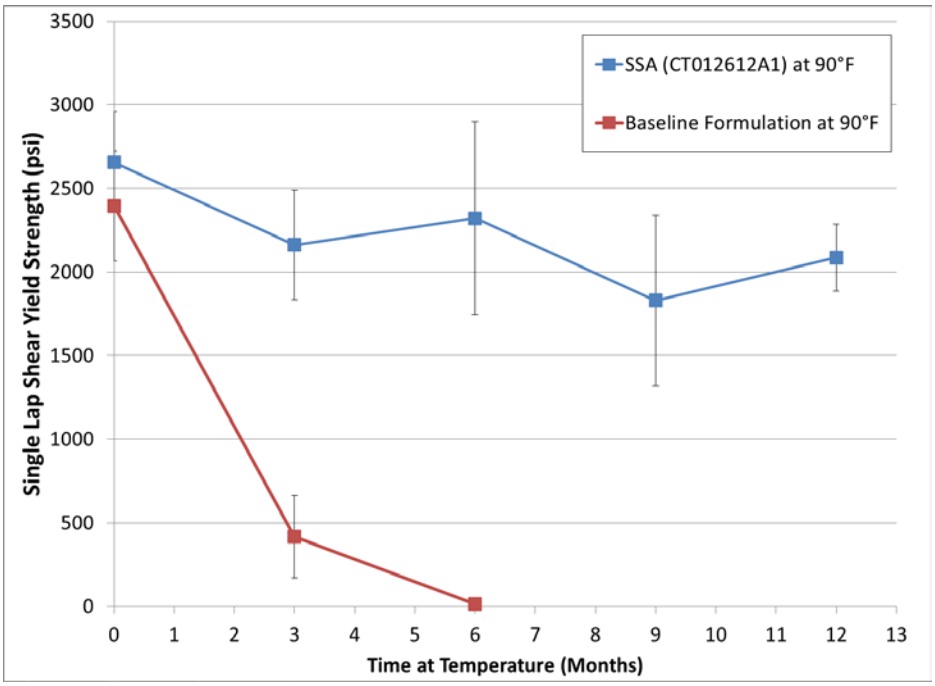


Figure 45. Graph of single lap shear tests (ASTM D1002-05) conducted on film samples stored side by side at 90°F for incremented time intervals and cured at 284^{0}F.

The mechanical properties of the SSA system were also assessed to verify that the encapsulant material would not adversely affect the adhesive performance during operational use. Tests were performed according to the AMS-A-2546 standard. Under this standard, the model epoxy resin system selected was classified as a Type I adhesive. Accordingly, performance metrics for the SSA film were determined based on Type I requirements. **Table 15** shows the test matrix performed on the SSA material relative to the baseline. These tests were conducted at Intertek Plastics Technology Laboratories (Pittsfield, MA). The 90-day environmental conditioning test confirmed that the capsules do not have a deleterious effect on the performance of the adhesive. In every test, the mechanical properties of the conditioned SSA samples were comparable or better than those of the baseline formulation.

Table 15. Test matrix for comparitive study between one-part epoxy baseline and SSA formulations. A 'Pass' indicates that the SSA formulation had comparable or better mechanical test results as the baseline.

Objective	Conditioning		ASTM D1002	ASTM D3528	ASTM D1781	ASTM C393	ASTM C297
	Post-Cure Aging	Test Conditions	Single Lap Shear	Double Lap Shear	Climbing Drum Peel	3-Point Sandwich Flex	Flatwise Tensile
Control	none	ambient	Pass	Pass	Pass	Pass	Pass
Dry Extreme Temperature	none	-67°F	Pass				Pass
Dry Extreme Temperature	none	180°F	Pass				Pass
Long Term Hot/Wet Testing	90 days @ 120°F, 50% RH	180°F	Pass				
Accelerated Aging	90 days @ 120°F, 50% RH	ambient	Pass		Pass		Pass
High Humidity Accelerated Aging	30 days @ 90°F, 95% RH	ambient	Pass	Pass	Pass	Pass	Pass

IST recommends two follow-on activities to push the SSA environmental technology towards commercialization. First, the encapsulated accelerator should be formulated and integrated into commercial film adhesive formulations. Mechanical testing of the SSA accelerators in the fully formulated film adhesive is a necessary conformational step before demonstration/validation trials on aircraft. In parallel with this, IST recommends scaling up the complex coacervation production process for the encapsulated accelerator. These validation studies will bring the SSA technology to a technology readiness level appropriate for repair operations testing and demonstration validation flight trials on military aircraft.

7. Literature Cited

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Appendix 1: Requirements Document

Shelf Stable Adhesive
Requirements Document

Version:	1.2
Print Date:	March 31, 2014
Release State:	Revised Draft
Approval State:	Draft
Approved by:	M. Cushman
Prepared by:	J. Belcher
Reviewed by:	N.A.
File Name:	WP-1763 Final Report - DRAFT - mc - CF

Document Change Control

Version	Date	Authors	Summary of Changes
1.0	5 June 11	M. Cushman	Original Draft
1.1	15 Aug 11	M. Cushman	Revised Draft to highlight adhesive type of Hysol EA 9696
1.2	31 Mar 14	J. Belcher	1) Revised Overview to reflect technology updates 2) Added additional reference document 3) Revised non-functional requirements

Document Sign-Off

Name (Position)	Signature	Date

1. Introduction

1.1 Purpose

This document details the requirements for materials under consideration for use as film adhesives with improved shelf stability. Such adhesives are intended for use in composite repair.

1.2 Overview

Conventional repair operations for composites used in military applications results in the generation of significant amounts of hazardous waste from unused expired film adhesives. Improvement of composite repair technologies is clearly needed to reduce environmental burdens and abatement costs. To address this need, Infoscitex is developing an innovative no-shelf-stable repair adhesive that will reduce the amount of waste generated during composite repair operations. Such an adhesive promises to revolutionize repair processes with respect to waste minimization, cost savings, and protection of the environment.

The objective of this three-year program is to develop one-part epoxy film adhesive that is stable at ambient temperatures for over one year. Our approach leverages novel microencapsulant formulations that do not interfere with the epoxy curing process and produce an adhesive that is compatible with existing film formulations and processes. The shelf stable adhesive (SSA) does not require activation or any other major departure from standard processes (TO-1-1-690) and can be rapidly, safely, and reliably used to repair military and commercial composite and metal structures found in aircraft, ships, amphibious and tactical vehicles.

1.3 References

Please refer to the following documents for more information about qualification of materials:

- AMS-A-25463 (replaces MIL-A-25463B, Notice 1). This document provides guidance on adhesive films for use in structural construction.
- ASTM C 297. Provides guidance on flatwise tensile strength testing.
- ASTM C 393. Provides guidance on sandwich flexure testing.
- ASTM D 1781. Provides guidance on sandwich peel strength testing.
- MIL-A-25463B. For perspective only, and is not to be used as a guiding document.
- MIL-A-83377. Provides guidance on structural adhesive bonding for aerospace systems.
- MIL-B-22191. Provides guidance on transparent barrier materials.
- MIL-C-7438. Provides guidance on aluminum core materials for sandwich construction.
- MIL-F-22191. Provides guidance on packaging materials with vapor barrier properties.
- MIL-T-5624. Provides guidance on aviation fuel properties, specifically JP-4 and JP-5.
- PPP-B-636. Provides guidance on boxes for packaging.
- T.O. 1-1-690 General advanced composite repair process manual

2. Requirements Specification

2.1 Chemical and Physical Property Requirements

AMS-A-25463 provides all requisite guidance for the chemical and physical performance of film adhesives. The baseline adhesive, Henkel Aerospace Hysol EA 9696 is classified as a Type I adhesive. Accordingly, performance metrics for the film adhesive featuring encapsulated accelerator will be based on Type I requirements. These performance metrics are summarized in **Table 1** below.

Table 1. Chemical and physical property requirements for shelf-stable adhesives

Property	Adhesive Type*	Acceptable Range		Test Method‡
		Minimum	Maximum	
Application Temperature (ºF)	All	65	85	N/A
Cure Time				
if at 100ºF or below	All	N/A	7 days	DSC
if at 100ºF to 200ºF	All	N/A	24 hrs	DSC
if at 200ºF to 300ºF	All	N/A	2 hrs	DSC
if at 300ºF or above	All	N/A	2 hrs	DSC
Shelf-Life (months)				
at 40ºF	All	12	N/A	DSC
at 0ºF	All	24	N/A	DSC
Sandwich Peel Strength (lb-in./in. width)				
70-80ºF	I II III, IV	12.5 10.0 3.5	N/A	ASTM D 1781
180±2ºF	I, II III, IV	10.0 3.5	N/A	ASTM D 1781
-67±2ºF	All	10	N/A	ASTM D 1781
Flatwise Tensile Strength (psi)				
70-80ºF	All	750	N/A	ASTM C 297
180±2ºF	I	400	N/A	ASTM C 297
300±5ºF	II	350	N/A	ASTM C 297
500±5ºF	III, IV	220	N/A	ASTM C 297
-67±2ºF	All	800	N/A	ASTM C 297
Flexural Strength (lbs)				
70-80ºF	All	2100	N/A	ASTM C 393
180±2ºF	I	1275	N/A	ASTM C 393
300±5ºF	II	1500	N/A	ASTM C 393
500±5ºF	III, IV	1200	N/A	ASTM C 393
-67±2ºF	All	2150	N/A	ASTM C 393
Flexural Strength (lbs) after 192 hrs Exposure				
180±2ºF	I	1500	N/A	ASTM C 393
300±5ºF	II, III	1200	N/A	ASTM C 393
500±5ºF	IV	600	N/A	ASTM C 393
Fluid Immersion Resistance *30 days ±2 hrs at 70-80ºF fully immersed in JP-4 complying with MIL-T-5624*	All	2100	N/A	ASTM C 393
High Humidity Resistance *30 days±2 hrs at 120±5ºF and 95-100%RH*	All	2100	N/A	ASTM C 393 Test at 70-80ºF
Creep Deflection in Flexure after 192 hrs under Load (in.)				
100 lb Load, 70-80ºF	All	N/A	0.025	ASTM C 393
800 lb Load, 180±2ºF	I	N/A	0.05	ASTM C 393
1000 lb Load, 300±5ºF	II, III	N/A	0.05	ASTM C 393
500 lb Load, 500±5ºF	IV	N/A	0.05	ASTM C 393

* **Type I**: for long-time exposure to -67 to 180ºF; **Type II**: for long-time exposure to -67 to 300ºF; **Type III**: for long-time exposure to -67 to 300ºF and short-time exposure to 300 to 500ºF; **Type IV**: for long-time exposure to -67 to500ºF

‡ All tests shall be performed upon reaching equilibrium as defined on Page 19 in AMS-A-25463

2.2 Non-functional Requirements

In addition to the chemical and physical property requirements listed in Section 2.1, the shelf-stable adhesive must meet several non-functional requirements. These include:

- Environmental. Must not emit more VOC or HAP than current Hysol EA 9696 product.
- Cost. The material must present economic sense when compared to the current state of the art. This target is not presently defined; however, material selection should be done with cost factored.
- Domestic Source. Supply chain security is of high concern for the US Government. All materials shall be sourced domestically where practical.

3. Dictionary

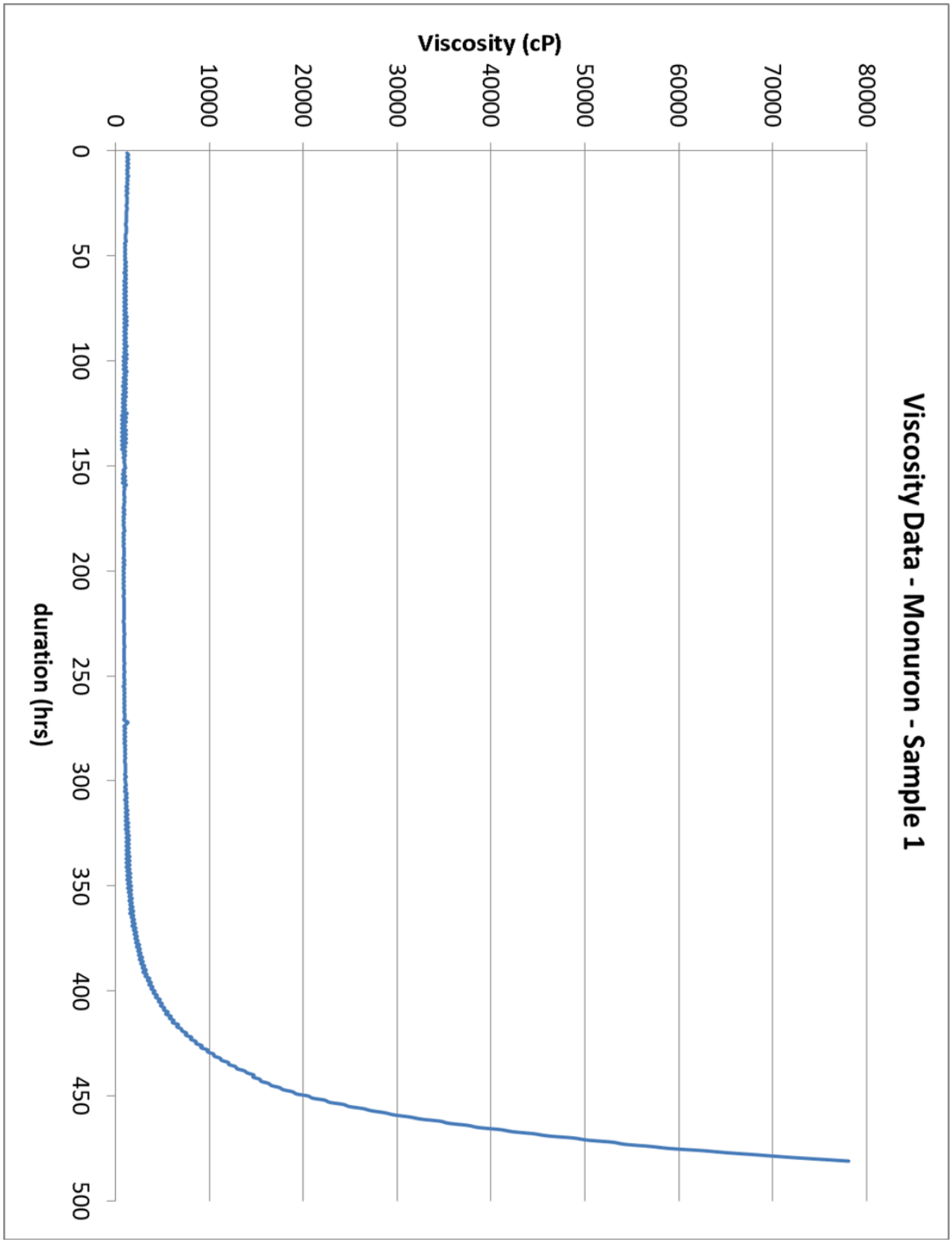
5.5 Terms and Abbreviations

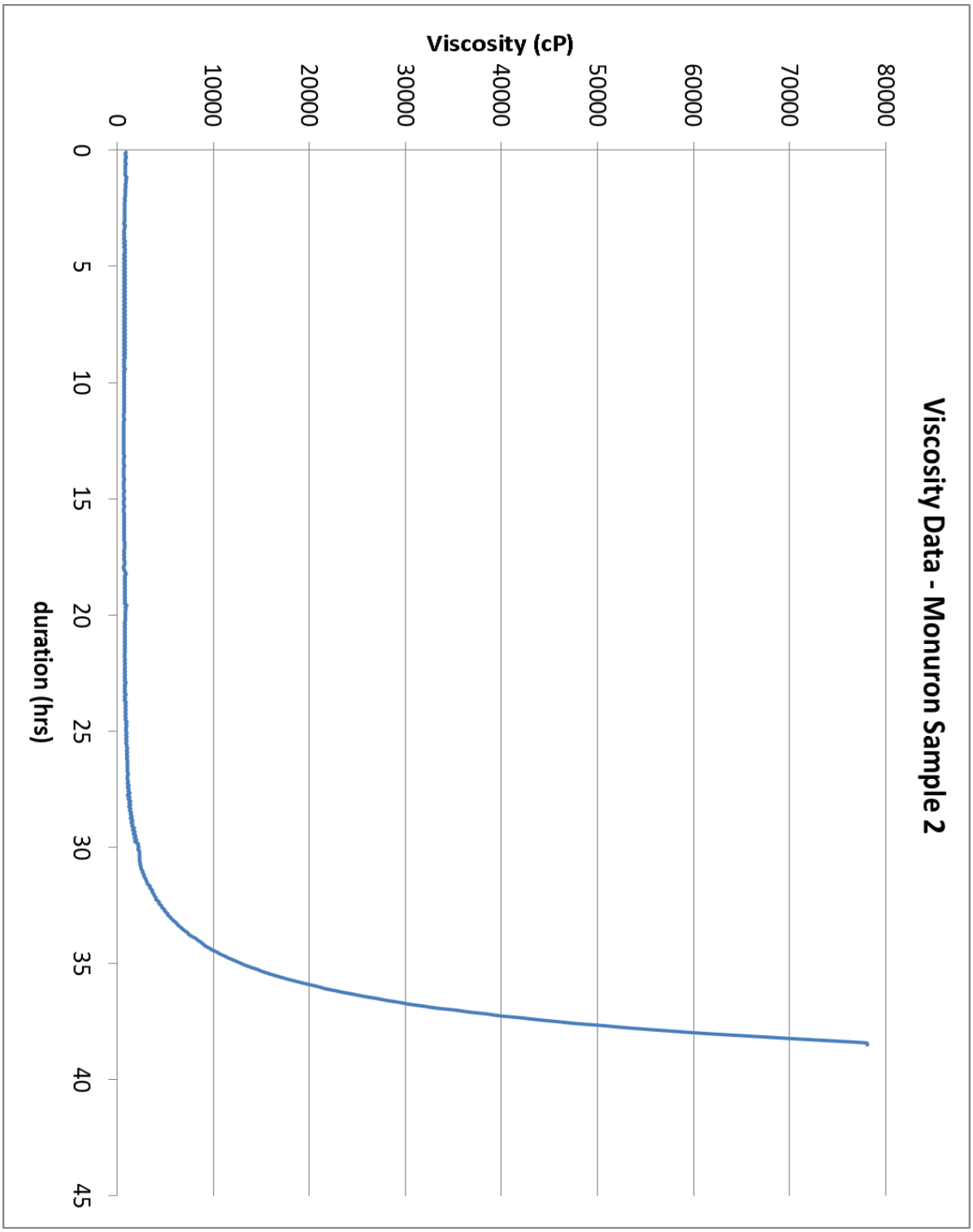
Term	Definition
ASTM	American Society for Testing and Materials
DSC	differential scanning calorimetry
^{o}F	degrees Fahrenheit
^{o}C	degrees Celsius
HAP	hazardous air pollutant
HRS	hours
IN	inch
JP-4	jet propellant 4
JP-5	jet propellant 5
LB	pound
LBS	pounds
PSI	pounds per square inch
SSA	Shelf-stable adhesive
US	United States
VOC	Volatile organic compound

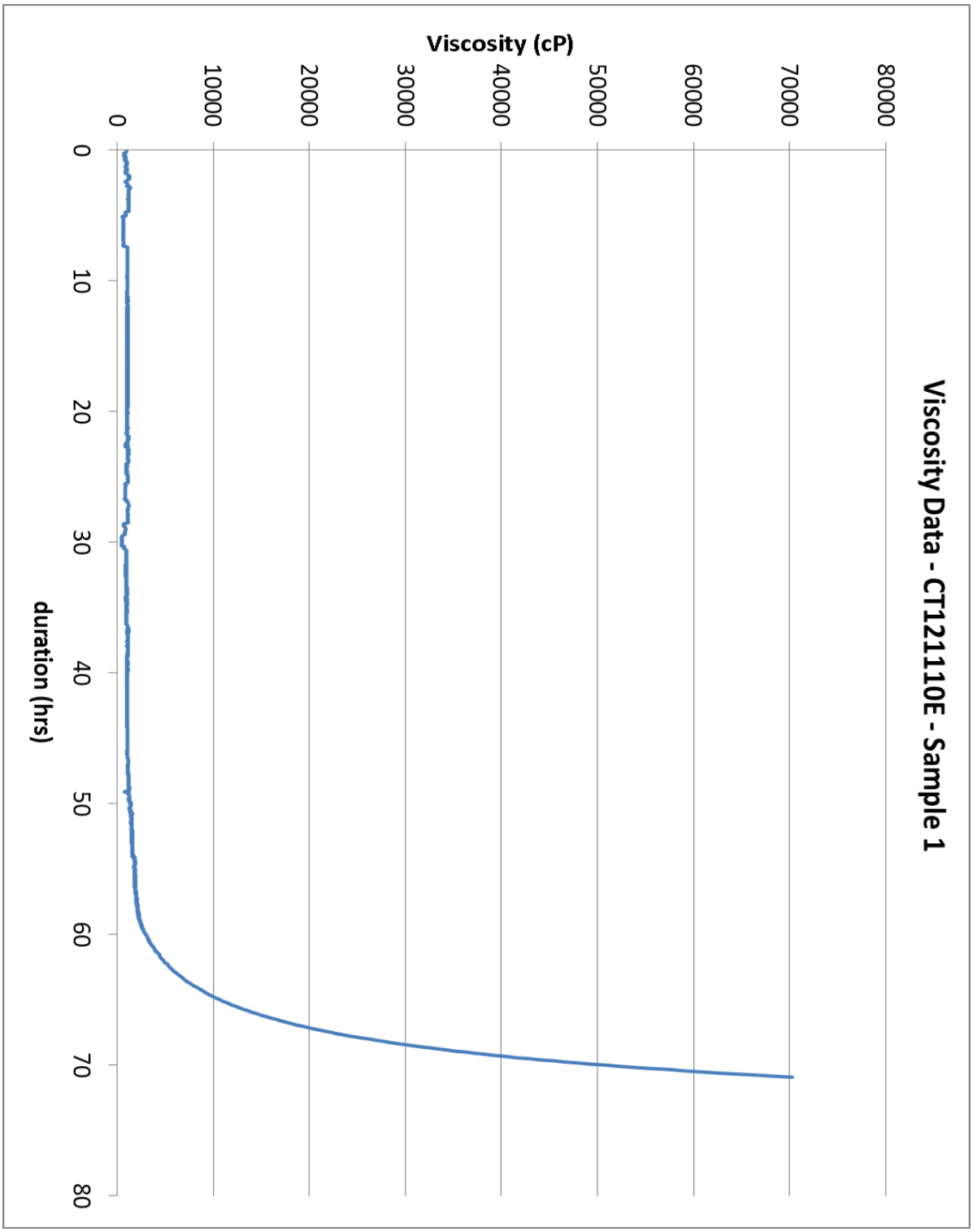
5.6 Notation/Formula

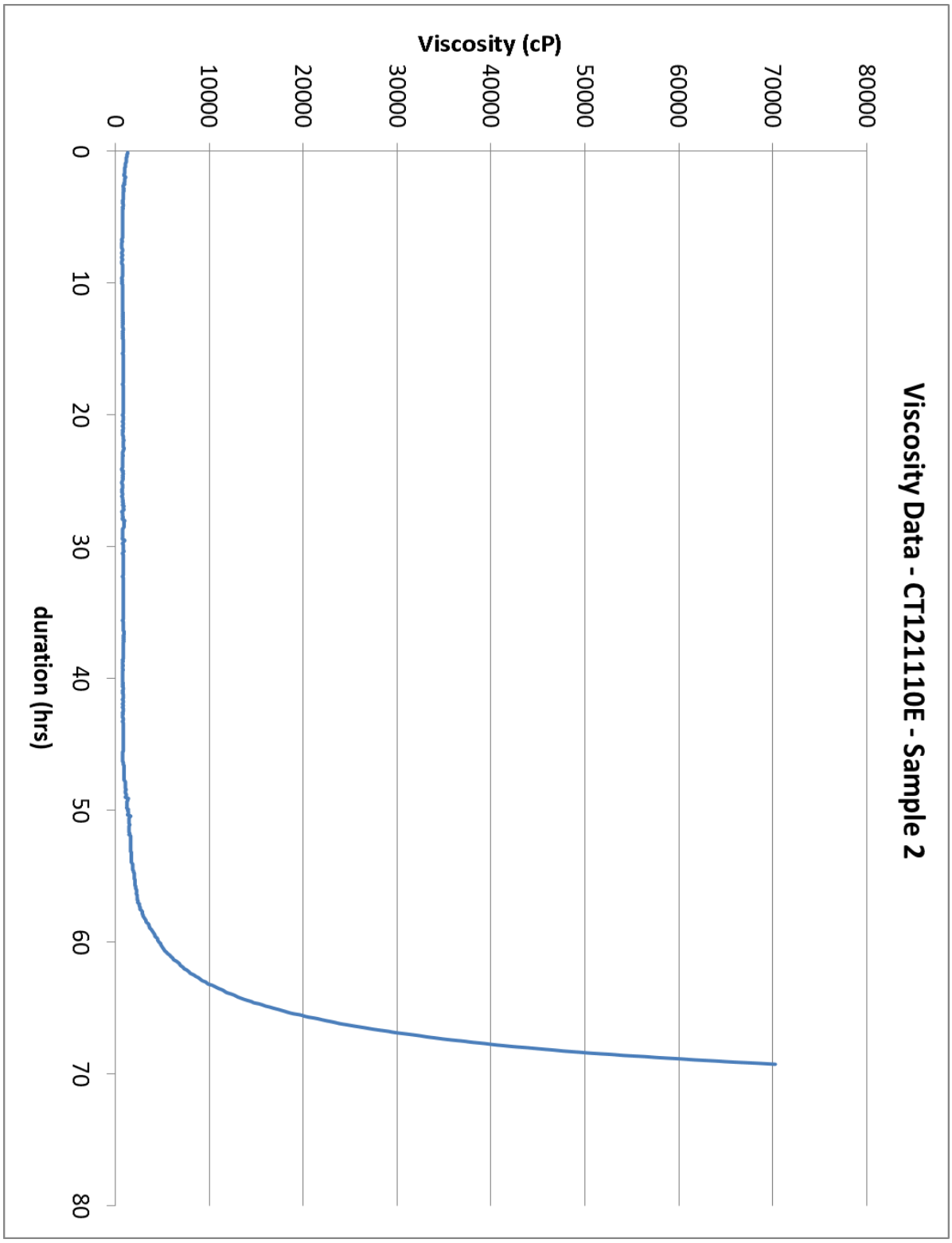
Notation	Definition

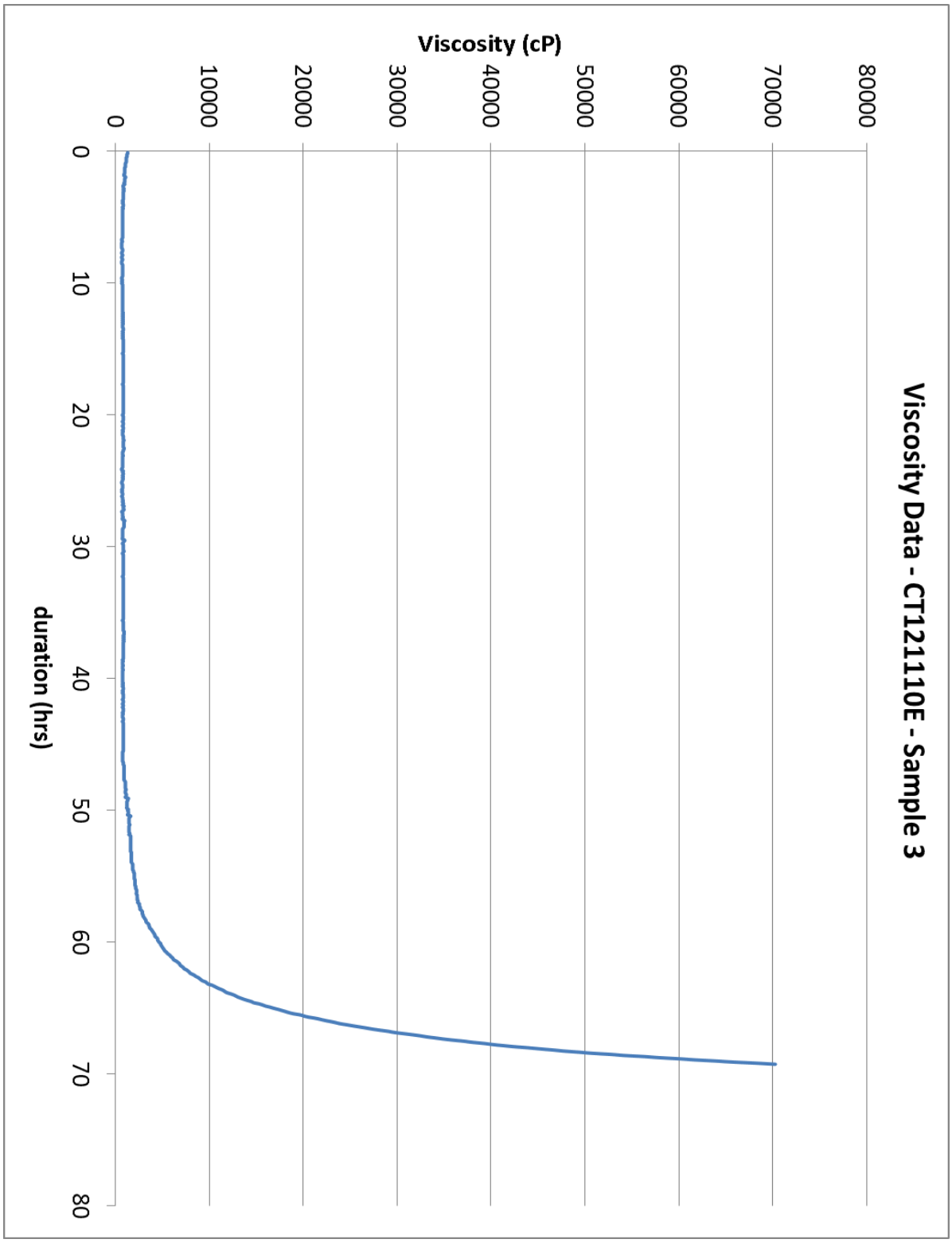
Appendix 2A: Viscosity Data for Monuron Based Samples

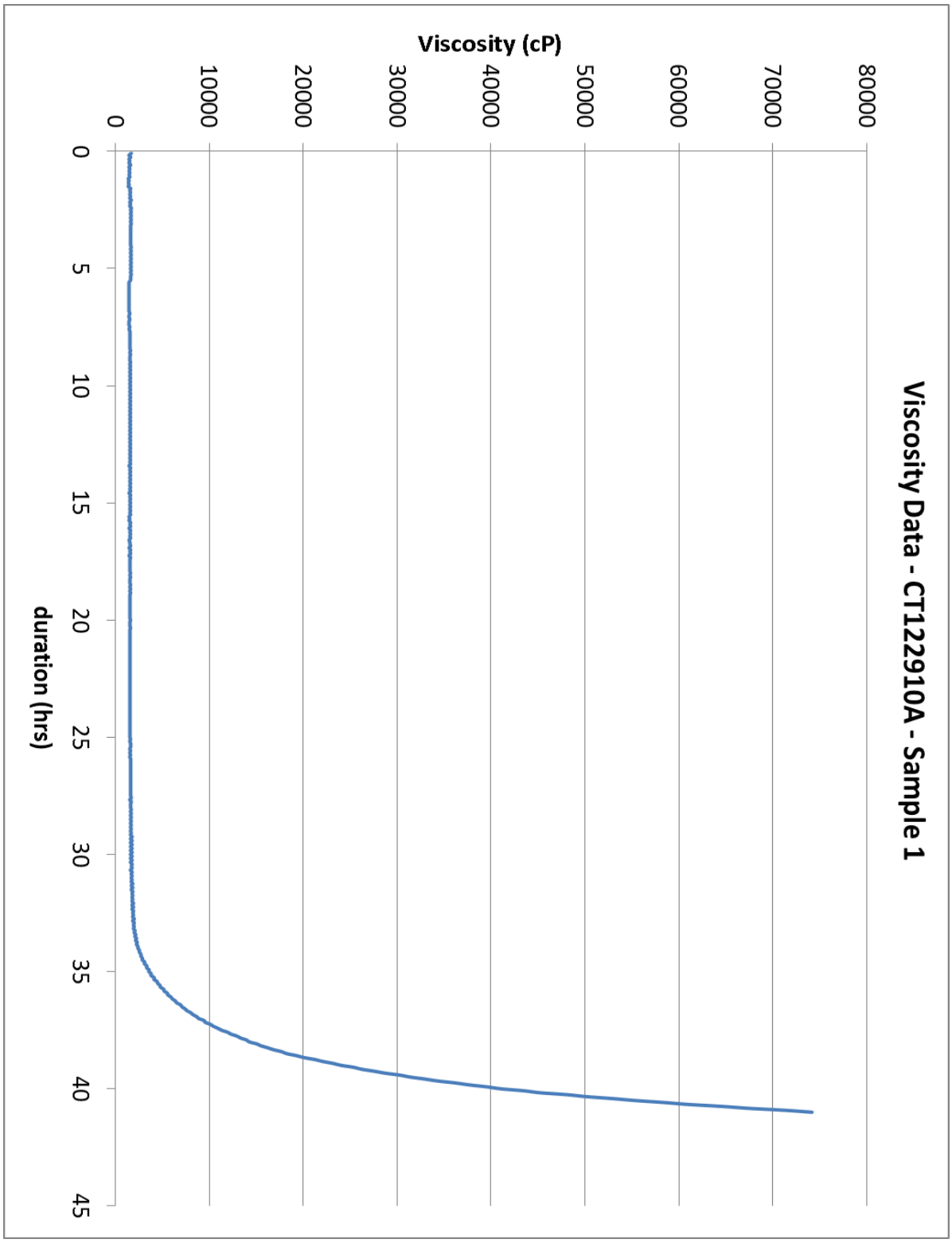


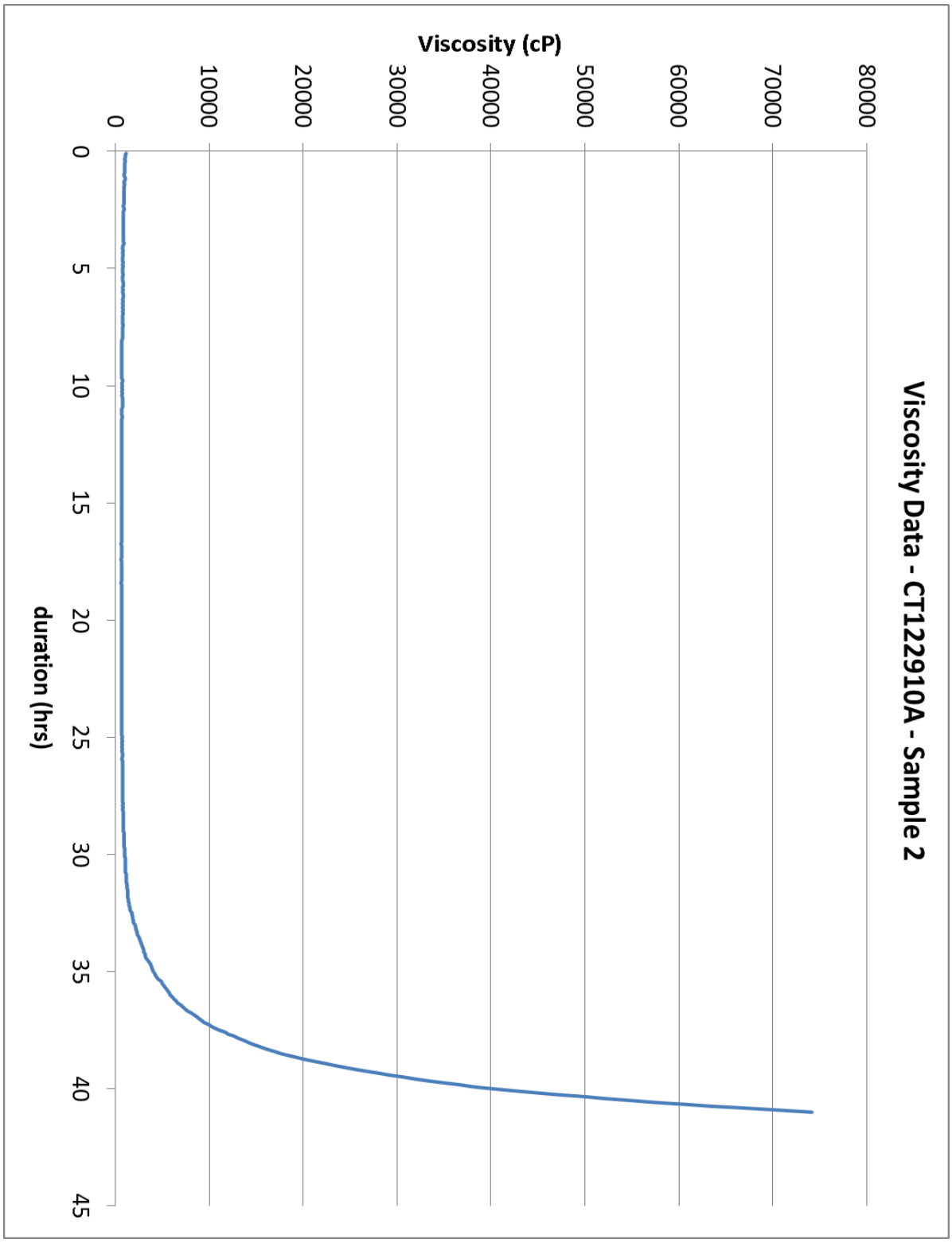

Viscosity Data - Monuron Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
duration (hrs)

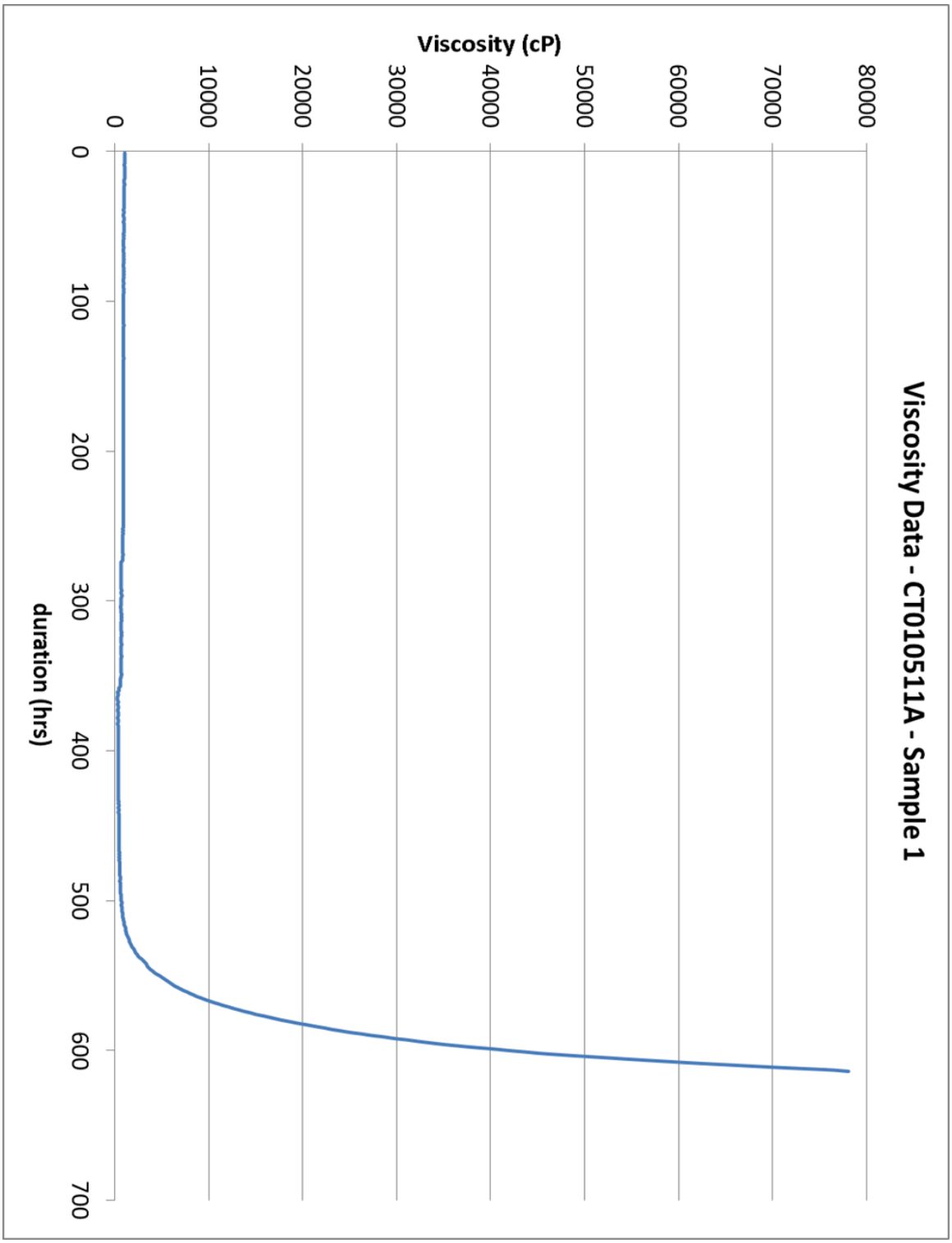

Viscosity Data - CT121110E - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
70
80
duration (hrs)

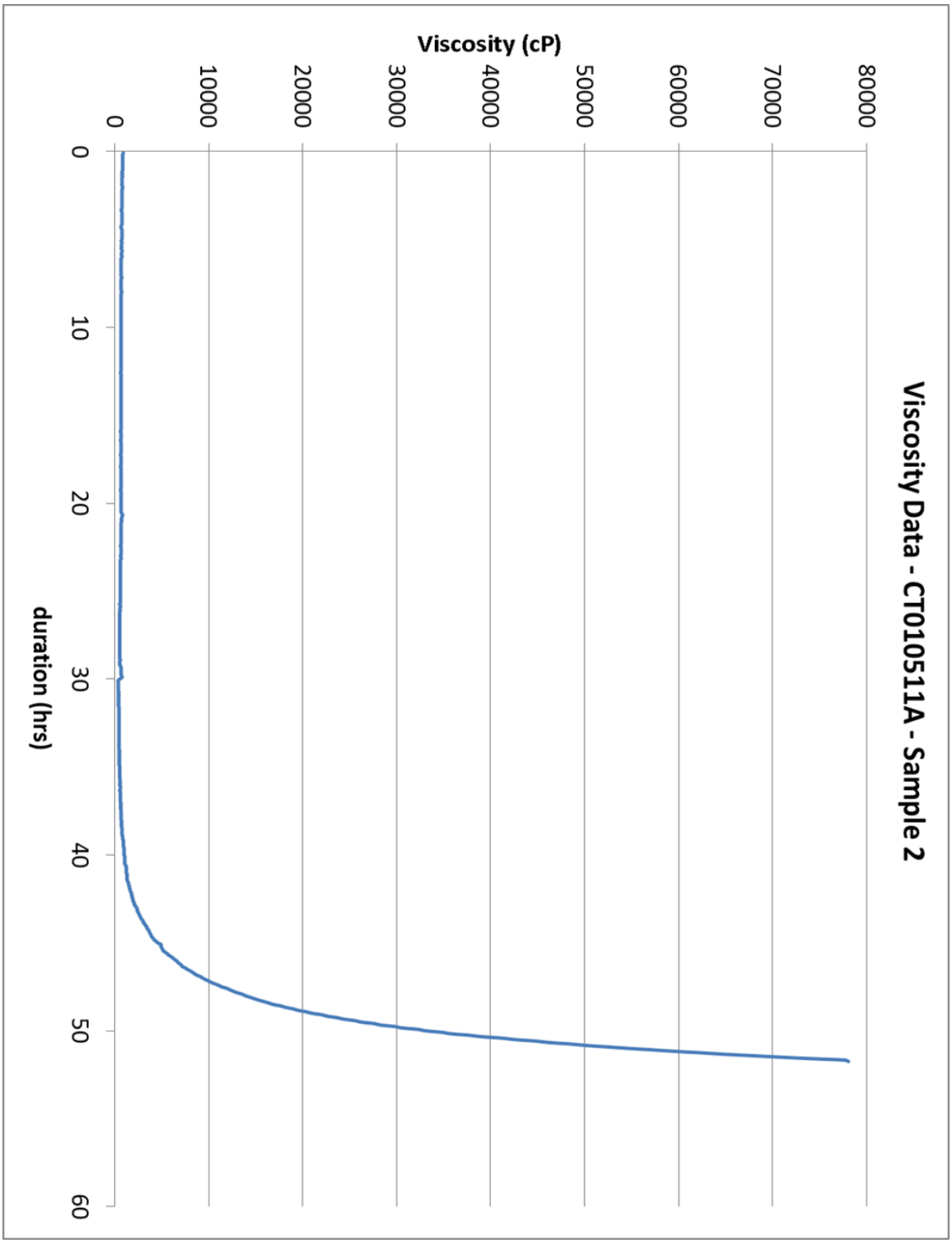

Viscosity Data - CT121110E - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
70
80
duration (hrs)

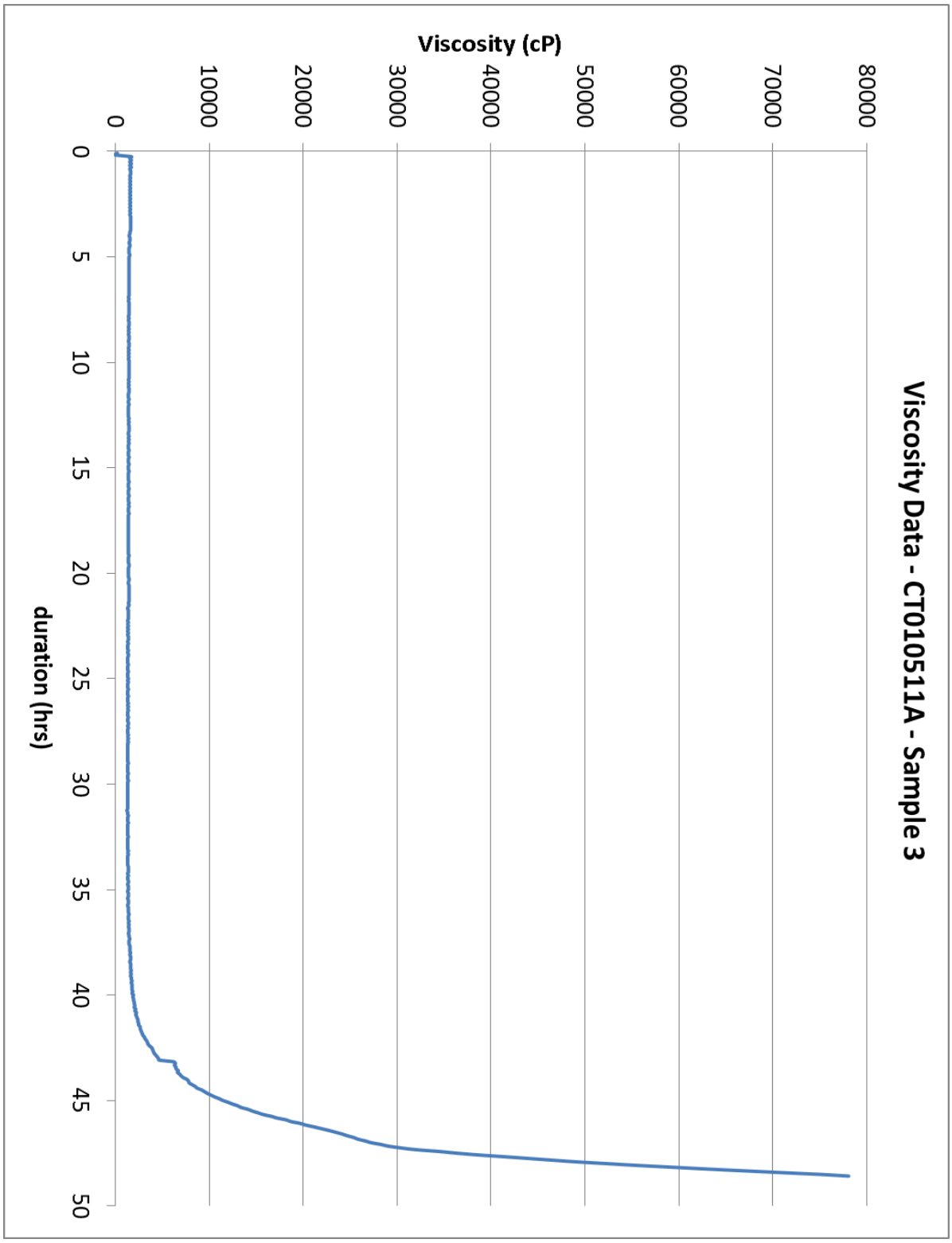

Viscosity Data - CT121110E - Sample 3
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
70
80
duration (hrs)

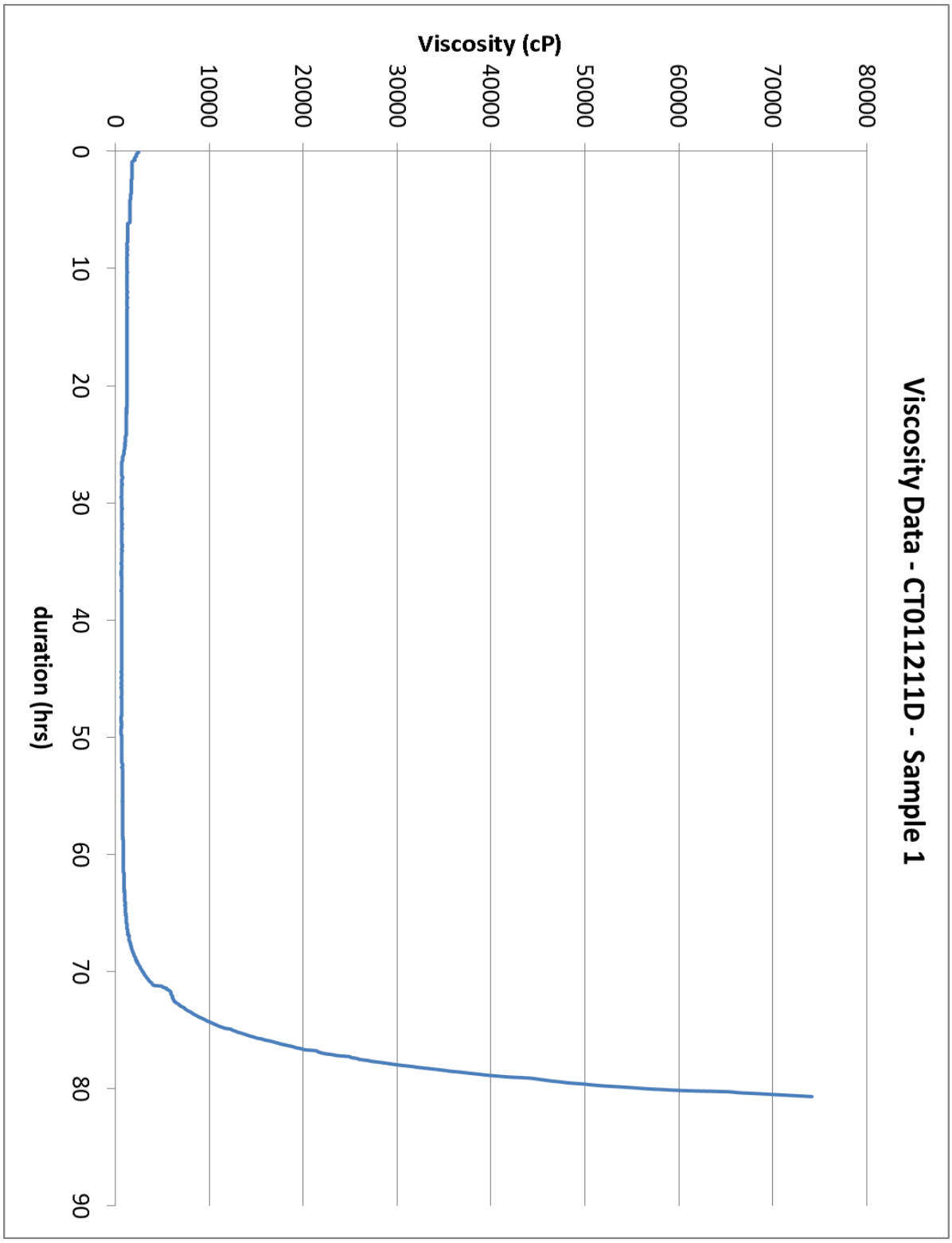

Viscosity Data - CT122910A - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
duration (hrs)

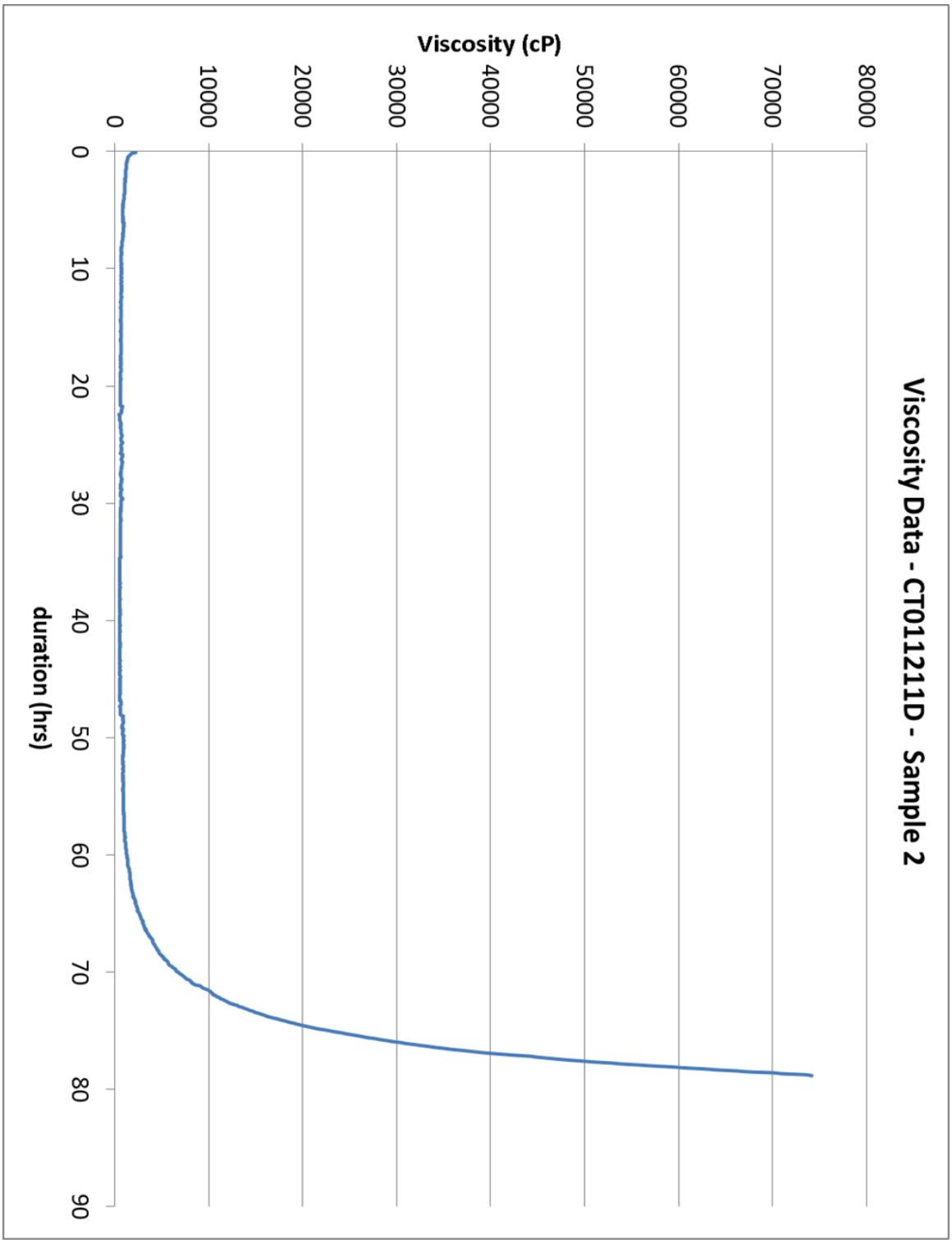

Viscosity Data - CT122910A - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
duration (hrs)

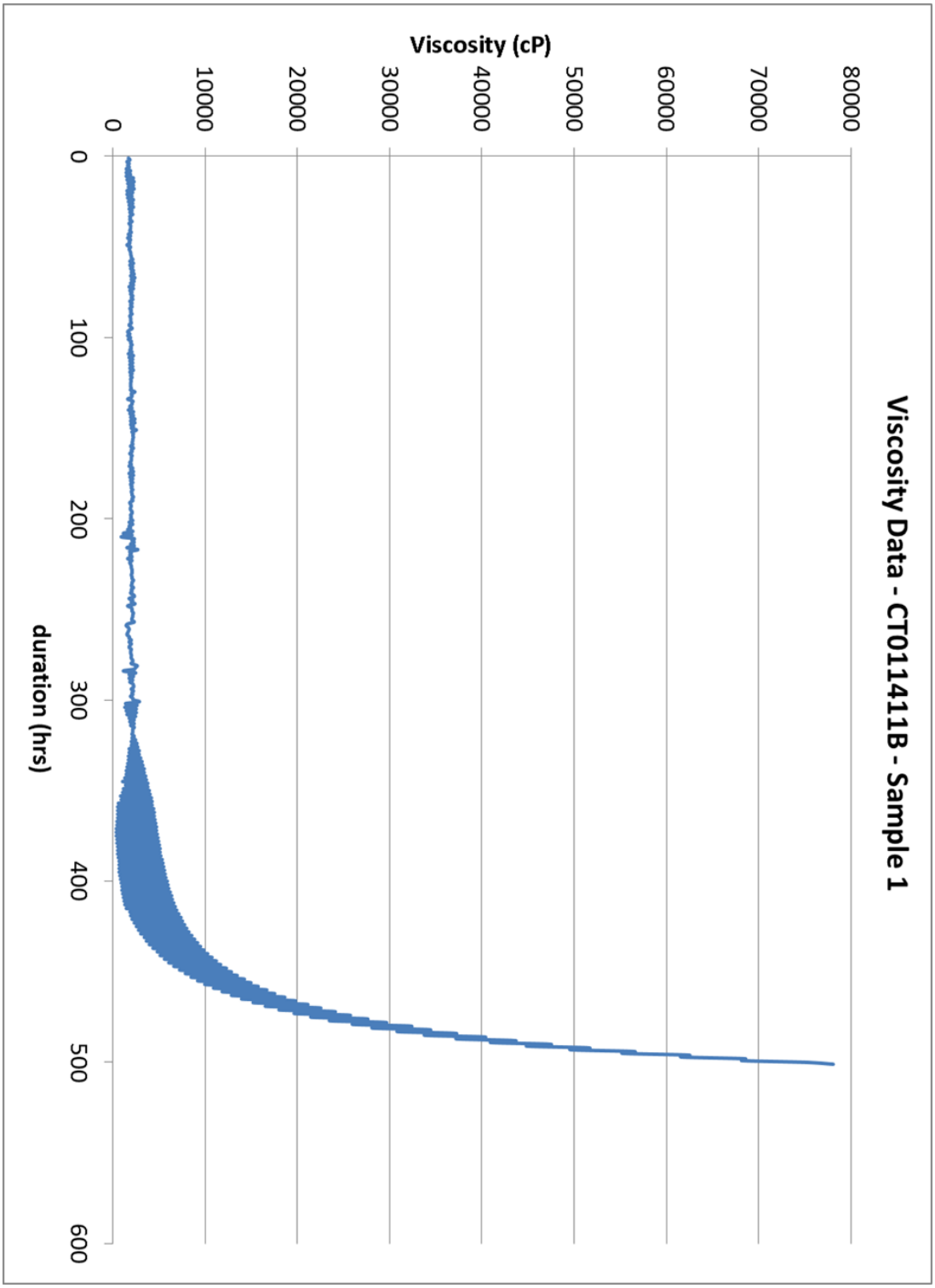

Viscosity Data - CT010511A - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
100
200
300
400
500
600
700
duration (hrs)

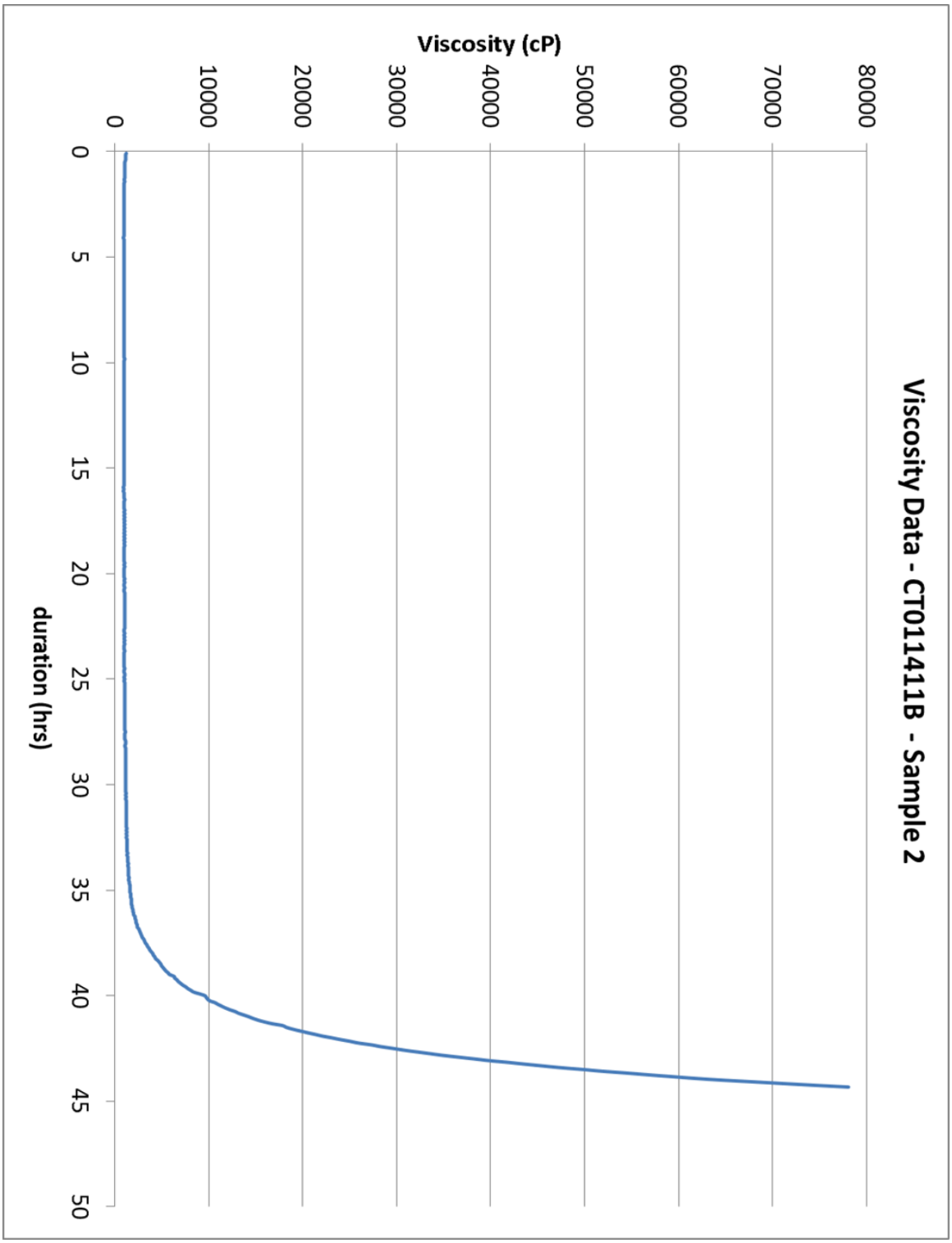

Viscosity Data - CT010511A - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
duration (hrs)

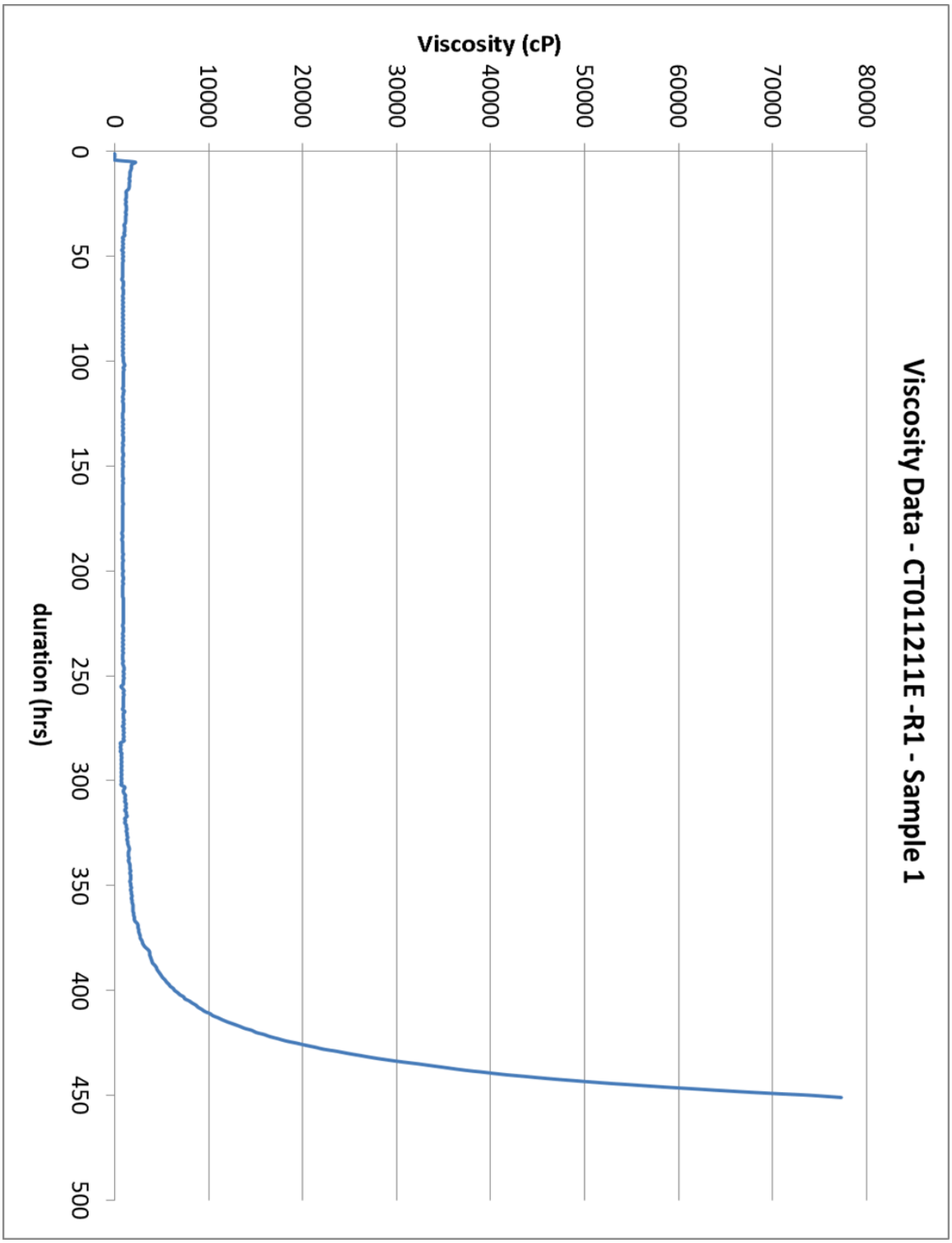

Viscosity Data - CT010511A - Sample 3
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
50
duration (hrs)

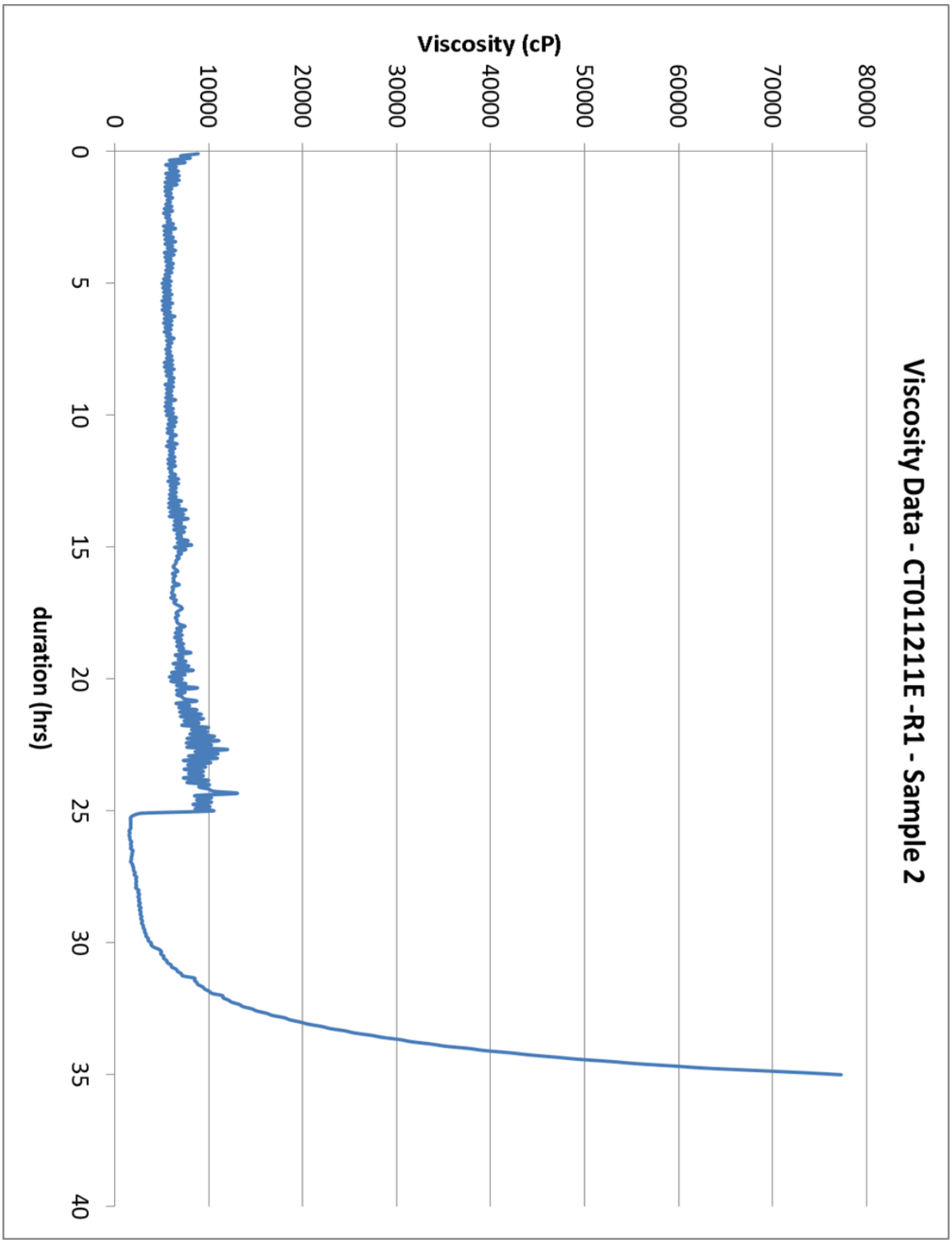

Viscosity Data - CT011211D - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
70
80
90
duration (hrs)

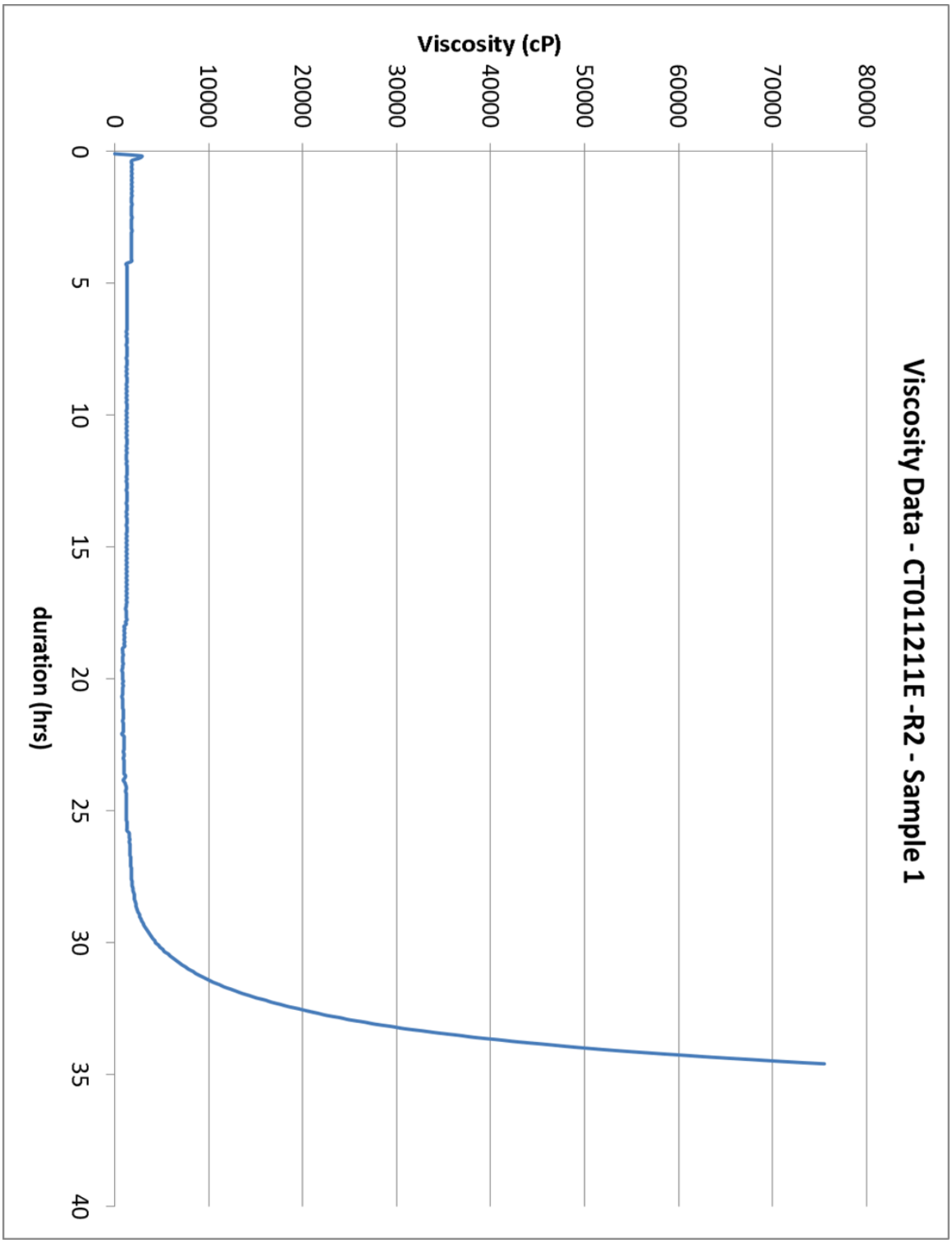

Viscosity Data - CT011211D - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
70
80
90
duration (hrs)

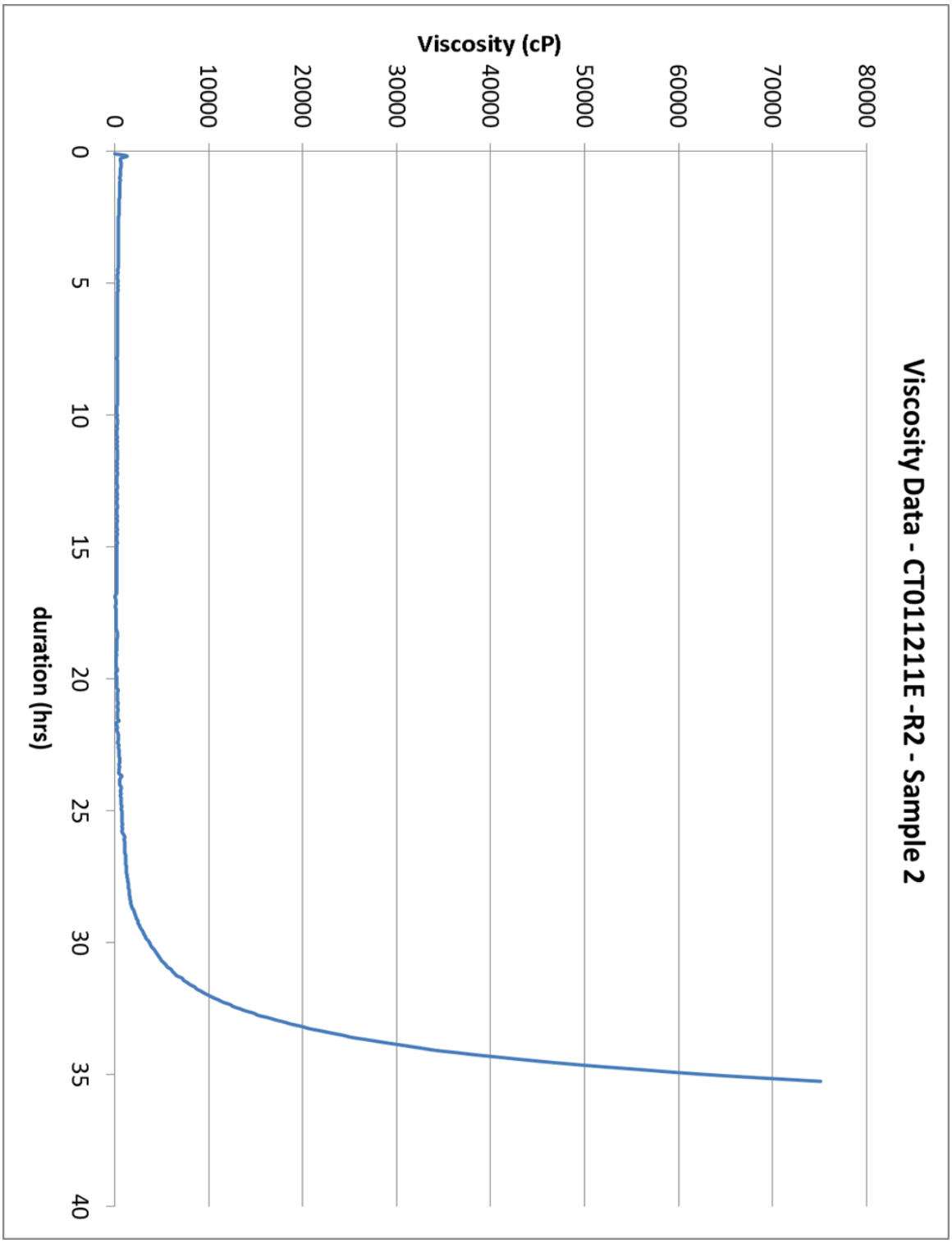

Viscosity Data - CT011411B - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
100
200
300
400
500
600
duration (hrs)

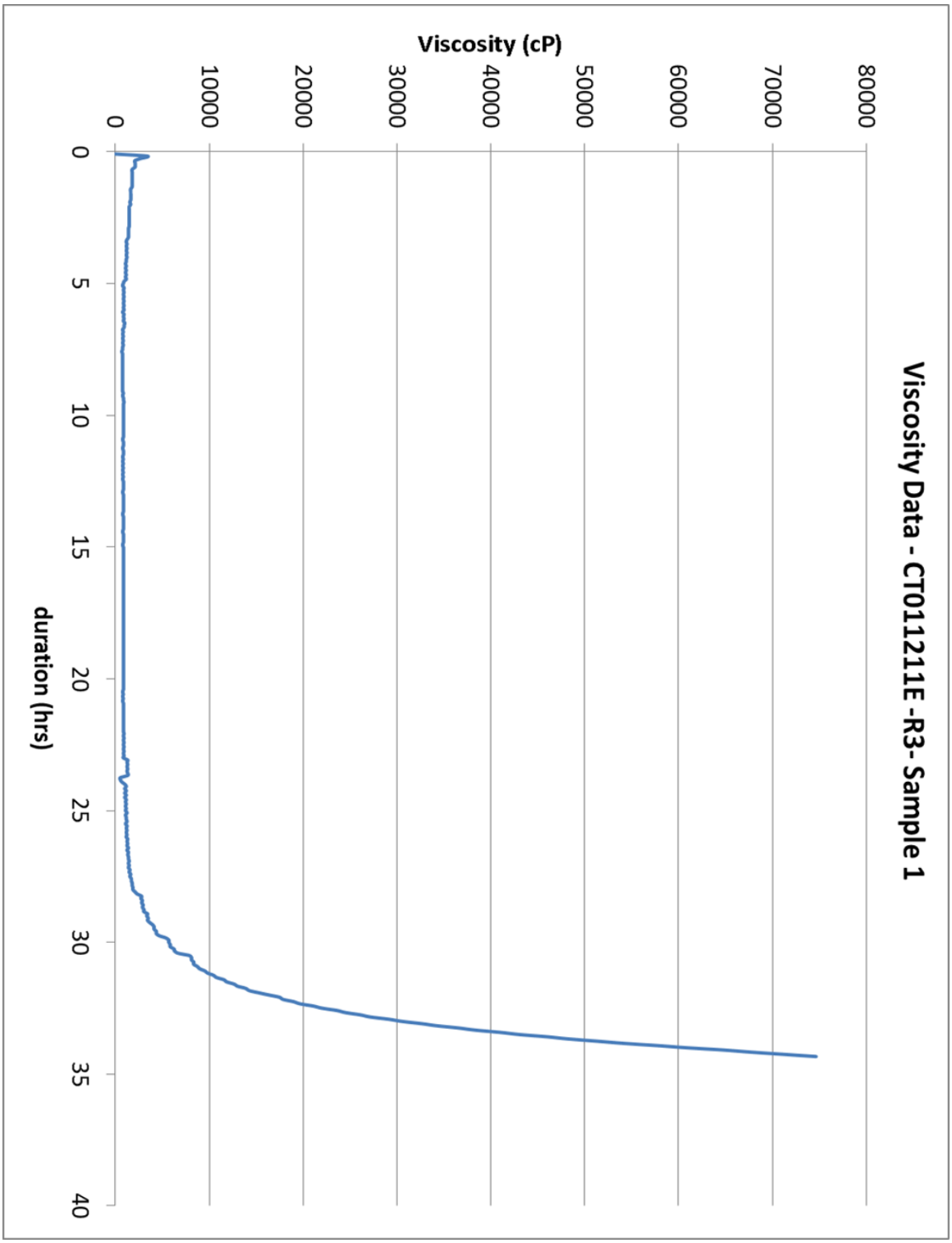

Viscosity Data - CT011411B - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
50
duration (hrs)

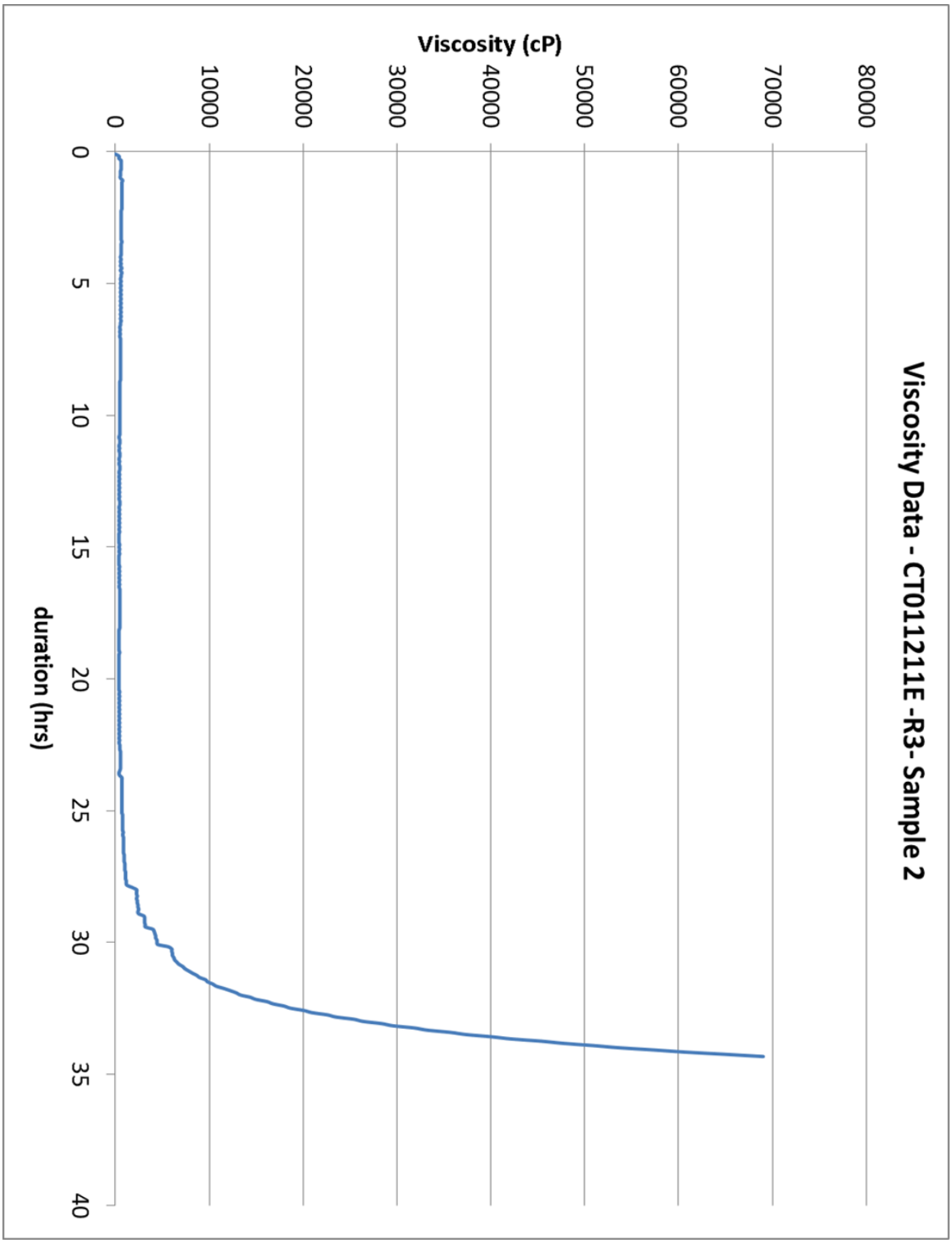

Viscosity Data - CT011211E -R1 - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
300
350
400
450
500
duration (hrs)

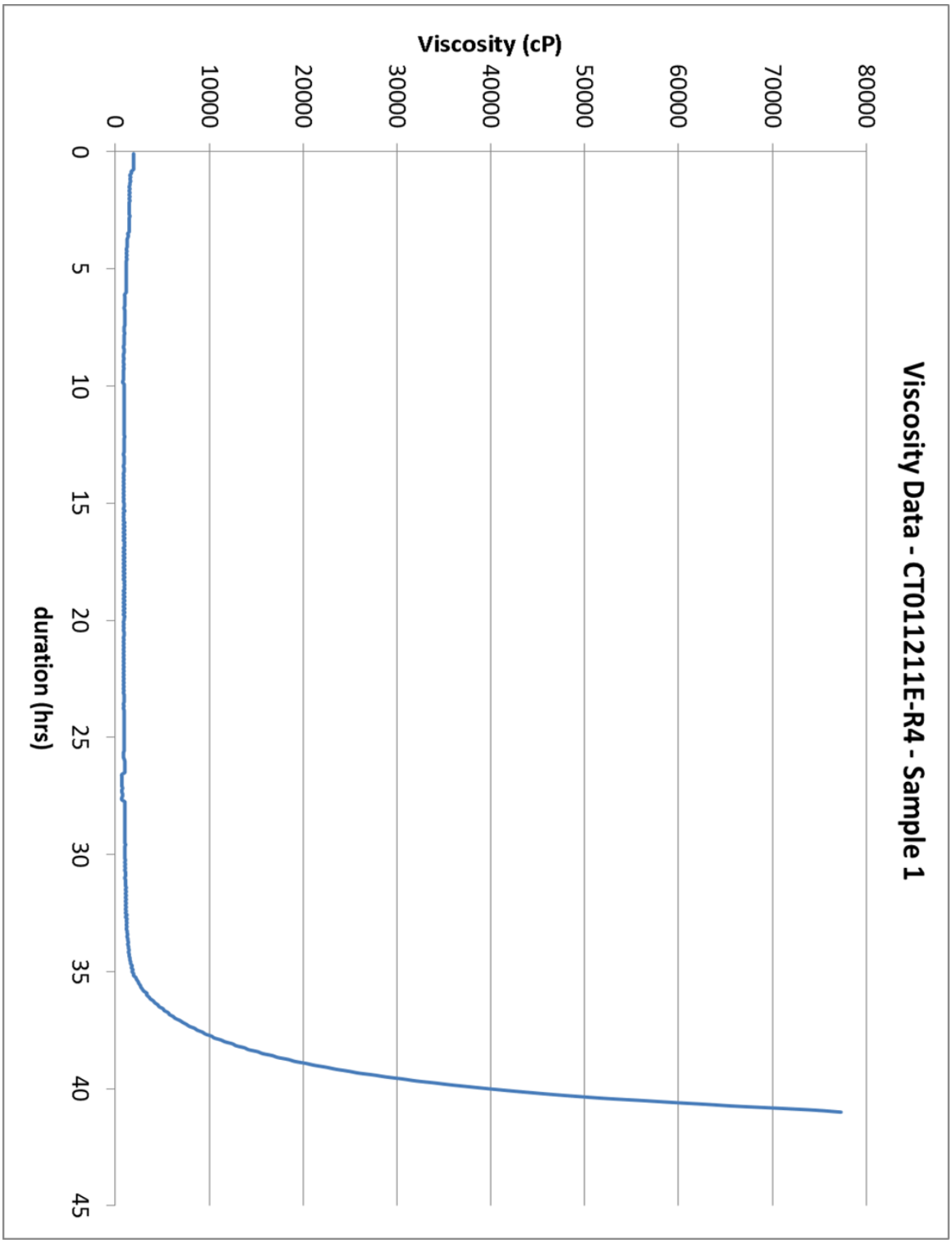

Viscosity Data - CT011211E -R1 - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

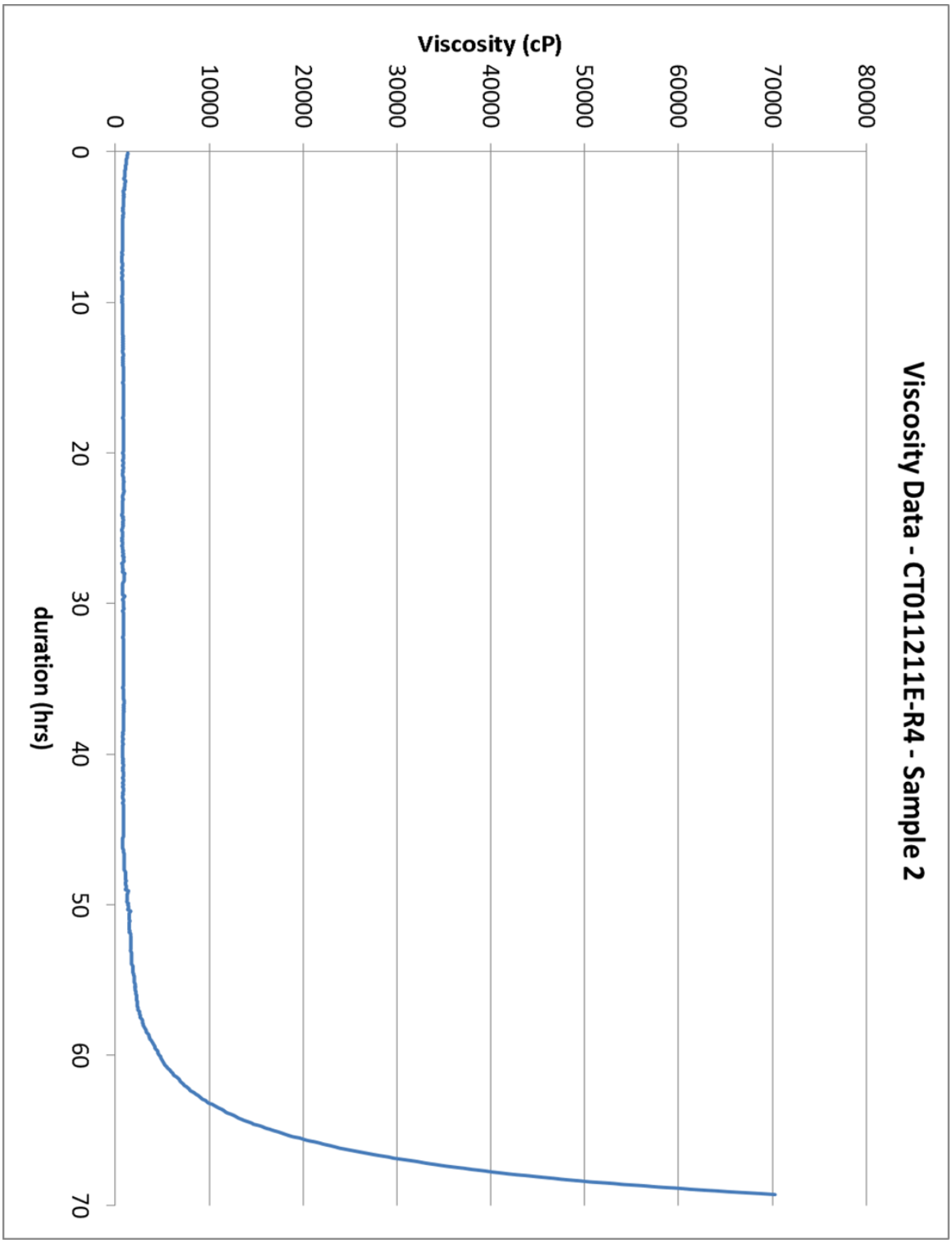

Viscosity Data - CT011211E -R2 - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

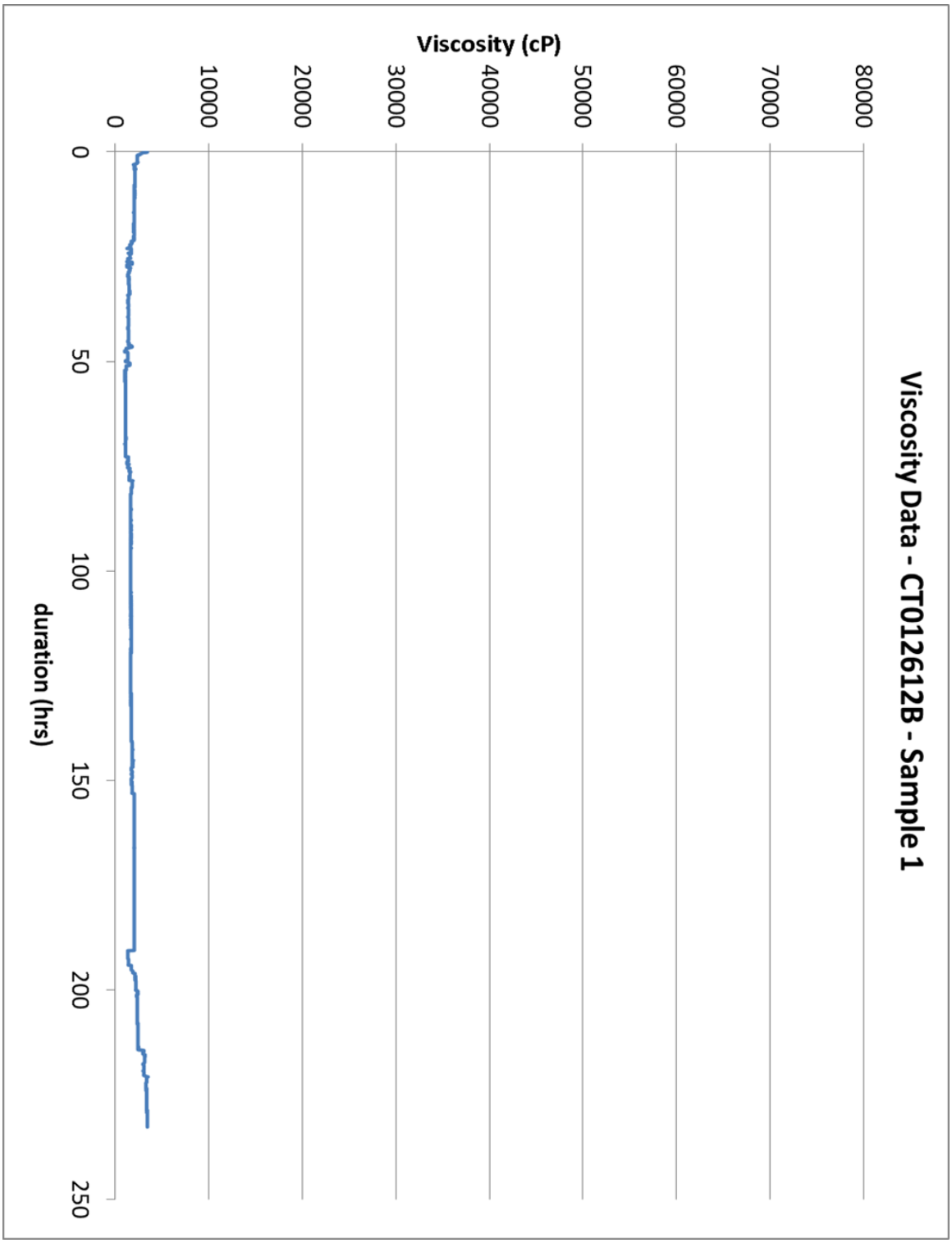

Viscosity Data - CT011211E -R2 - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

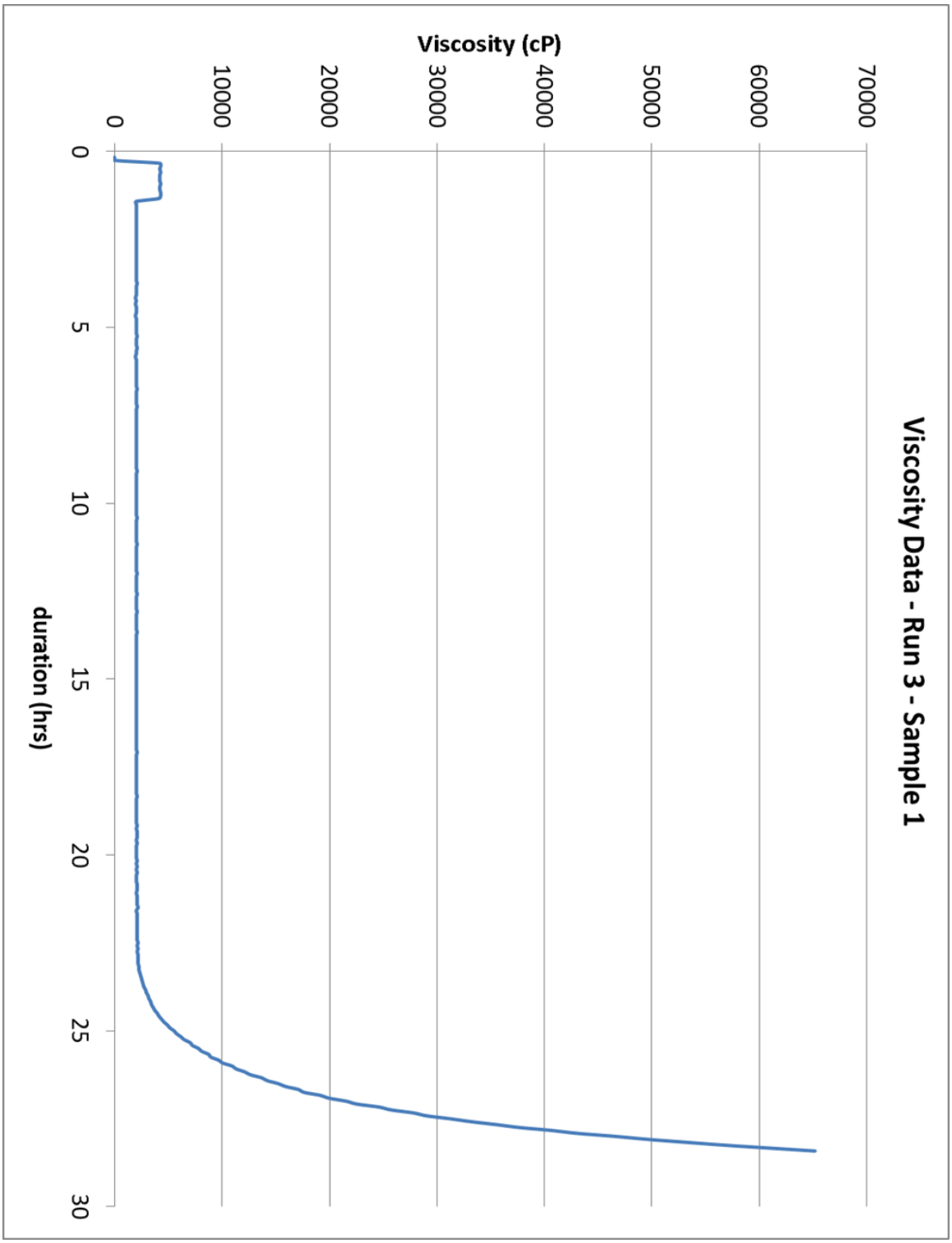

Viscosity Data - CT011211E -R3- Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

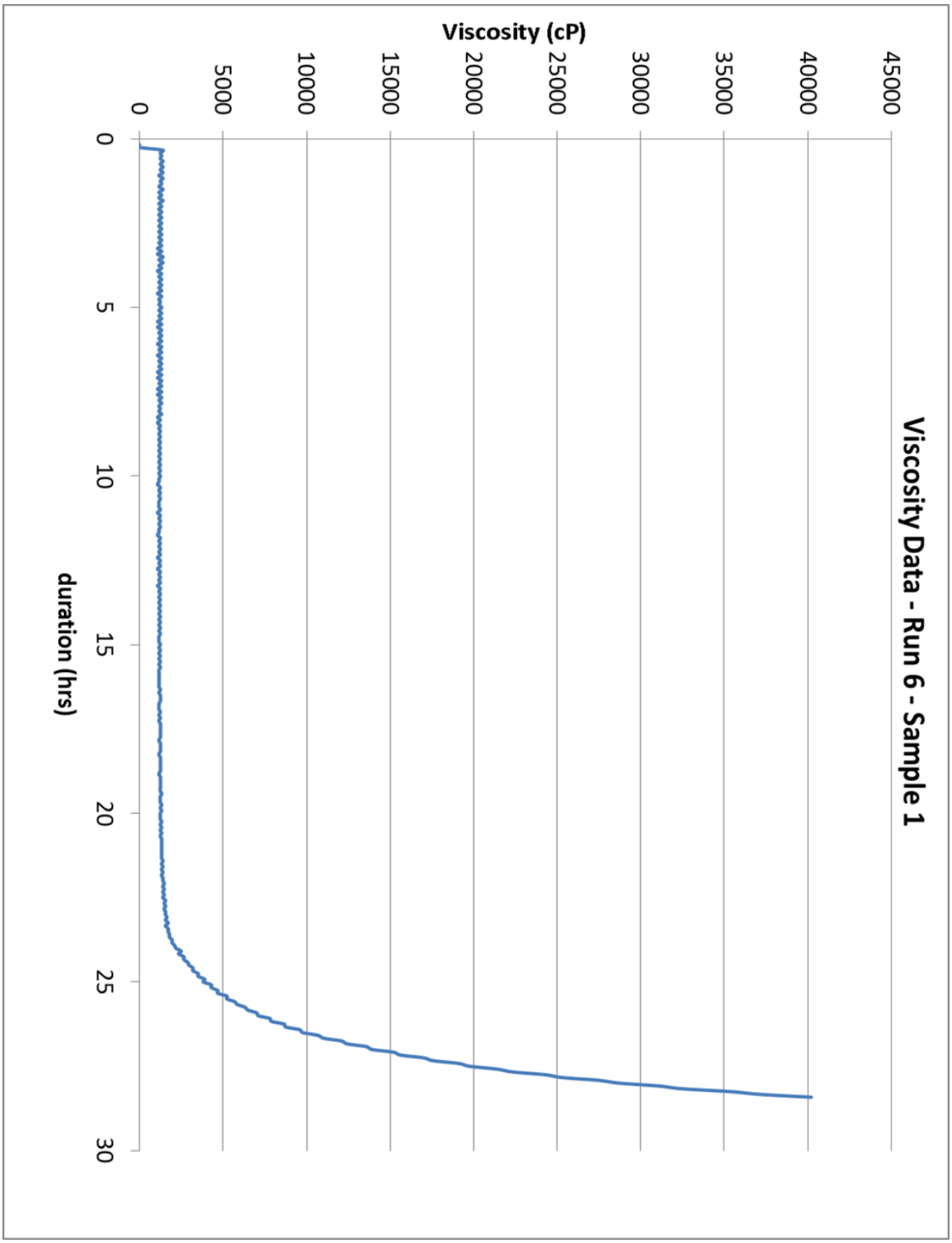

Viscosity Data - CT011211E -R3- Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)


Viscosity Data - CT011211E-R4 - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
duration (hrs)

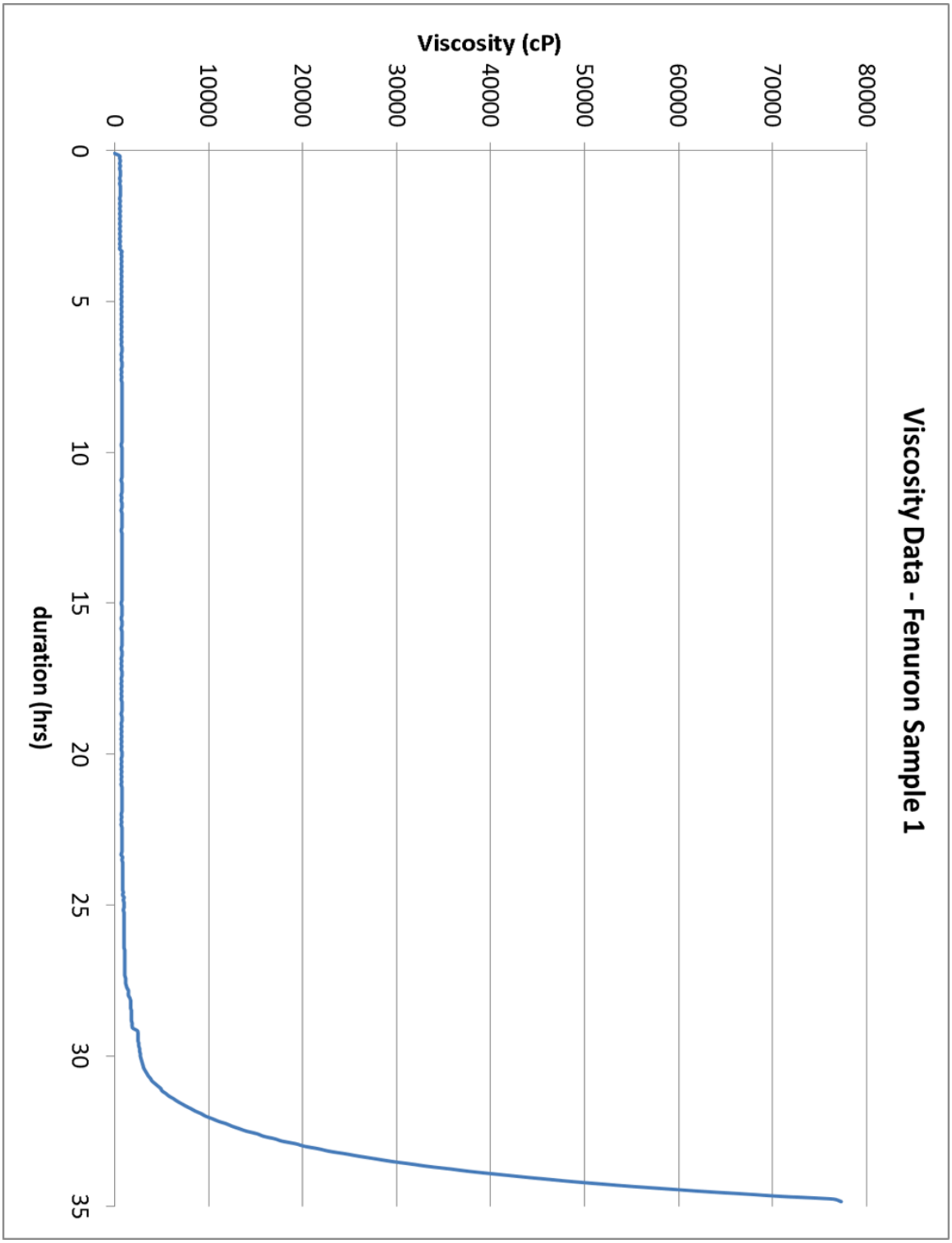

Viscosity Data - CT011211E-R4 - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
70
duration (hrs)

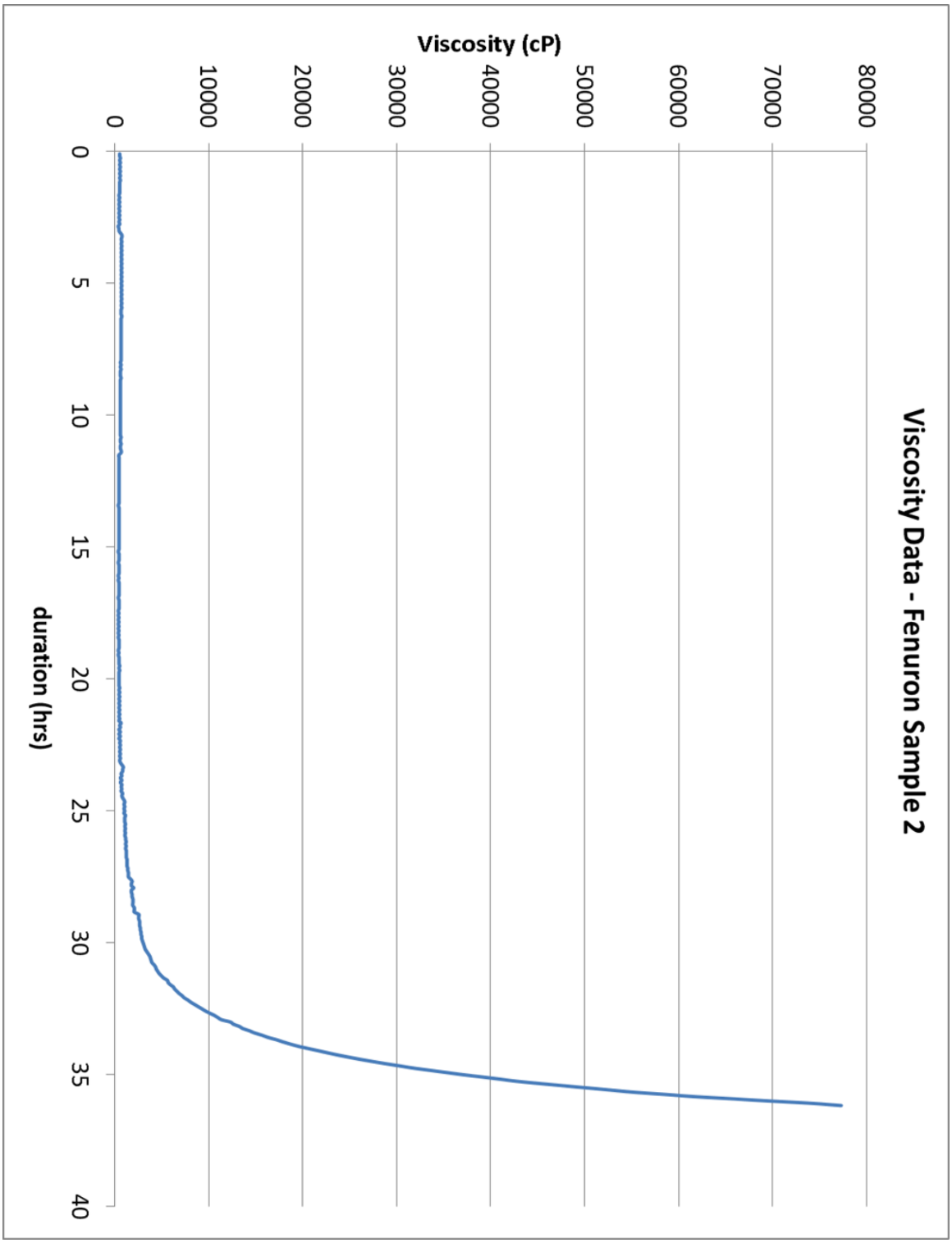

Viscosity Data - CT012612B - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
duration (hrs)

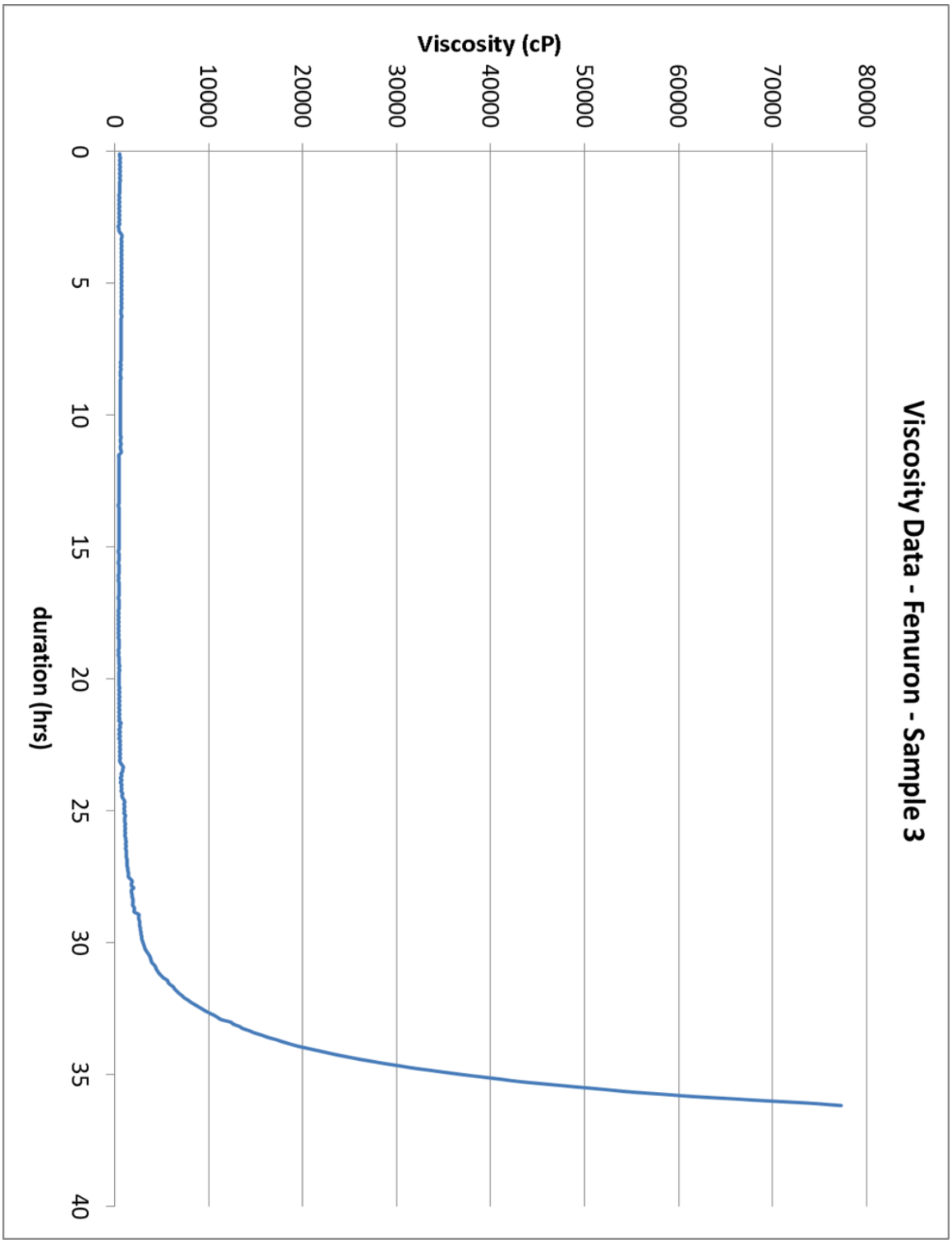

Viscosity Data - Run 3 - Sample 1
Viscosity (cP)
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
duration (hrs)

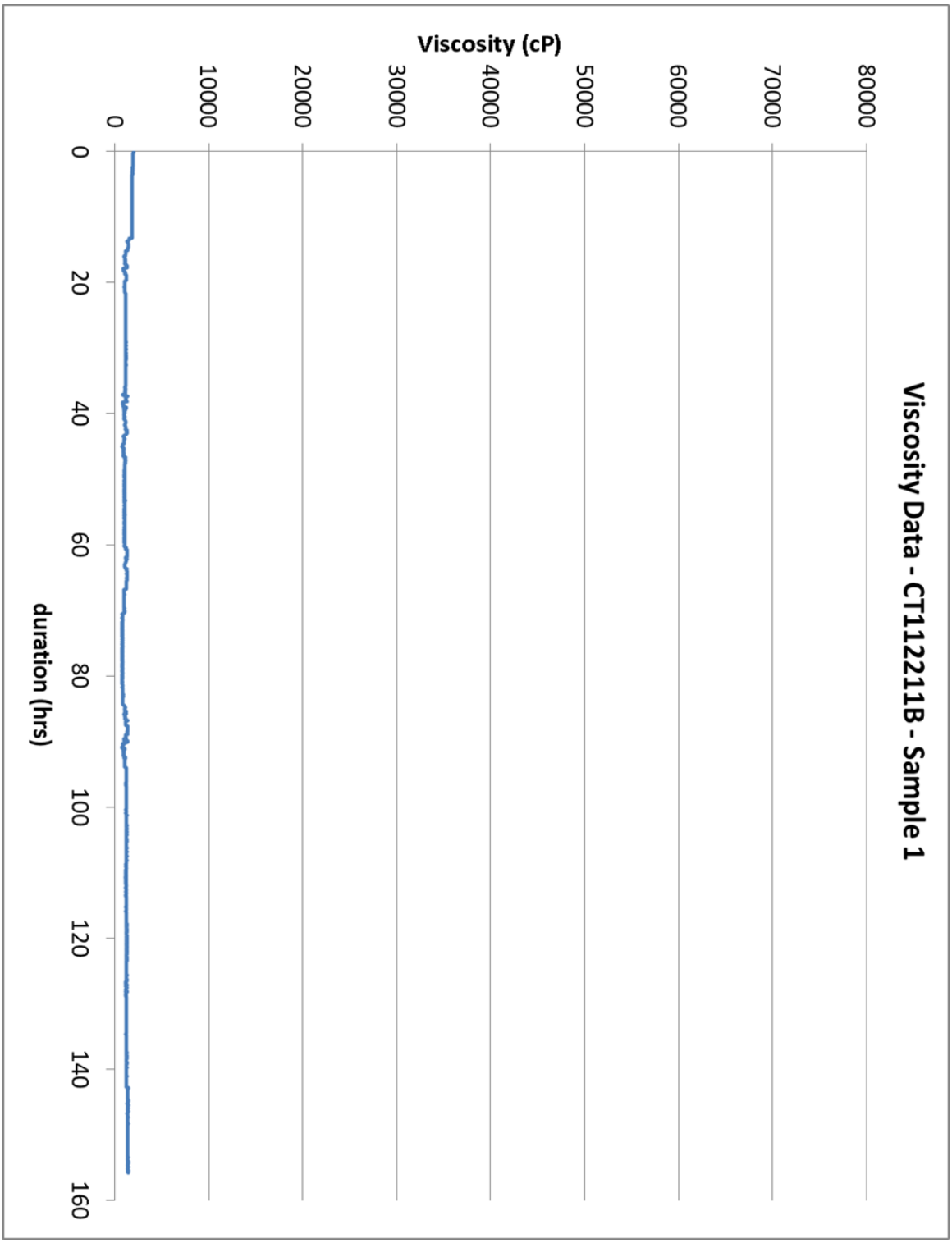

Viscosity Data - Run 6 - Sample 1
Viscosity (cP)
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
0
5
10
15
20
25
30
duration (hrs)

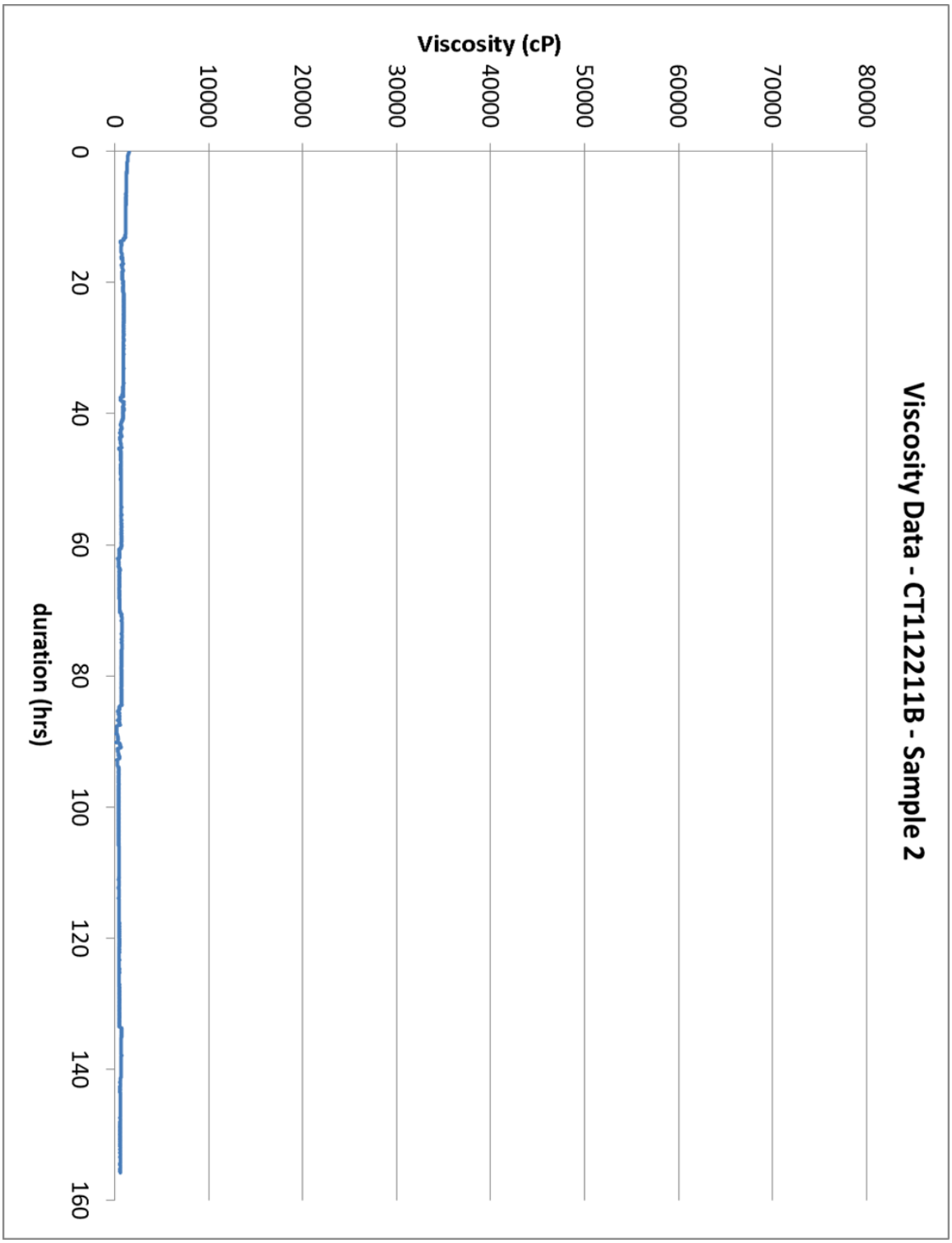
Appendix 2B: Viscosity Data for Fenuron Based Samples

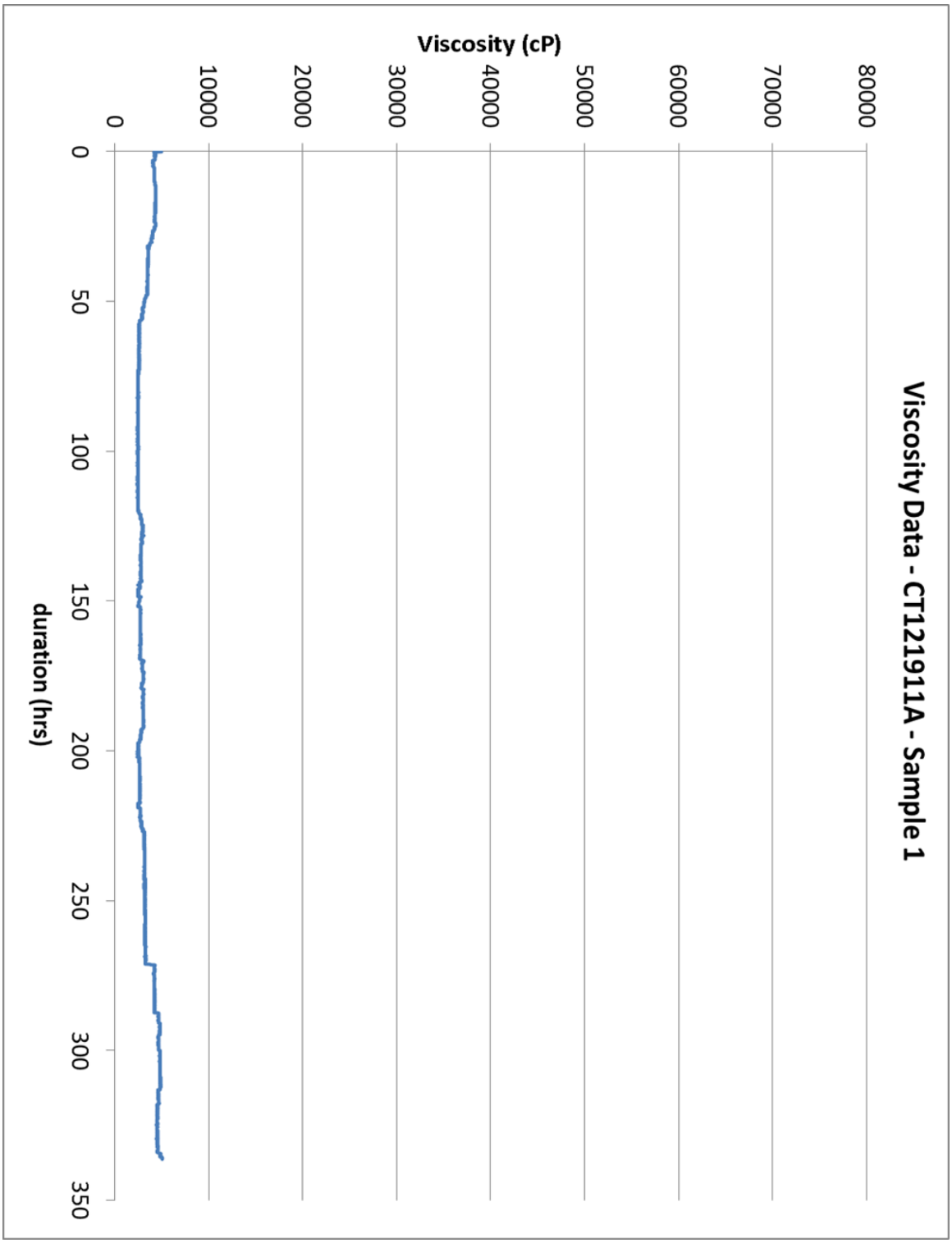


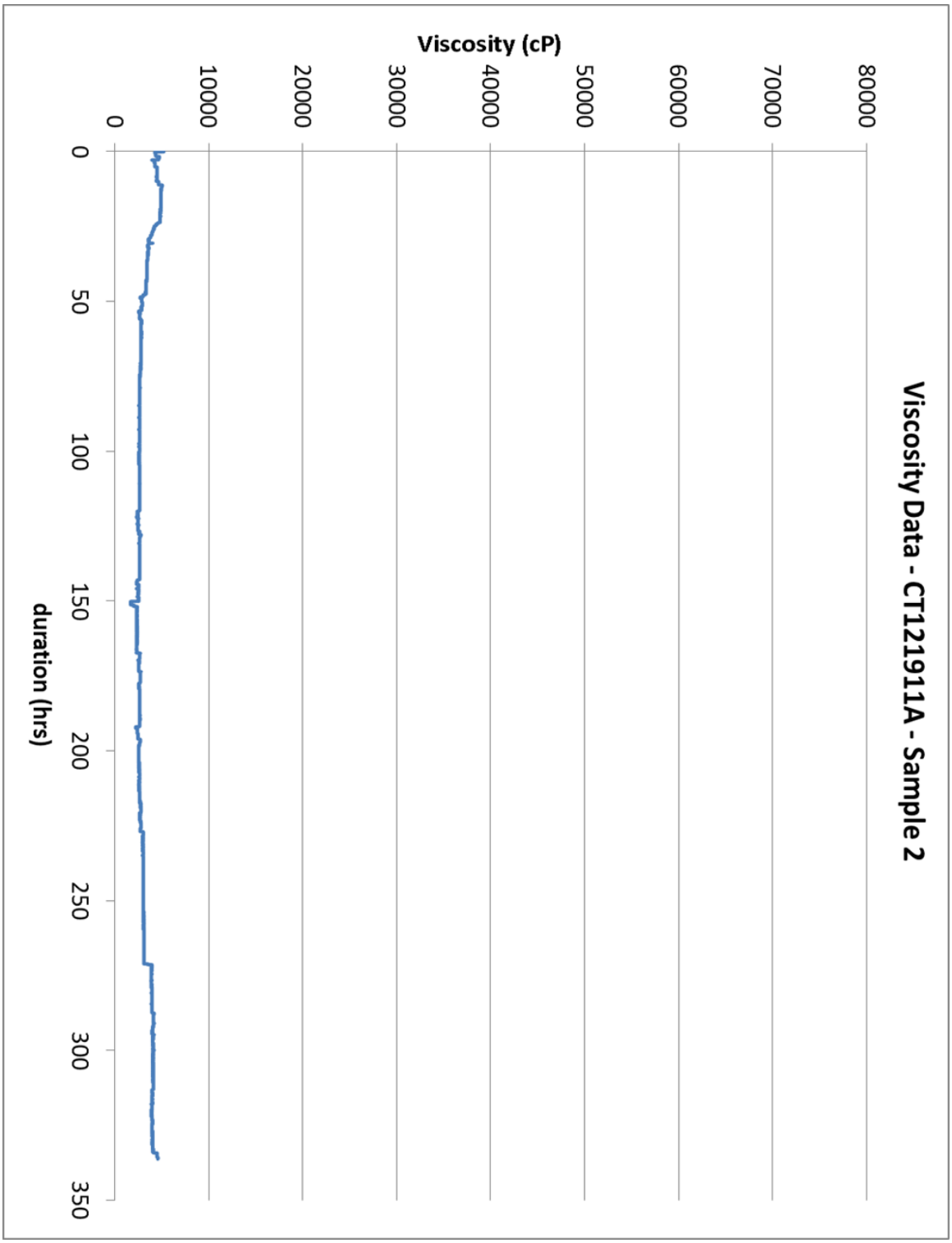

Viscosity Data - Fenuron Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

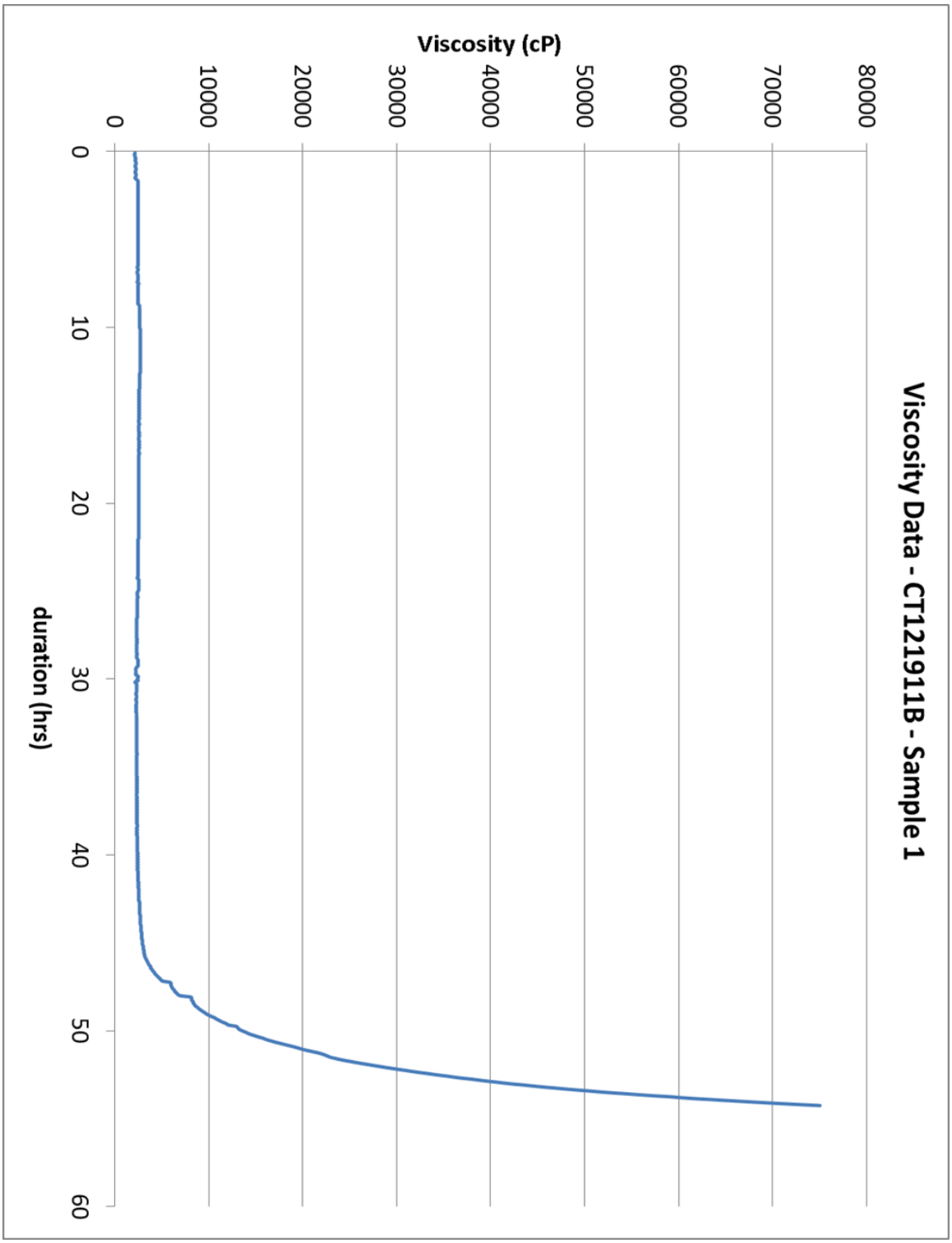

Viscosity Data - Fenuron - Sample 3
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

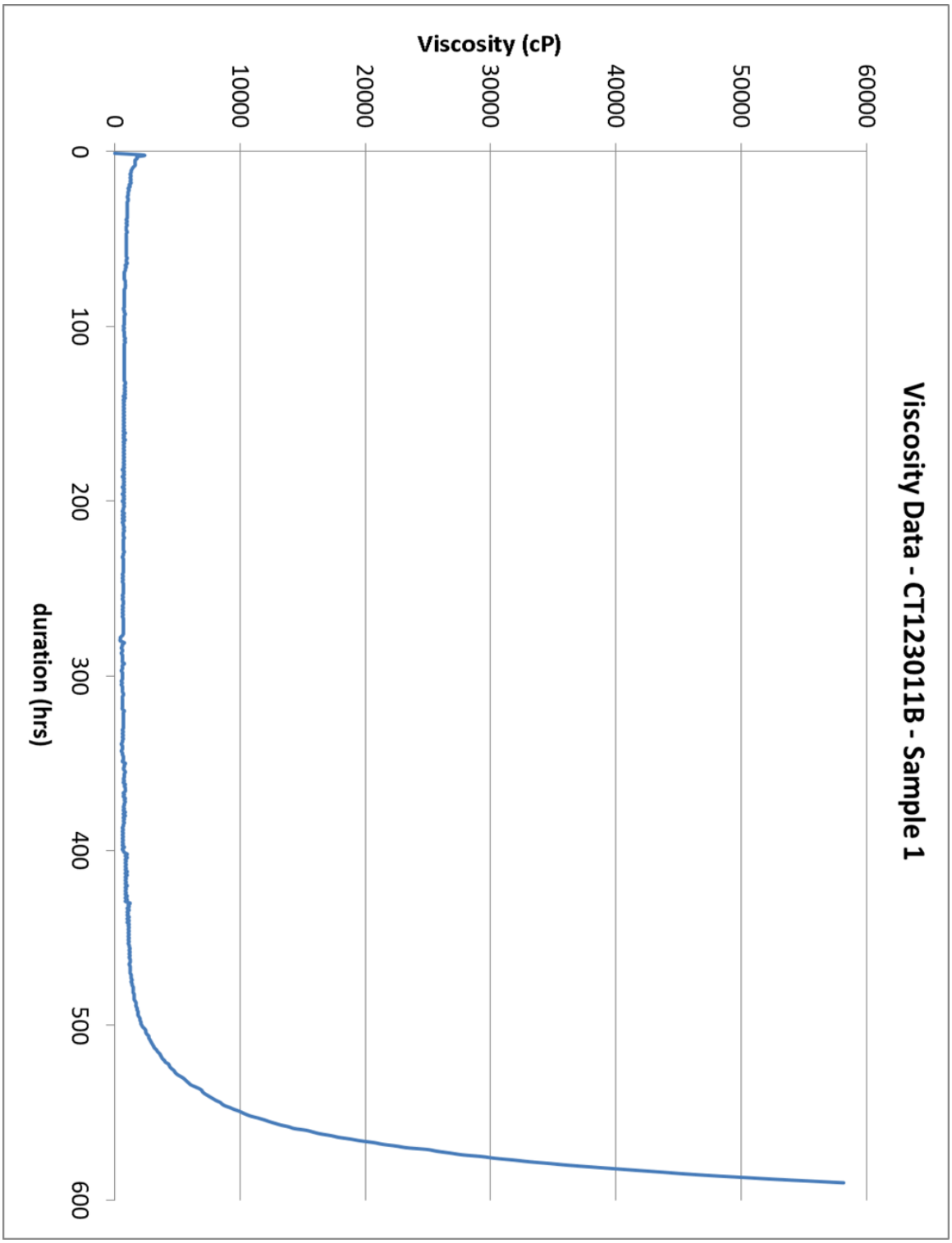

Viscosity Data - CT112211B - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
20
40
60
80
100
120
140
160
duration (hrs)

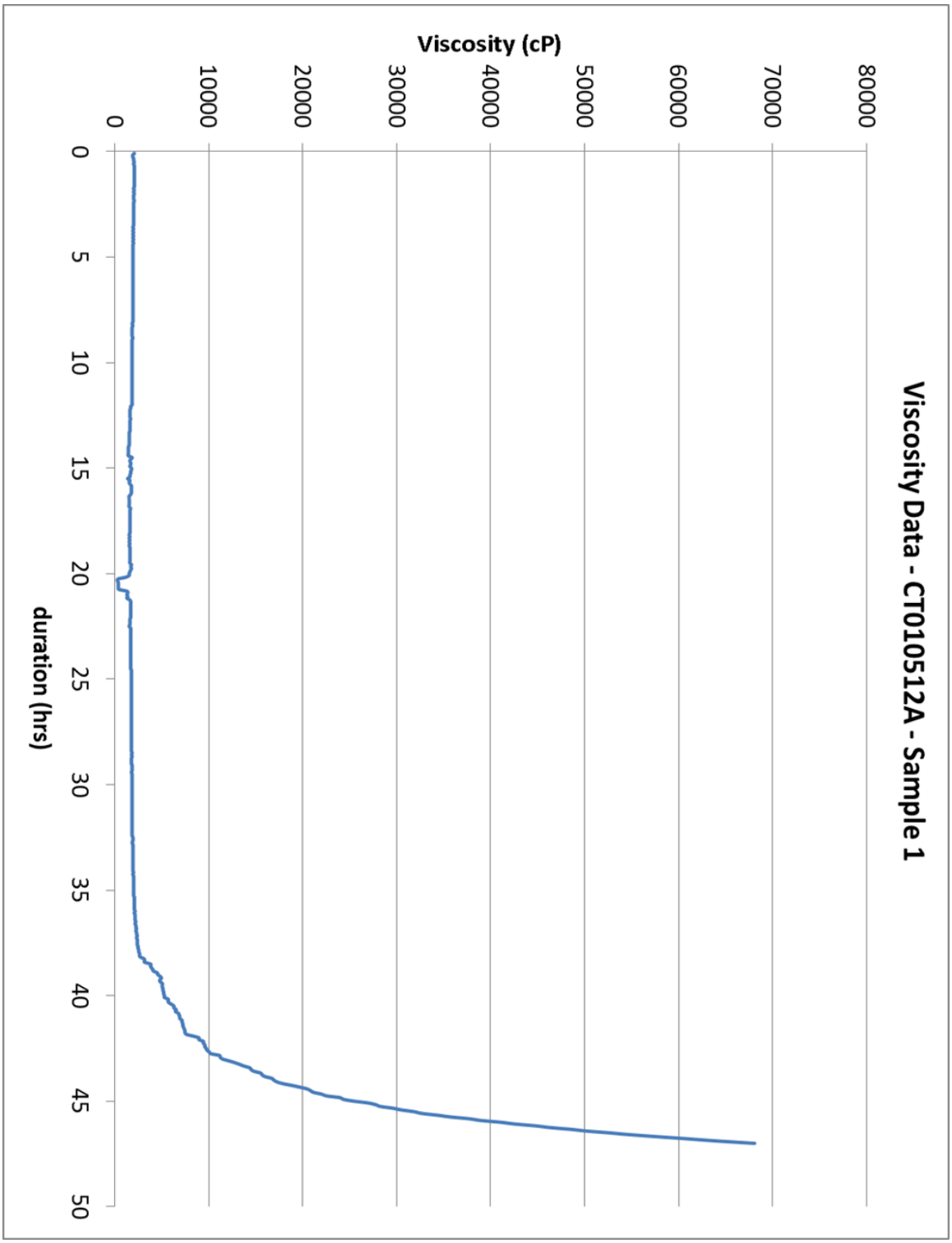

Viscosity Data - CT112211B - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
20
40
60
80
100
120
140
160
duration (hrs)

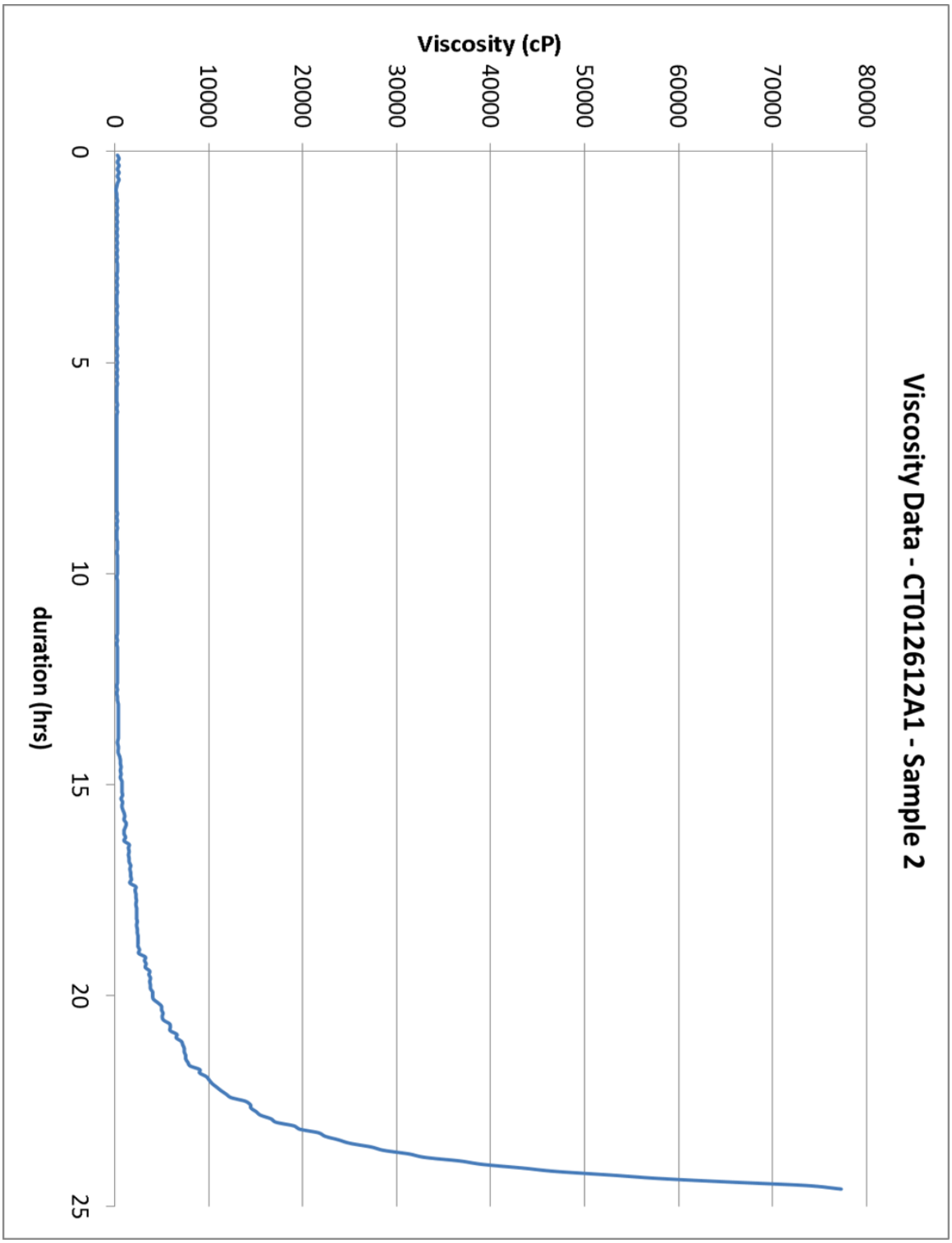

Viscosity Data - CT121911A - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
300
350
duration (hrs)

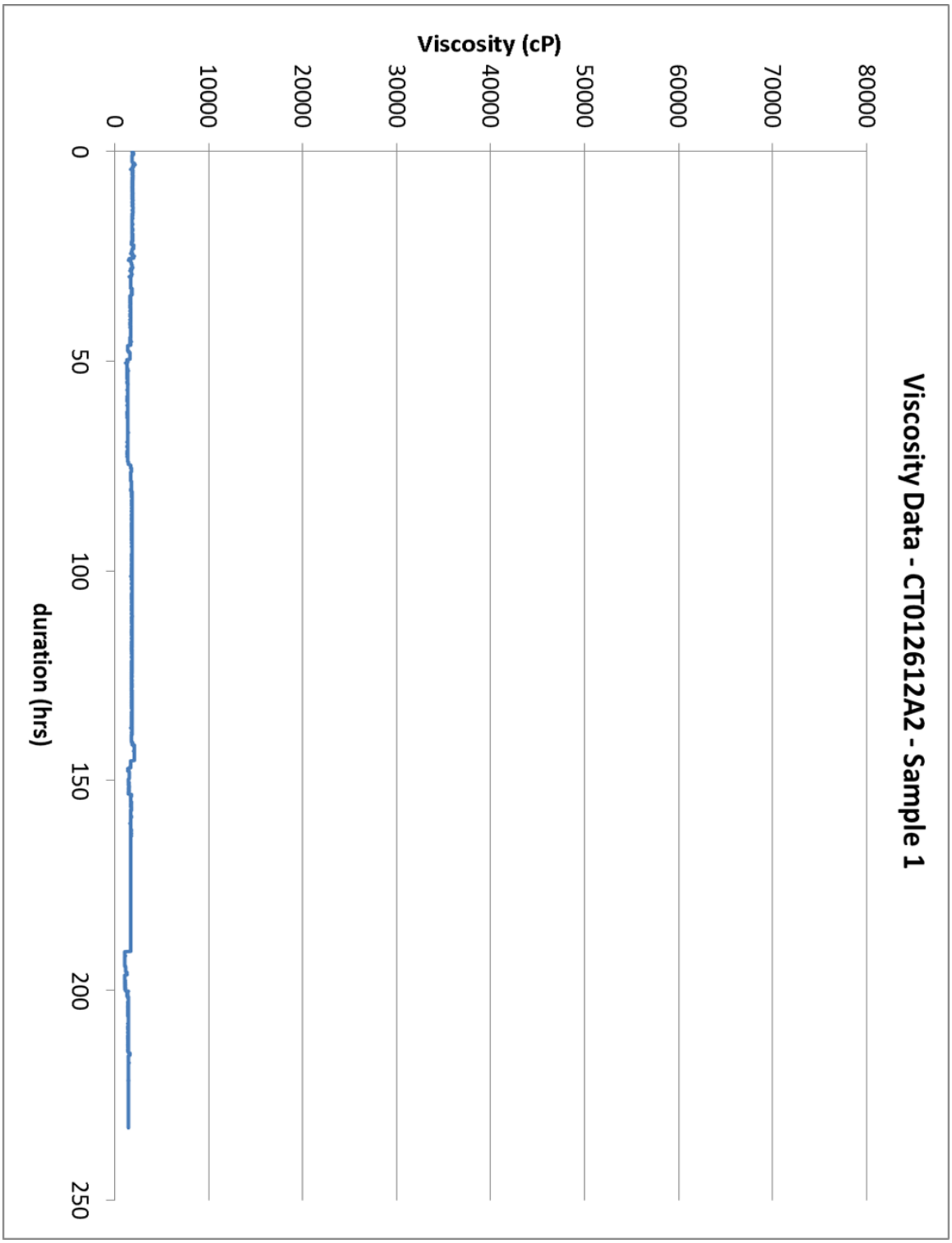

Viscosity Data - CT121911A - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
300
350
duration (hrs)

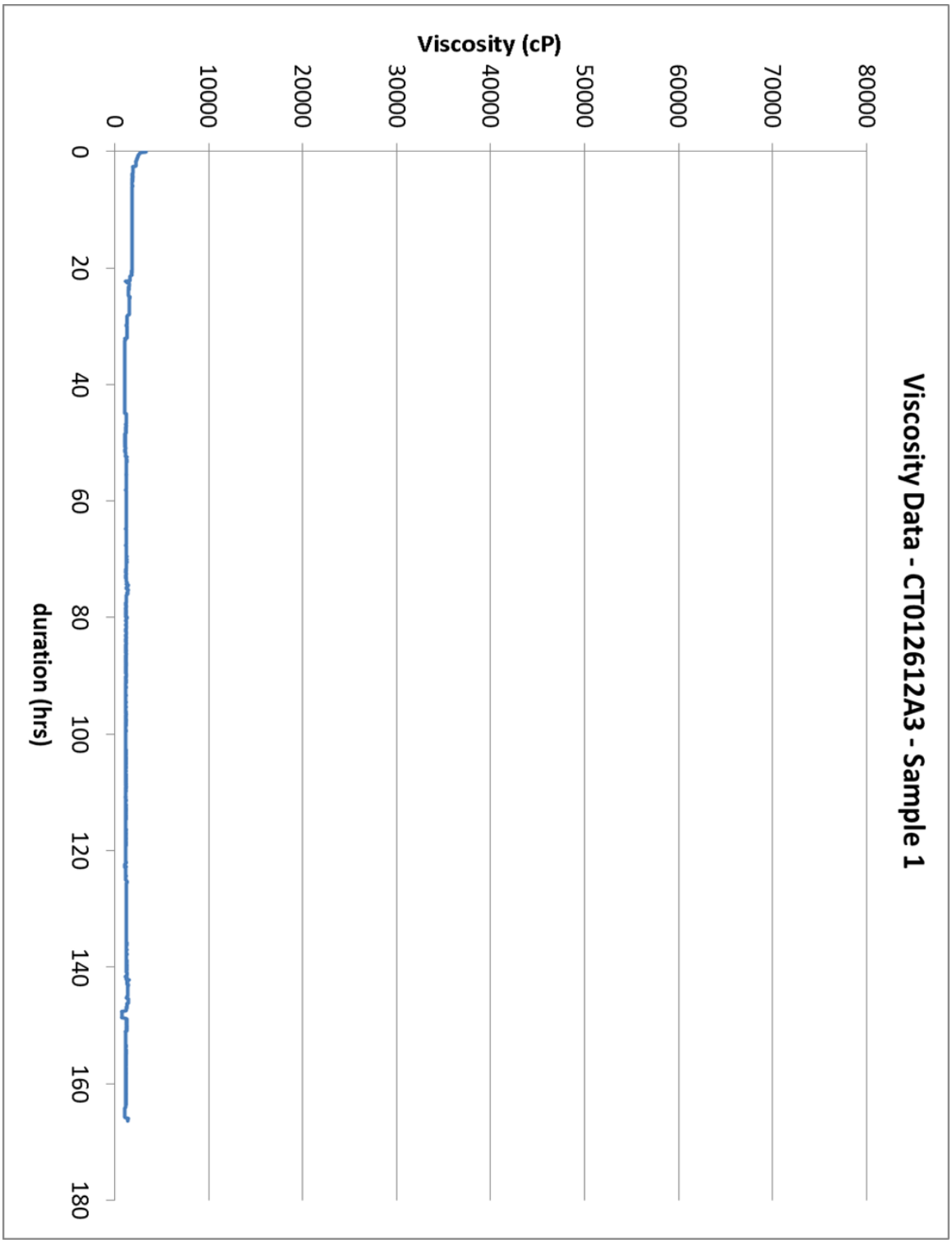

Viscosity Data - CT121911B - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
10
20
30
40
50
60
duration (hrs)

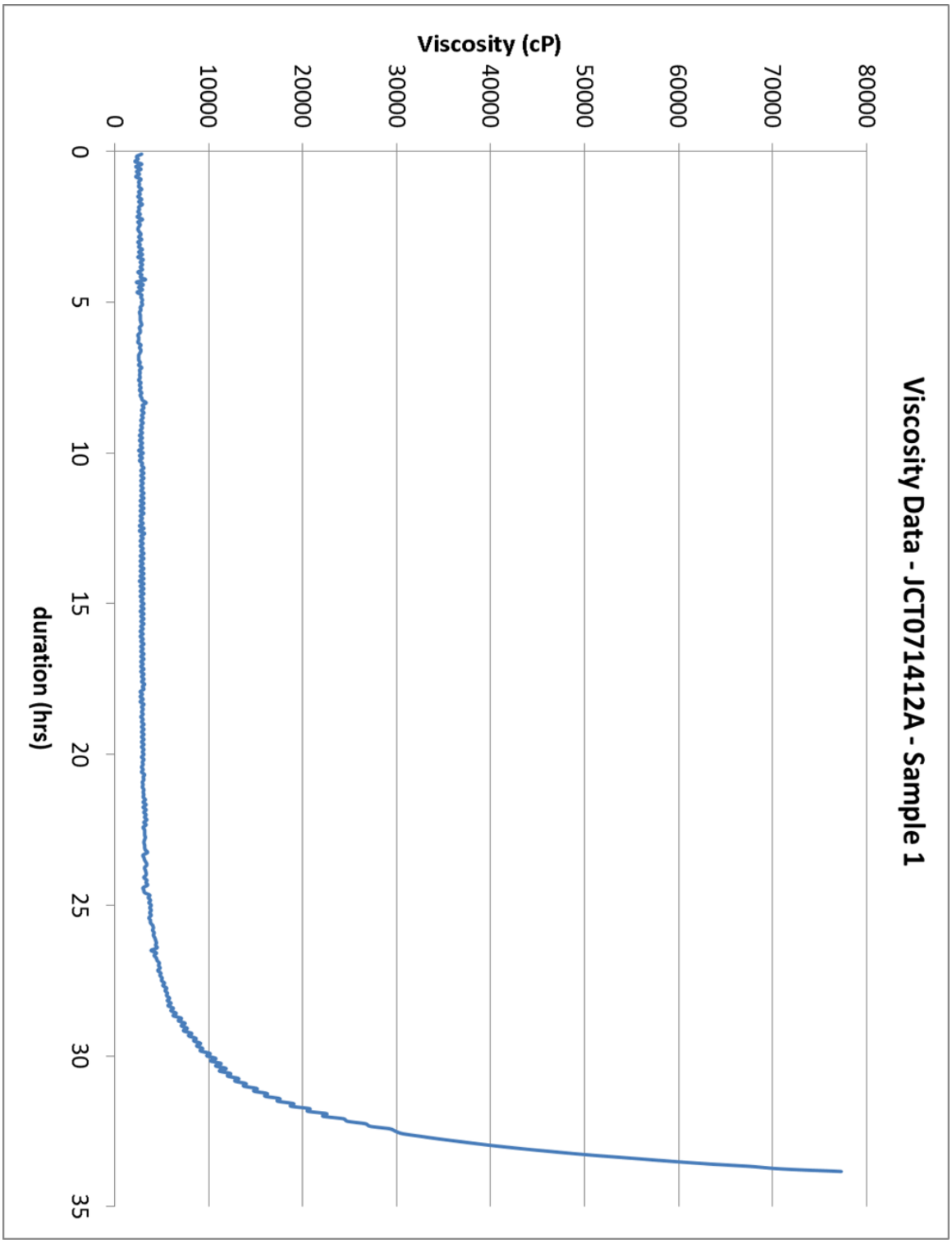

Viscosity Data - CT123011B - Sample 1
Viscosity (cP)
60000
50000
40000
30000
20000
10000
0
0
100
200
300
400
500
600
duration (hrs)

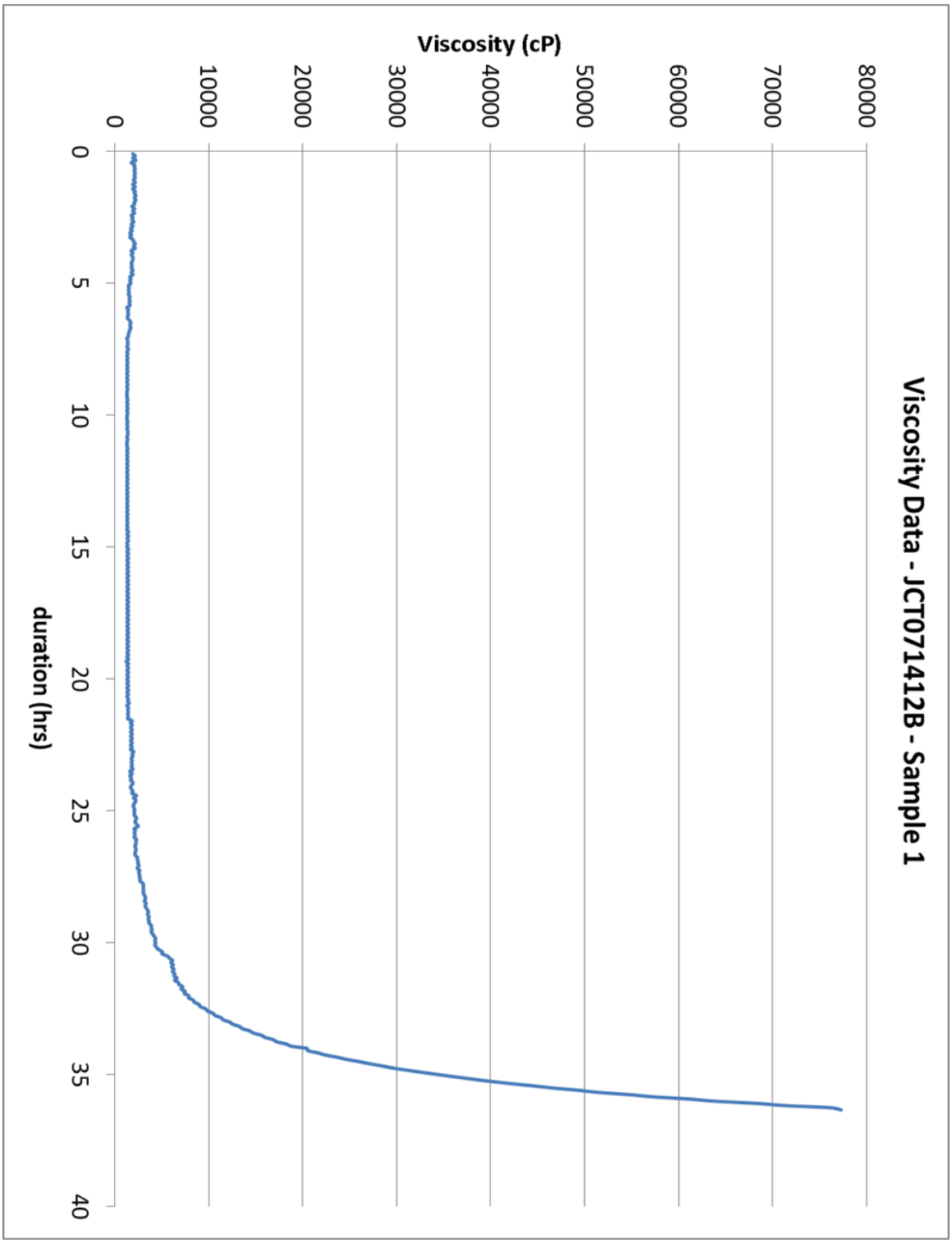

Viscosity Data - CT010512A - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
45
50
duration (hrs)

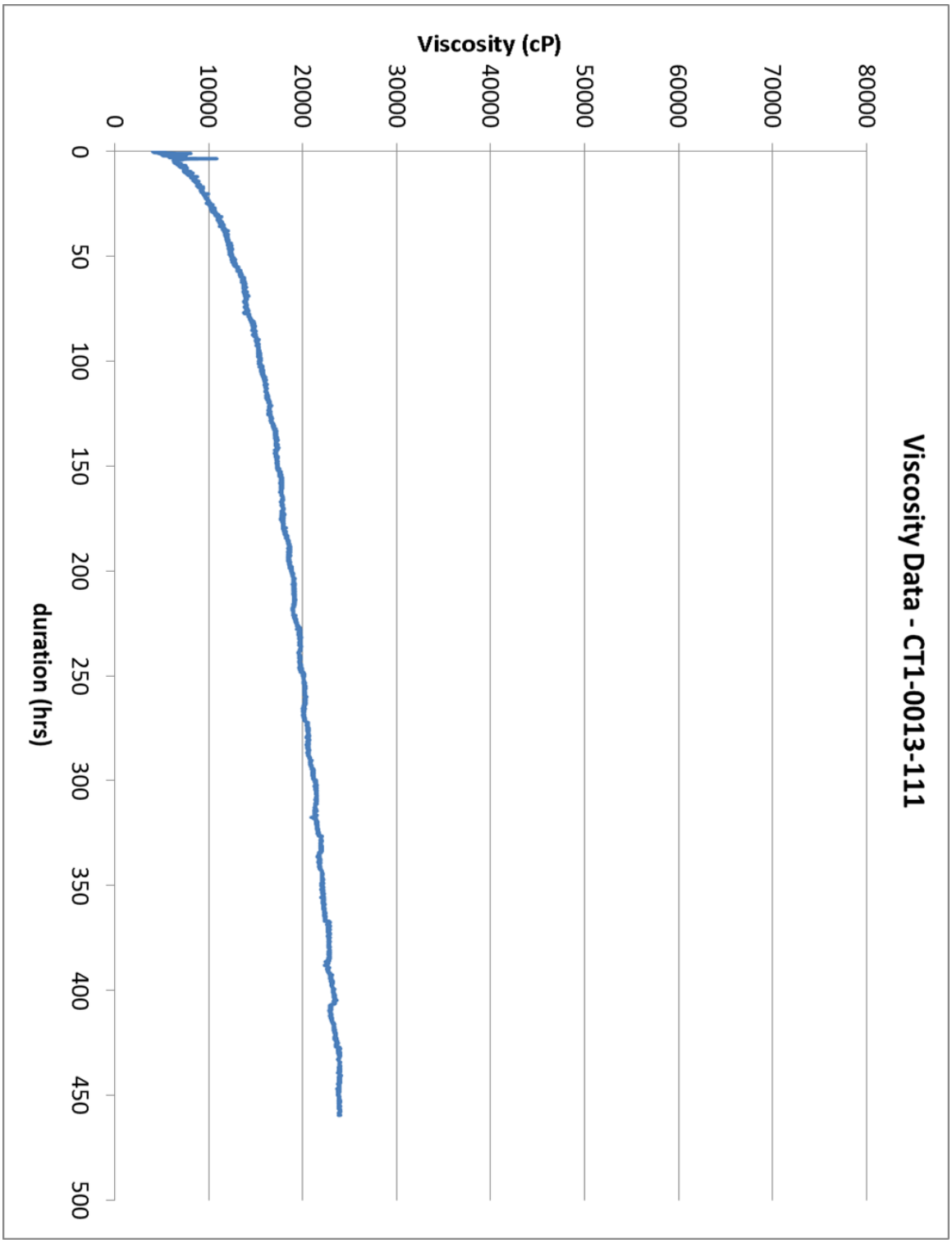

Viscosity Data - CT012612A1 - Sample 2
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
duration (hrs)

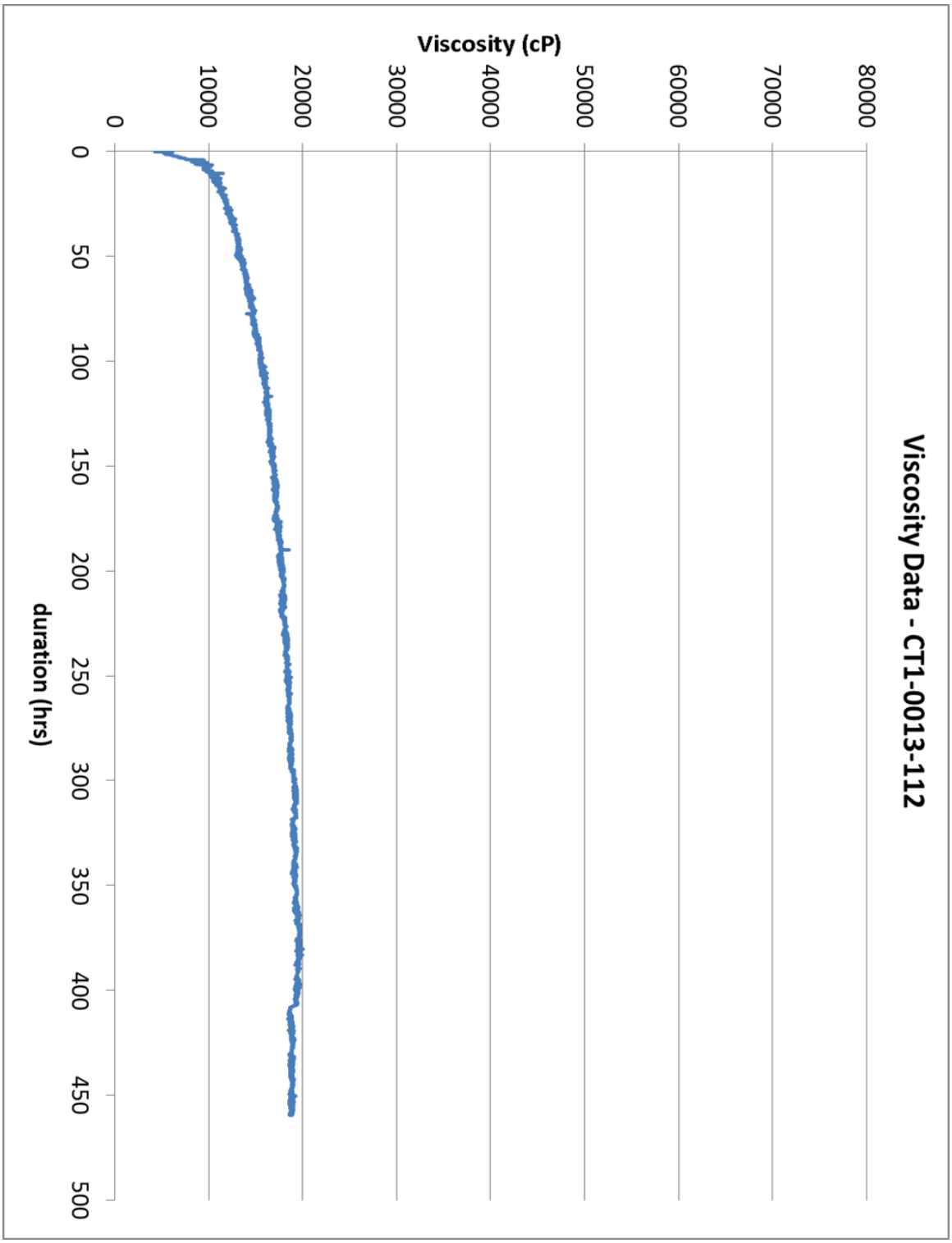

Viscosity Data - CT012612A2 - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
duration (hrs)

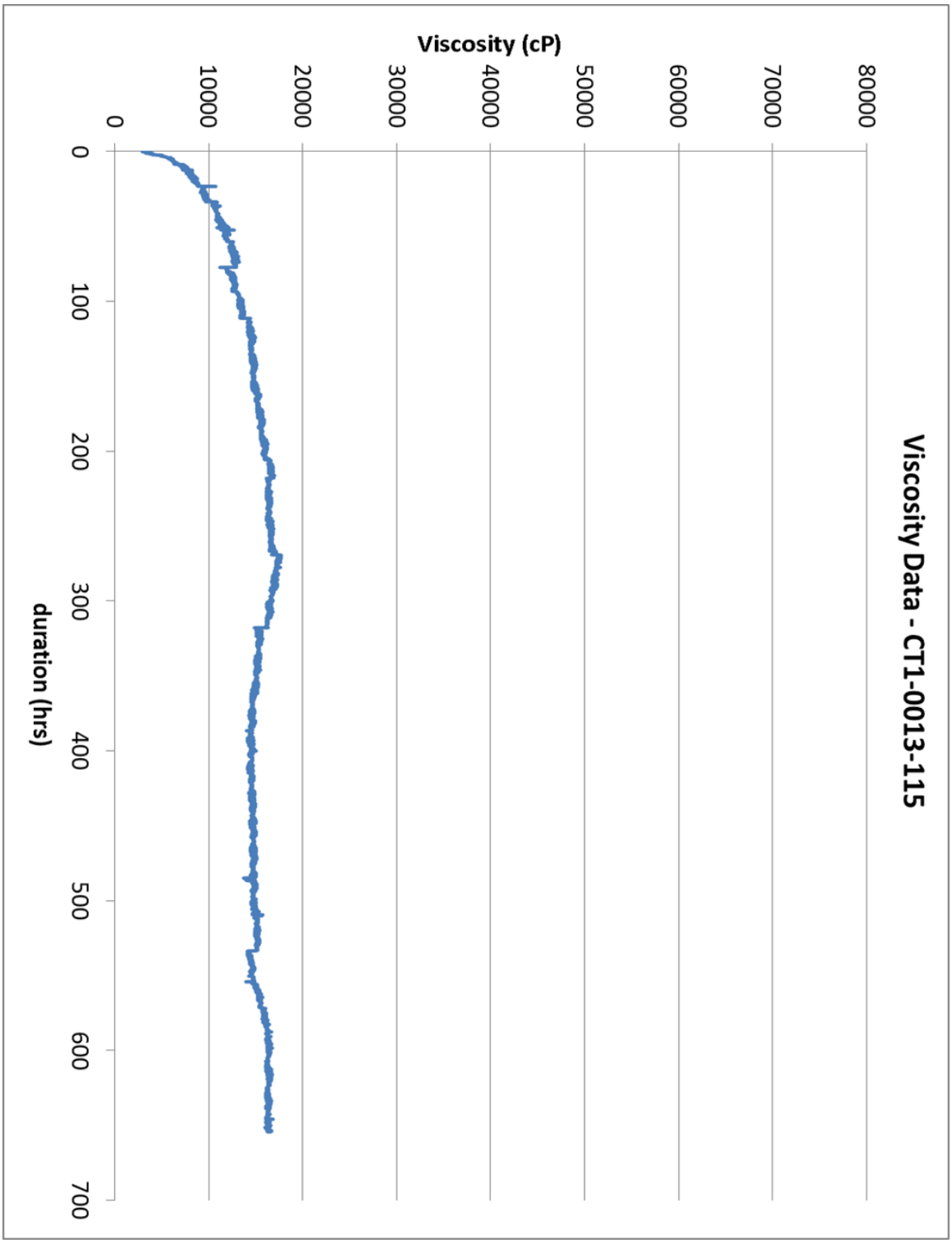

Viscosity Data - CT012612A3 - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
20
40
60
80
100
120
140
160
180
duration (hrs)


Viscosity Data - JCT071412A - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
duration (hrs)

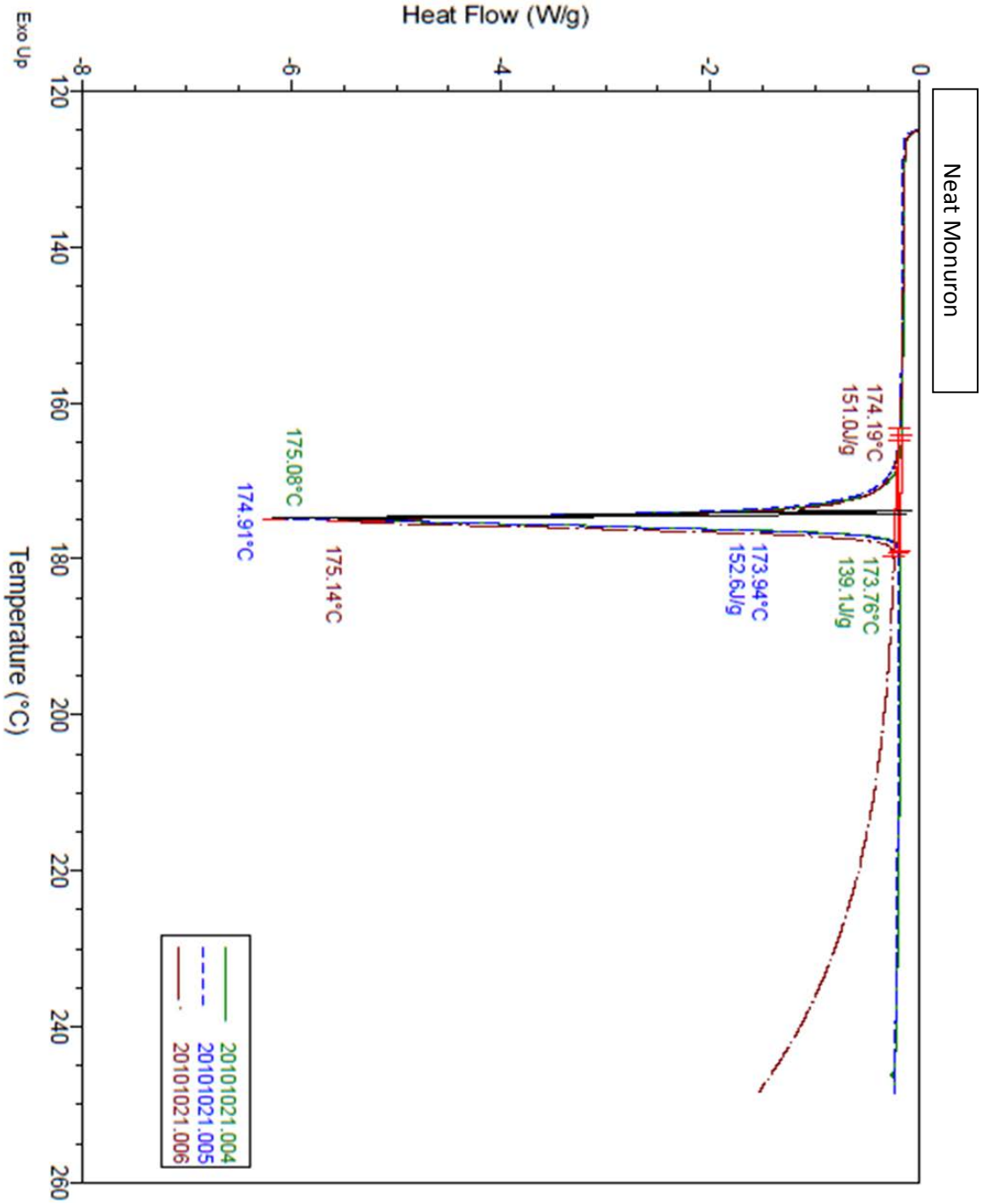

Viscosity Data - JCT071412B - Sample 1
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
5
10
15
20
25
30
35
40
duration (hrs)

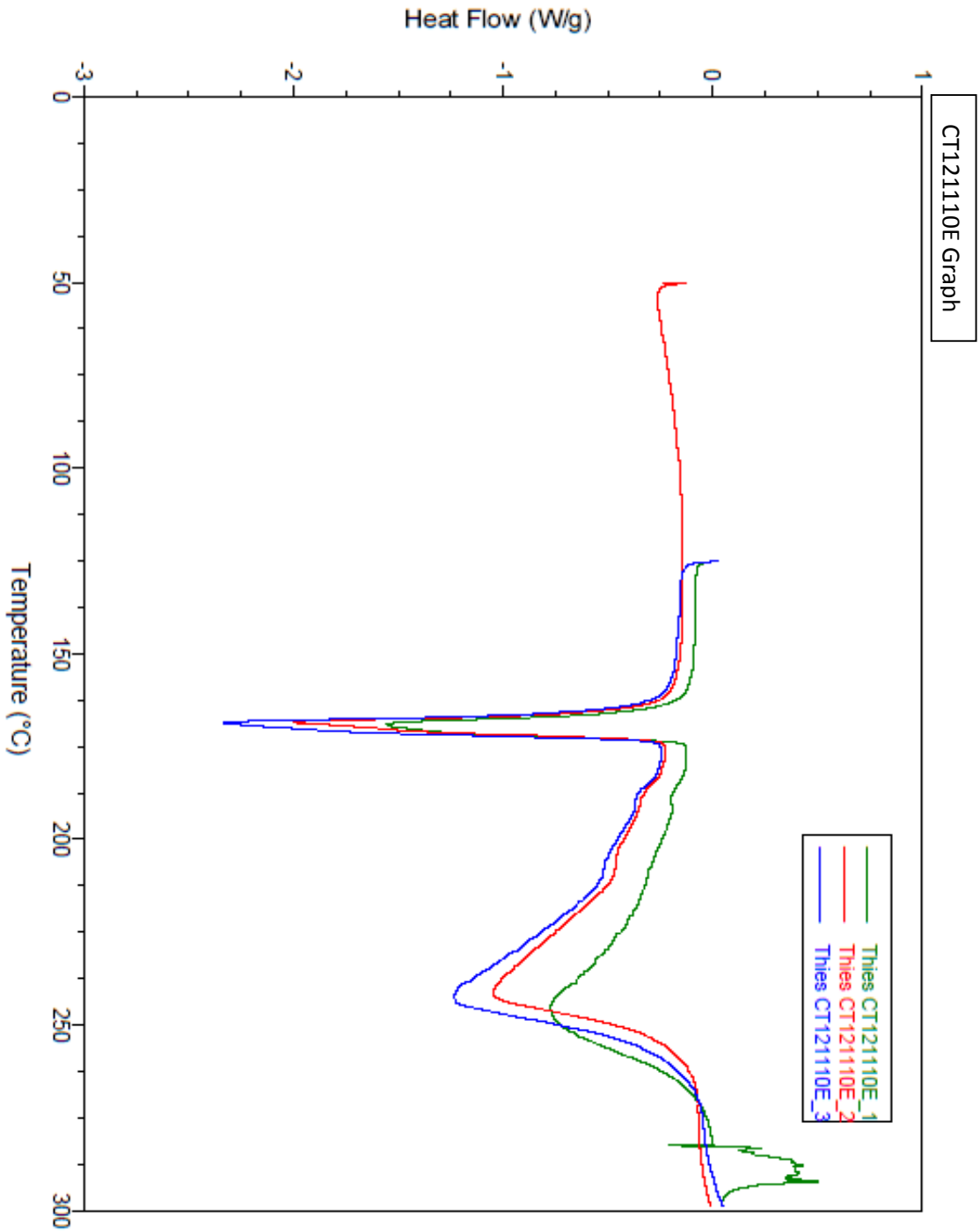

Viscosity Data - CT1-0013-111
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
300
350
400
450
500
duration (hrs)

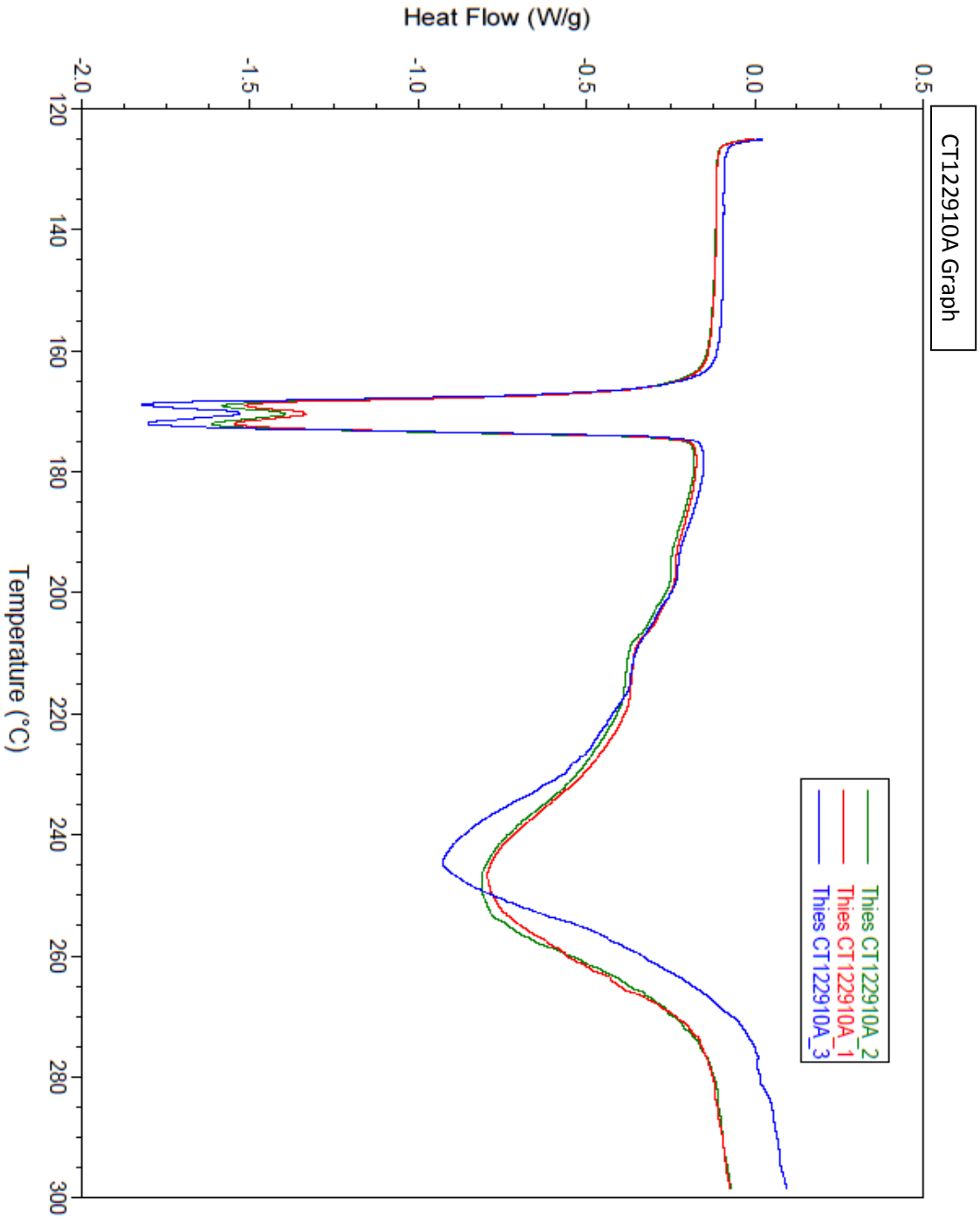

Viscosity Data - CT1-0013-112
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
300
350
400
450
500
duration (hrs)

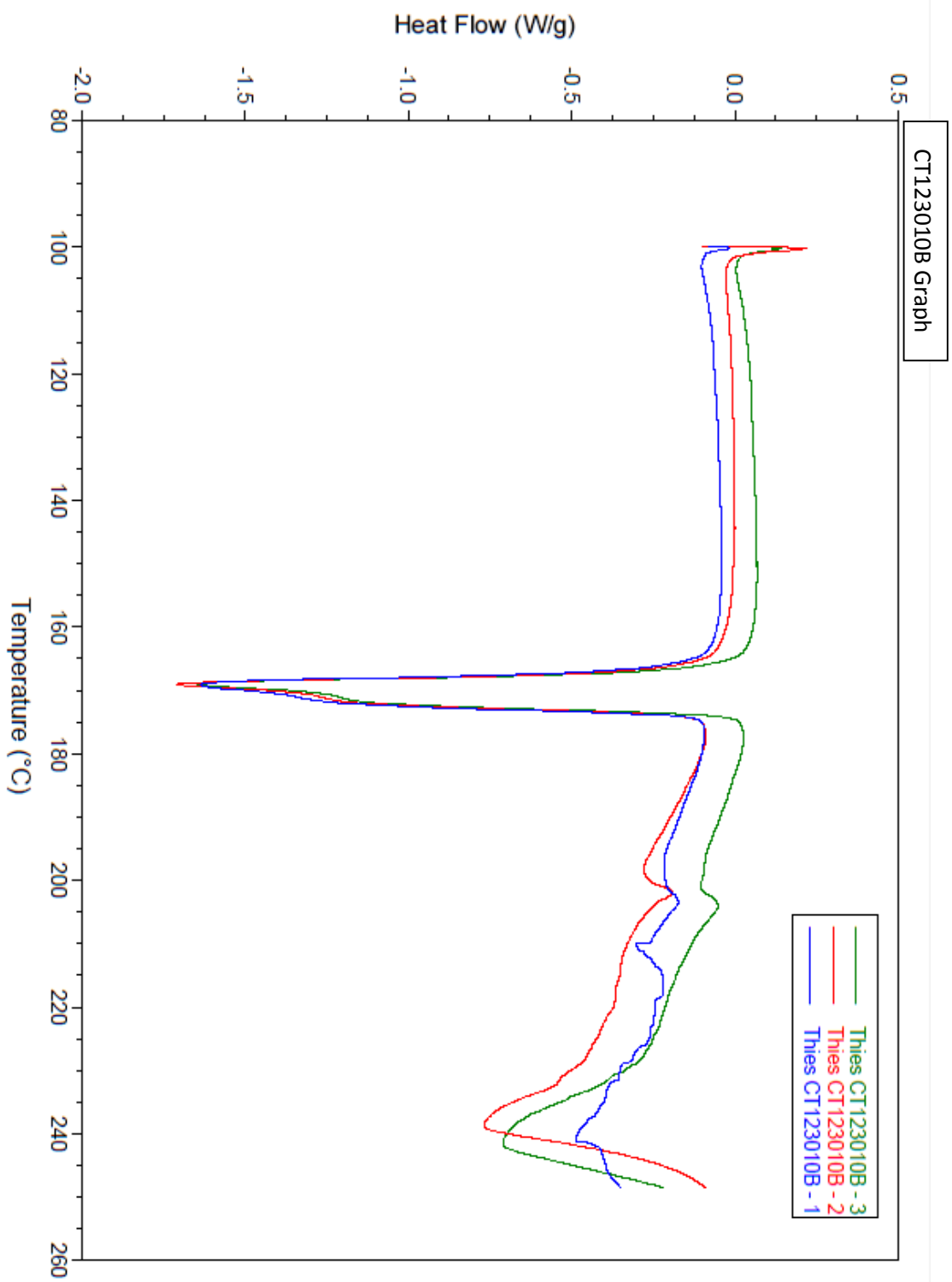

Viscosity Data - CT1-0013-115
Viscosity (cP)
80000
70000
60000
50000
40000
30000
20000
10000
0
0
100
200
300
400
500
600
700
duration (hrs)

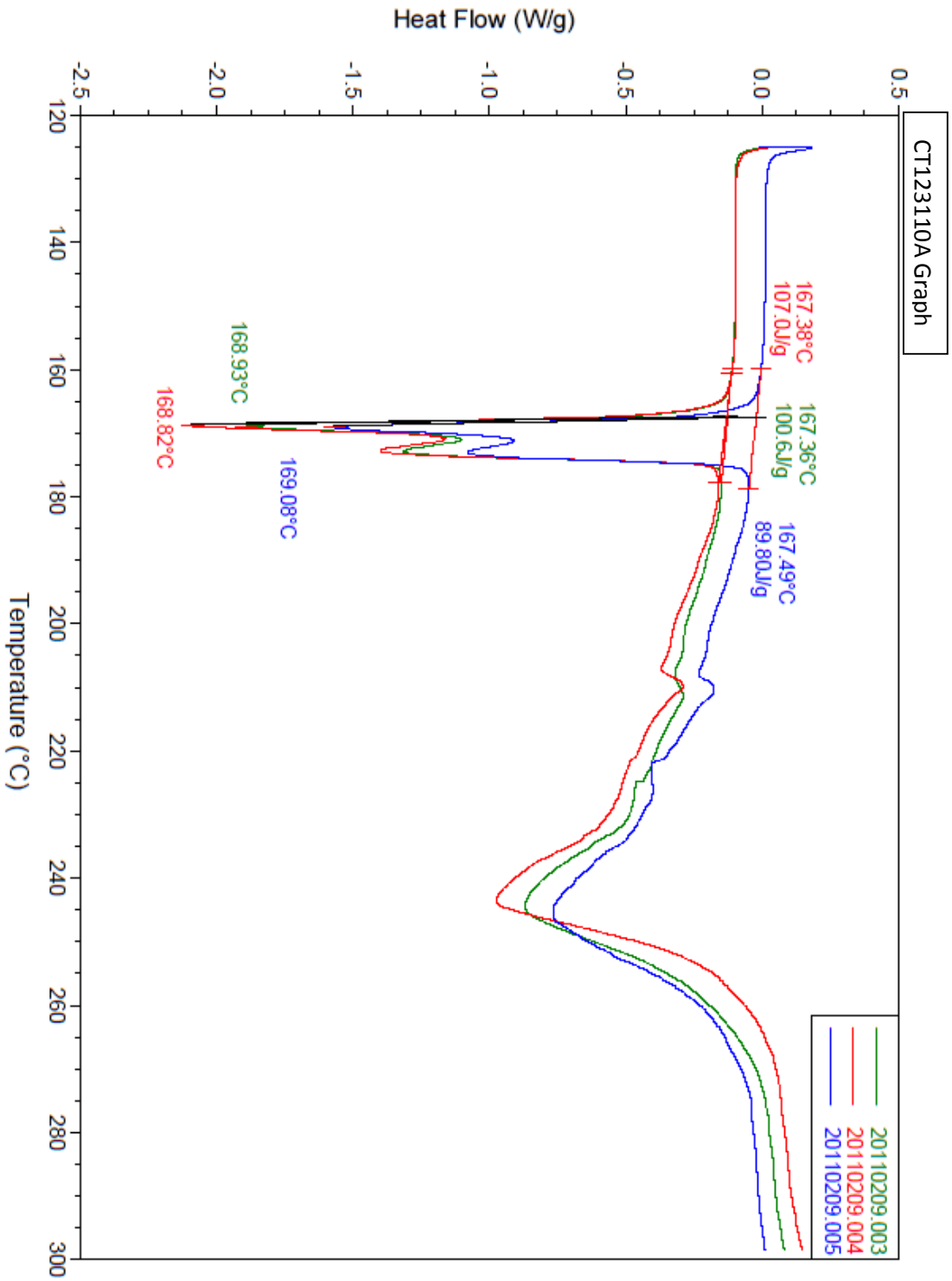
Appendix 3A: DSC Data for Monuron Based Samples

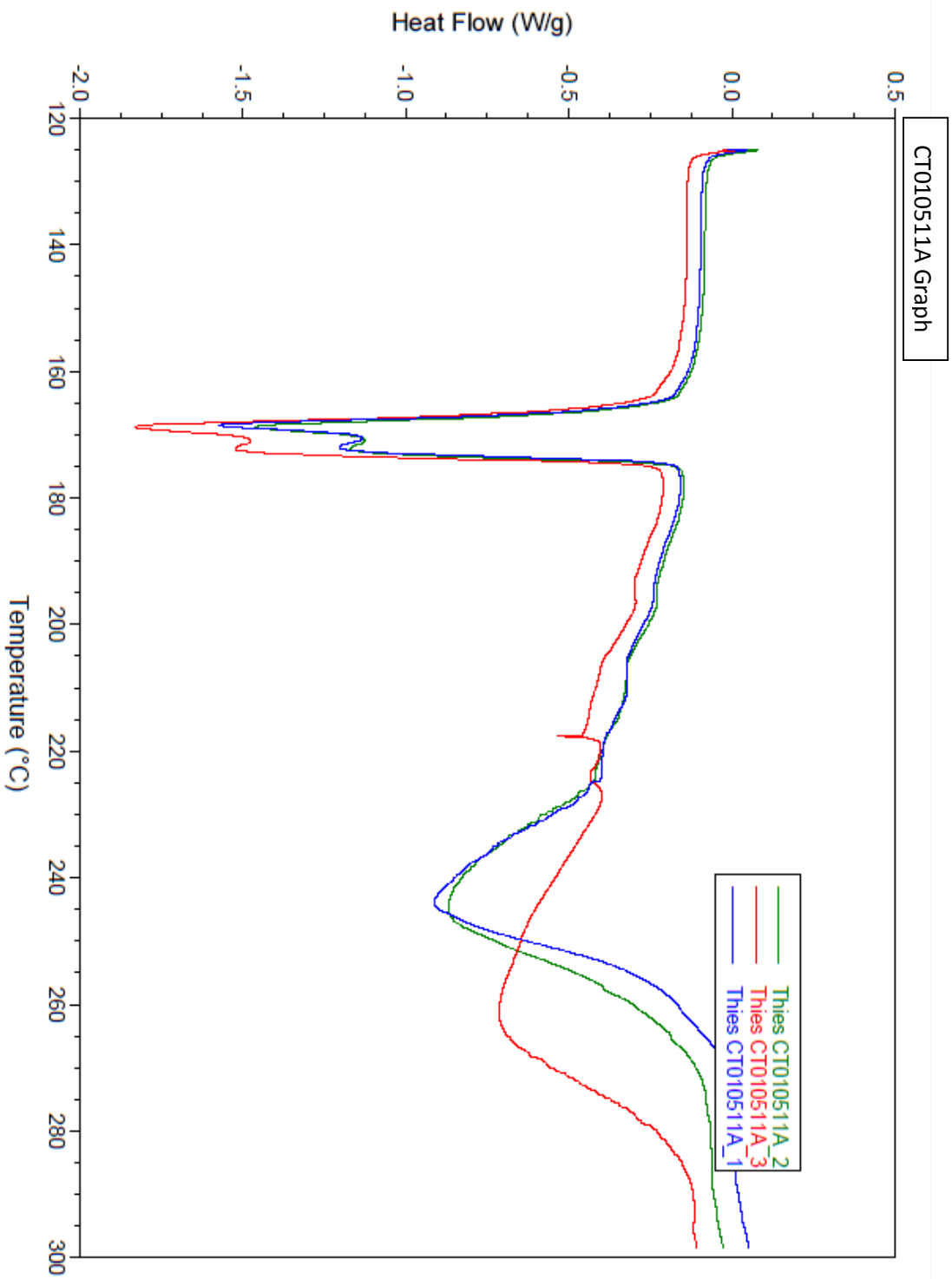


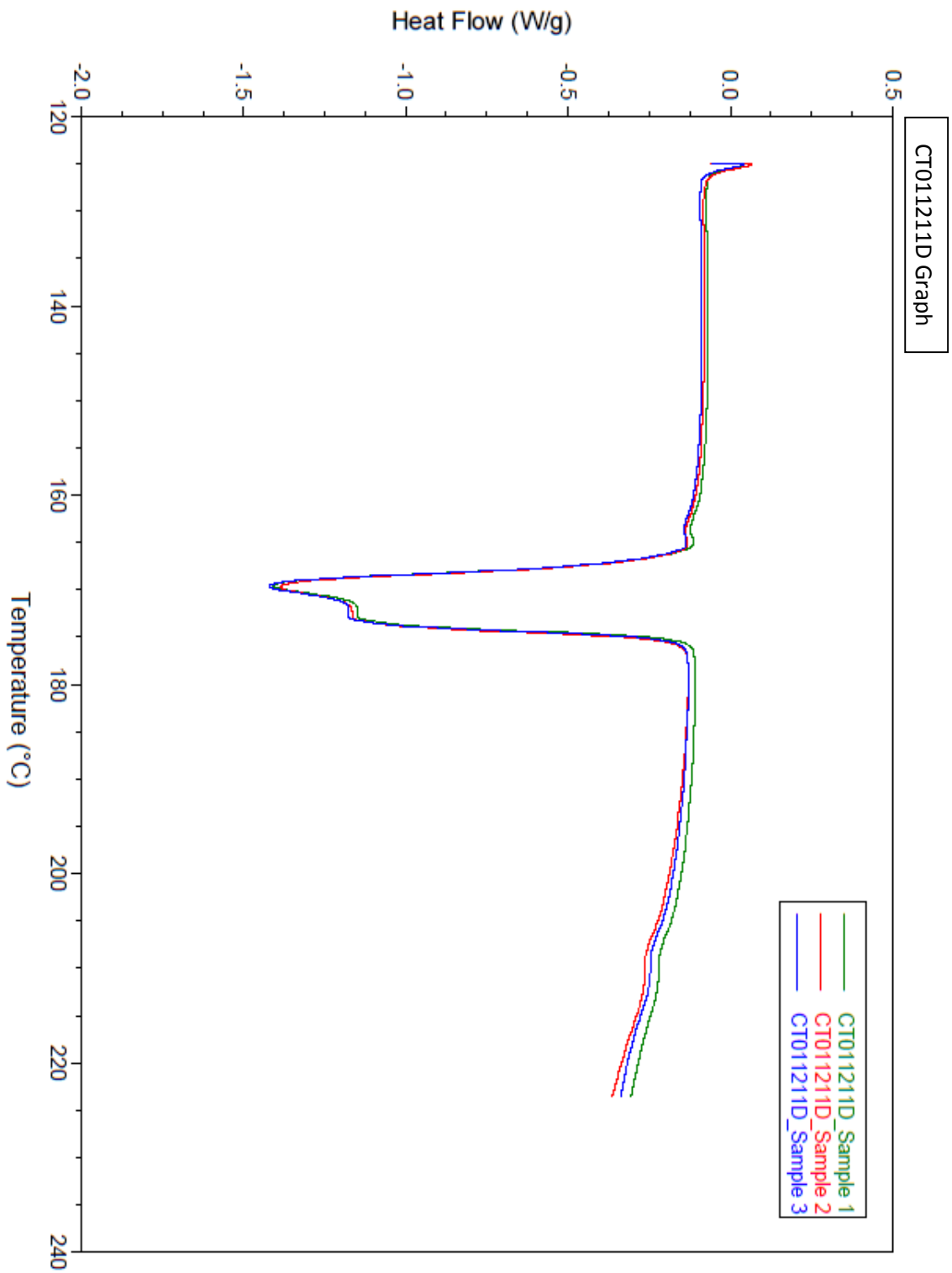

CT121110E Graph
Thies CT121110E_1
Thies CT121110E_2
Thies CT121110E_3
Heat Flow (W/g)
1
0
-1
-2
-3
Temperature (°C)
0
50
100
150
200
250
300

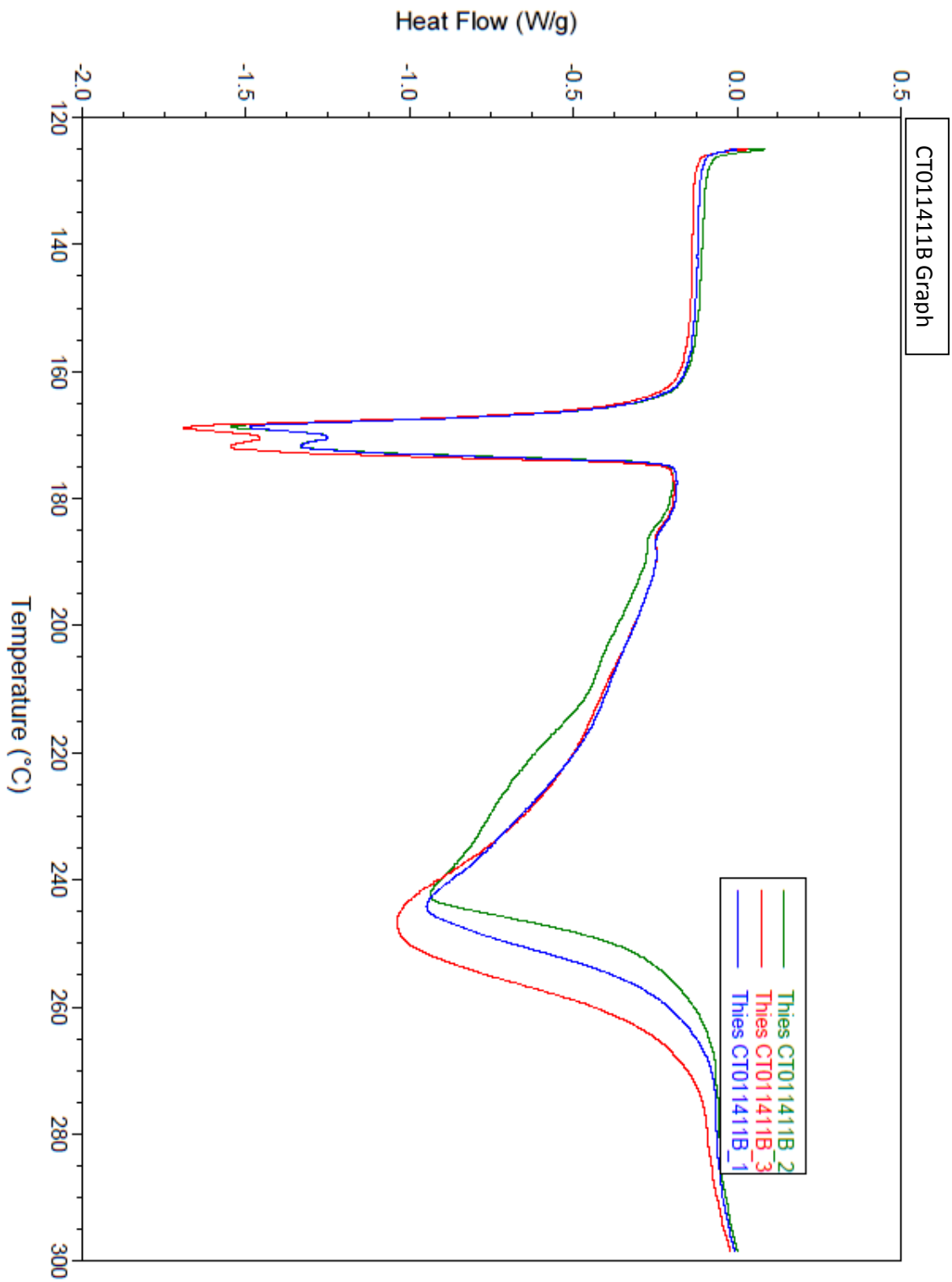

CT122910A Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
Temperature (°C)
120
140
160
180
200
220
240
260
280
300
Thies CT122910A_2
Thies CT122910A_1
Thies CT122910A_3

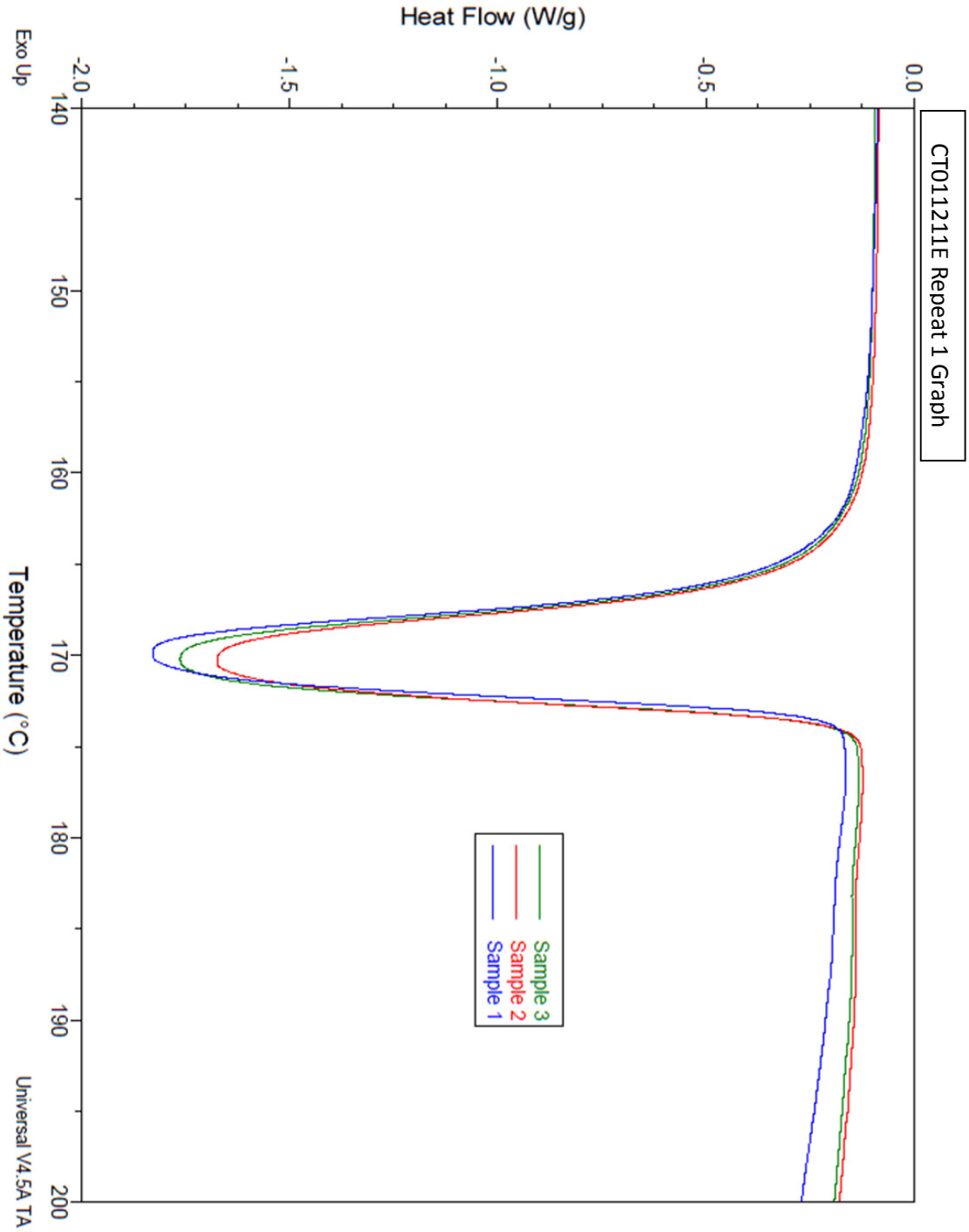

CT123010B Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
80
100
120
140
160
180
200
220
240
260
Temperature (°C)
Thies CT123010B - 3
Thies CT123010B - 2
Thies CT123010B - 1

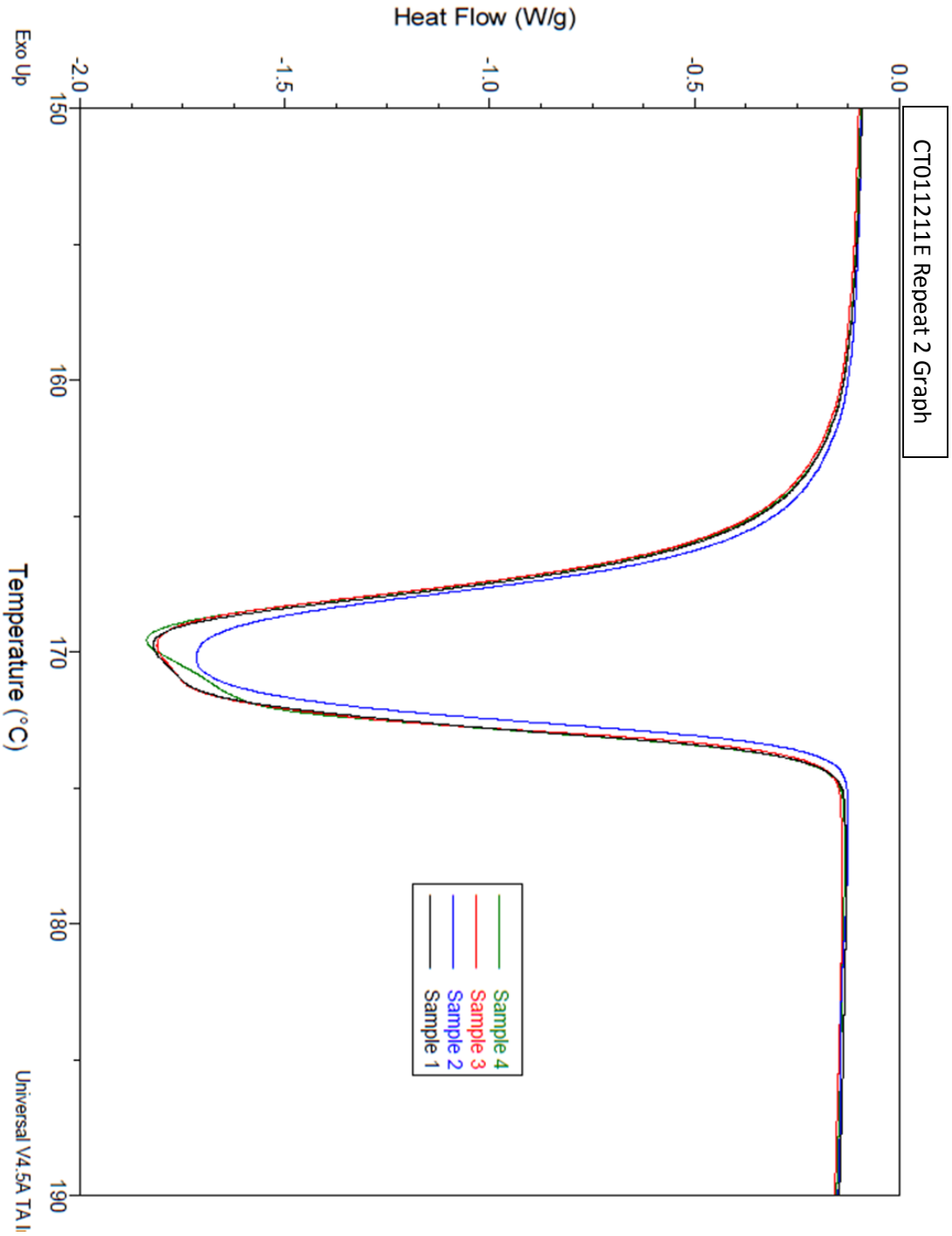

CT123110A Graph
20110209.003
20110209.004
20110209.005
167.38°C
107.0J/g
167.36°C
100.6J/g
167.49°C
89.80J/g
168.93°C
168.82°C
169.08°C
Heat Flow (W/g)
Temperature (°C)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
-2.5
120
140
160
180
200
220
240
260
280
300

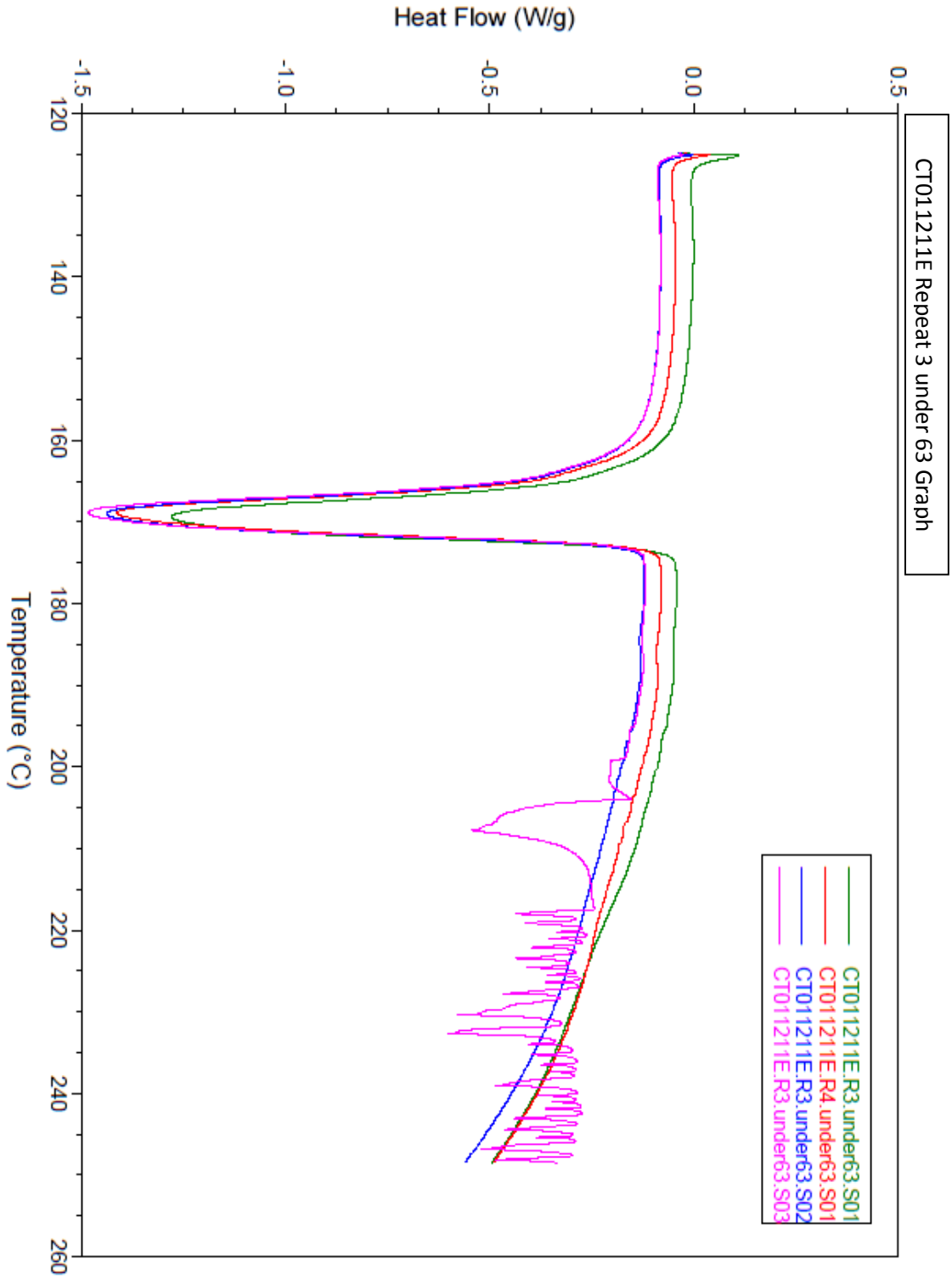

CT010511A Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
120
140
160
180
200
220
240
260
280
300
Temperature (°C)
Thies CT010511A_2
Thies CT010511A_3
Thies CT010511A_1

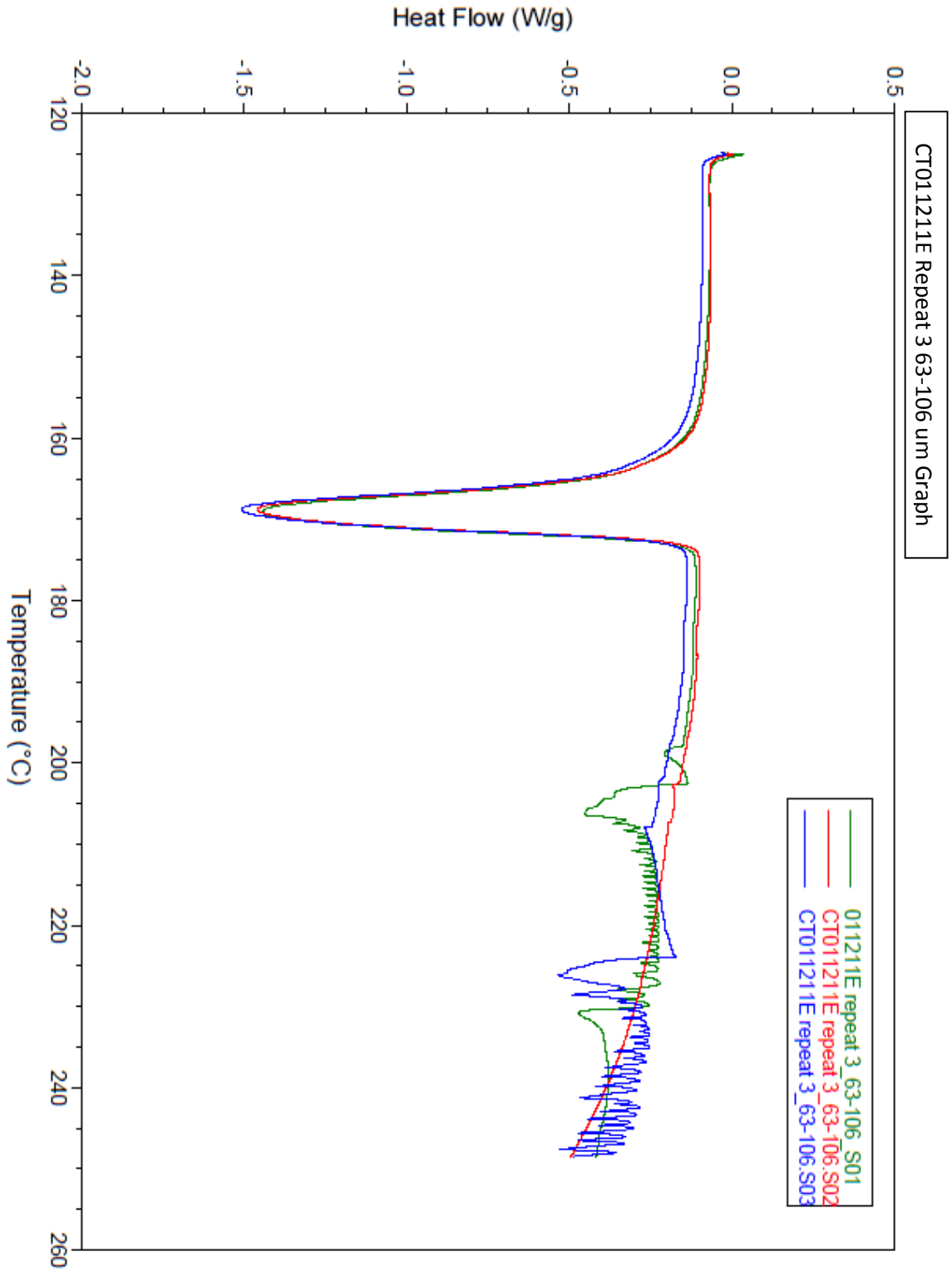

CT011211D Graph
CT011211D_Sample 1
CT011211D_Sample 2
CT011211D_Sample 3
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
120
140
160
180
200
220
240
Temperature (°C)

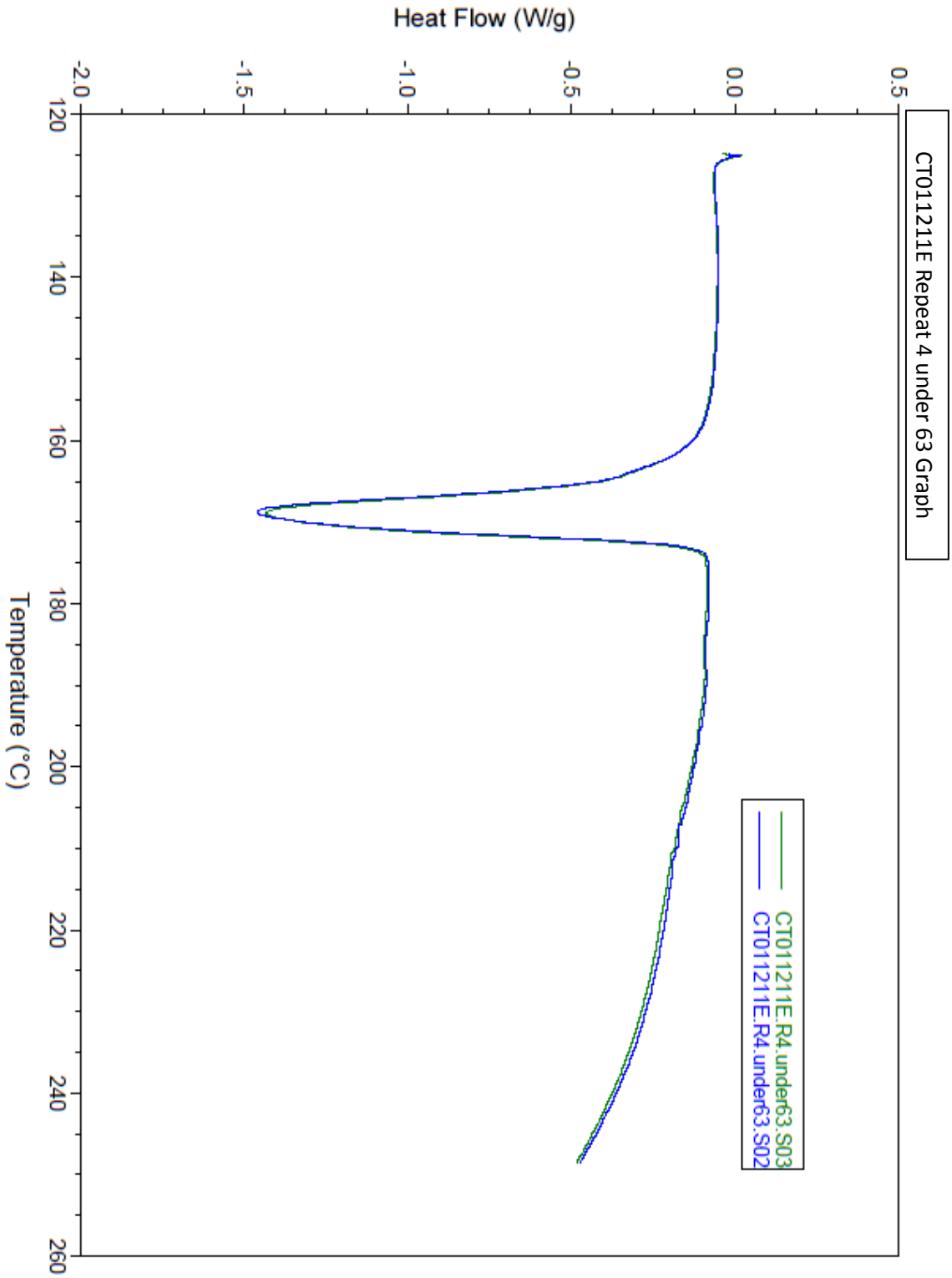

CT011411B Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
120
140
160
180
200
220
240
260
280
300
Temperature (°C)
Thies CT011411B_2
Thies CT011411B_3
Thies CT011411B_1

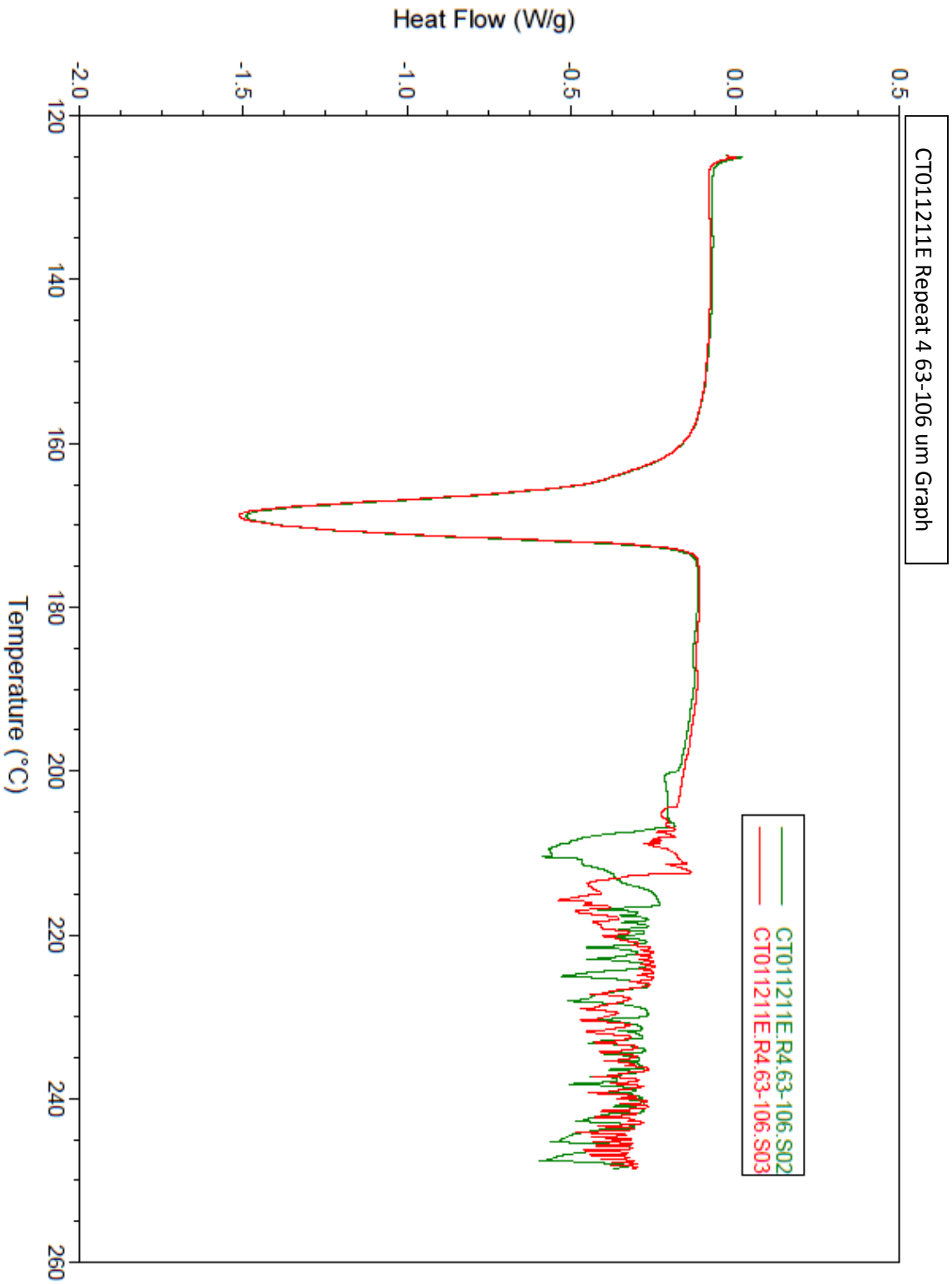

CT011211E Repeat 1 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
140
150
160
170
180
190
200
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

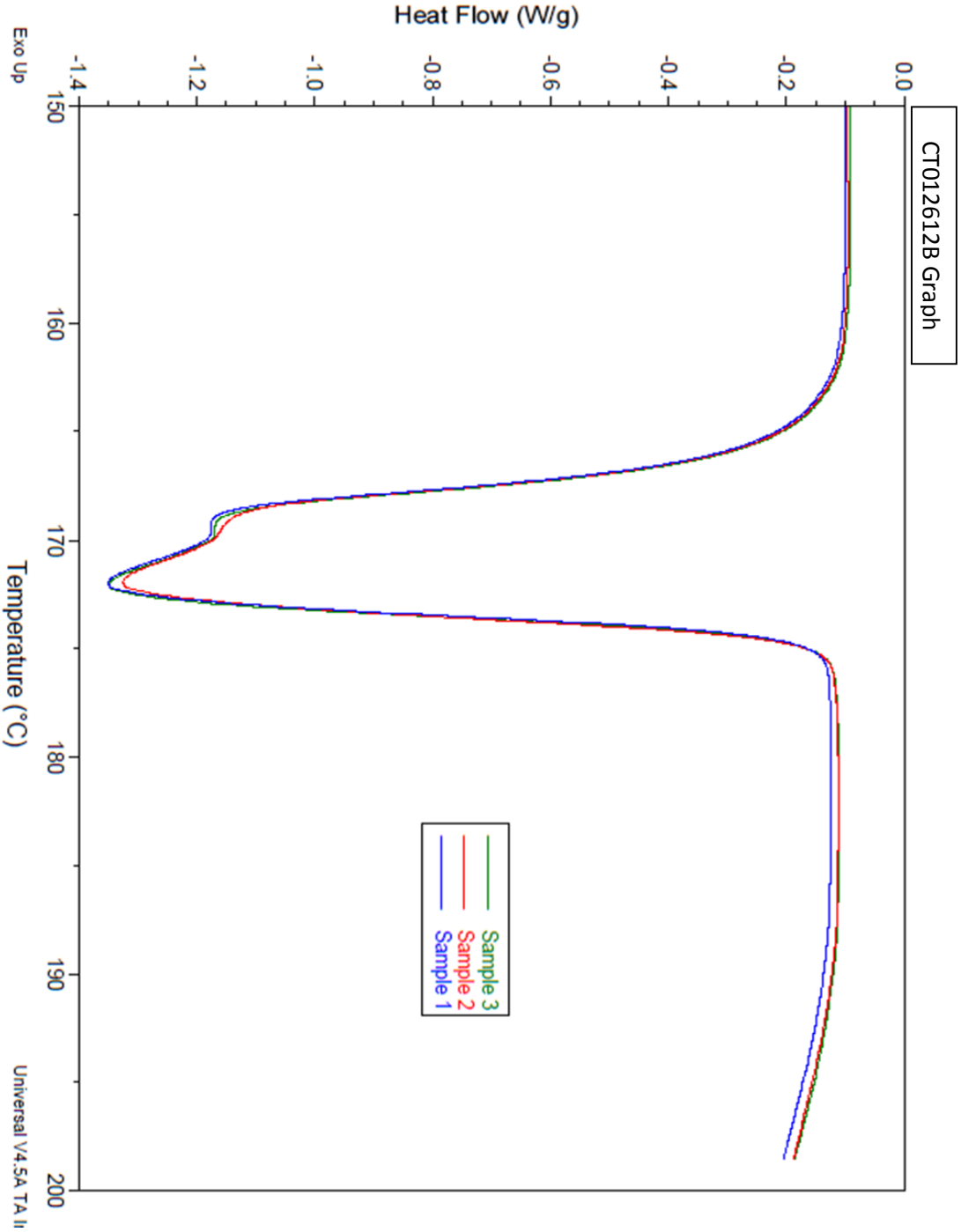

CT011211E Repeat 2 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
150
160
170
180
190
Temperature (°C)
Sample 4
Sample 3
Sample 2
Sample 1
Universal V4.5A TA I

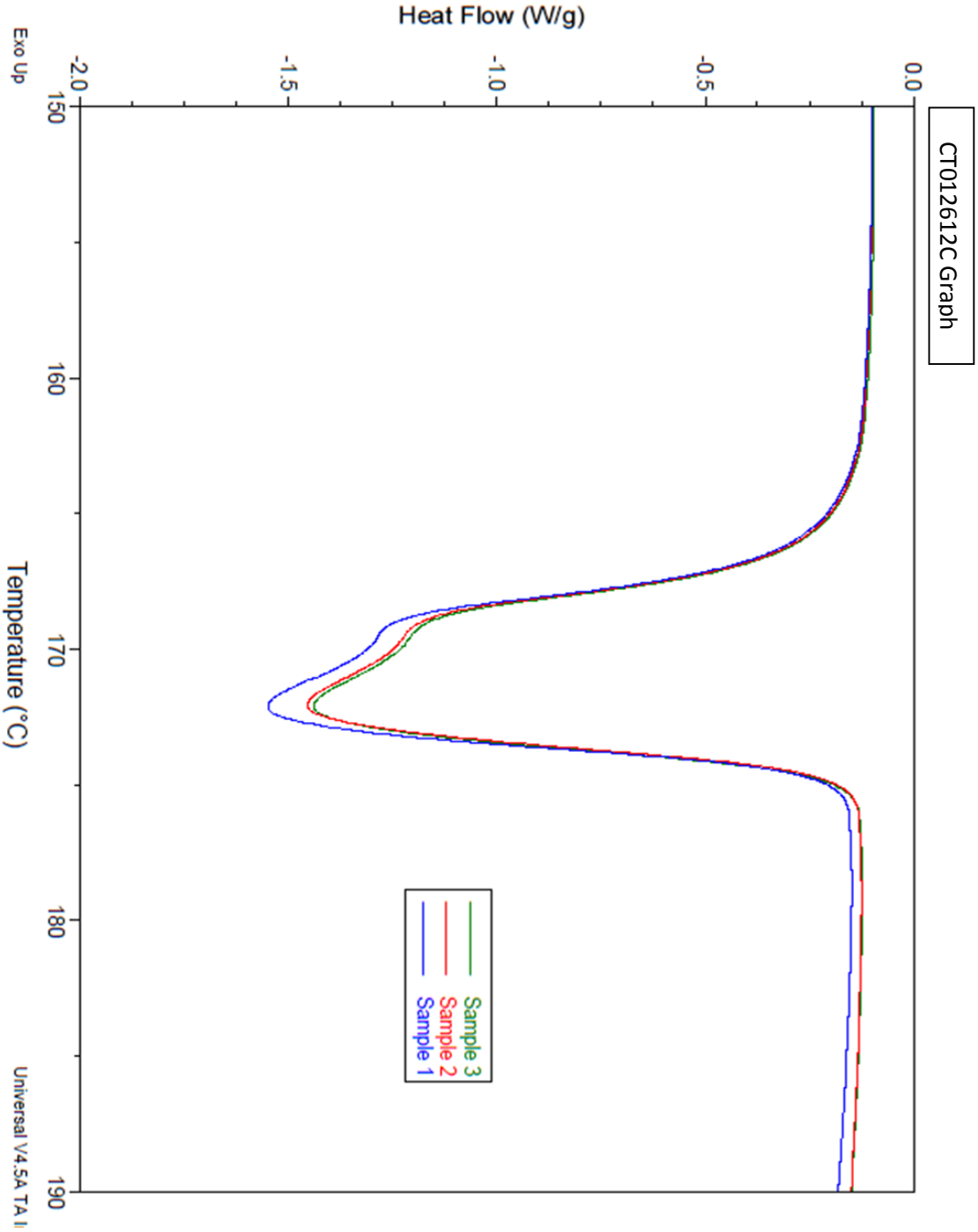

CT011211E Repeat 3 under 63 Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
120
140
160
180
200
220
240
260
Temperature (°C)
CT011211E.R3.under63.S01
CT011211E.R4.under63.S01
CT011211E.R3.under63.S02
CT011211E.R3.under63.S03

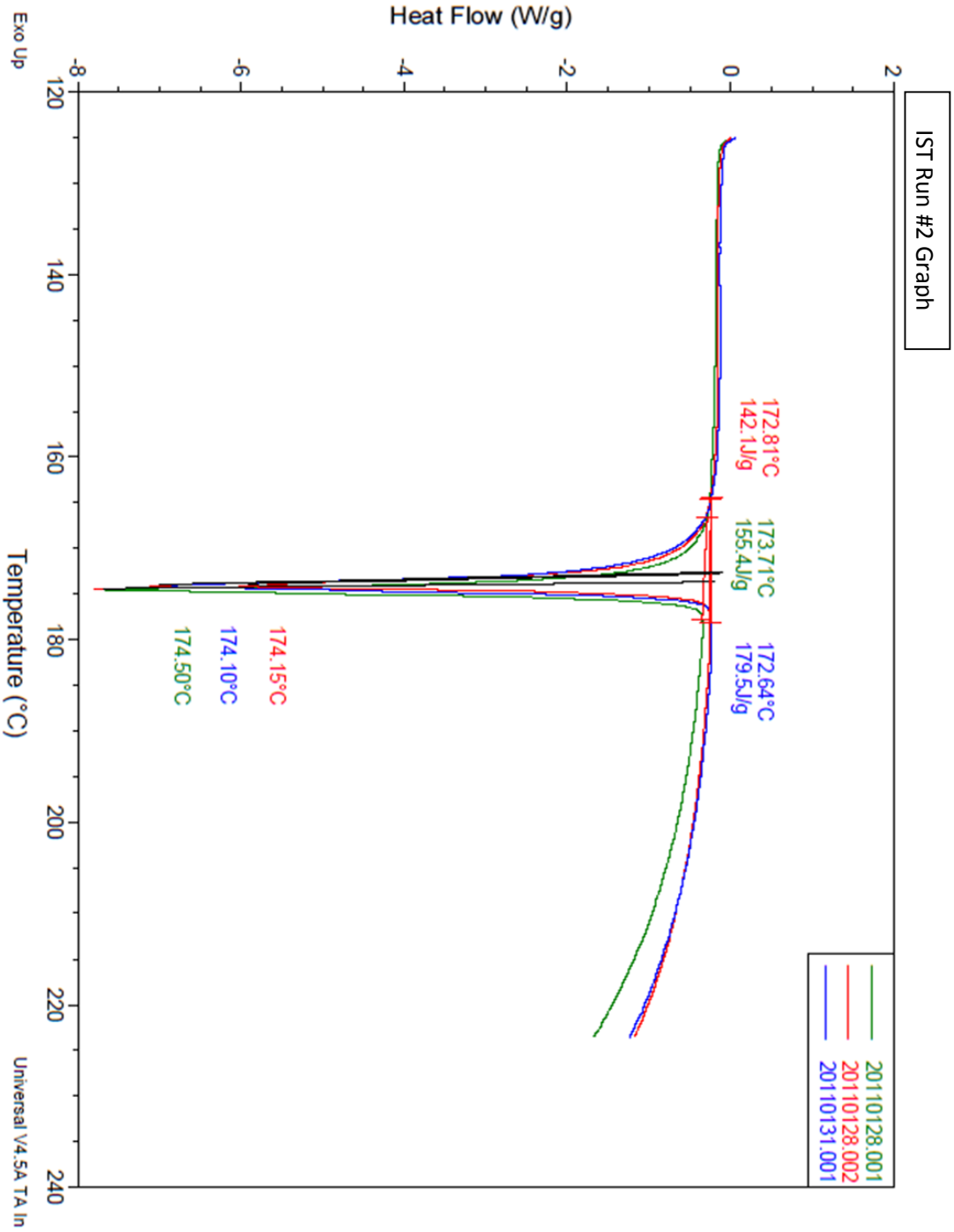

CT011211E Repeat 3 63-106 um Graph
011211E repeat 3_63-106_S01
CT011211E repeat 3_63-106.S02
CT011211E repeat 3_63-106.S03
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
Temperature (°C)
120
140
160
180
200
220
240
260

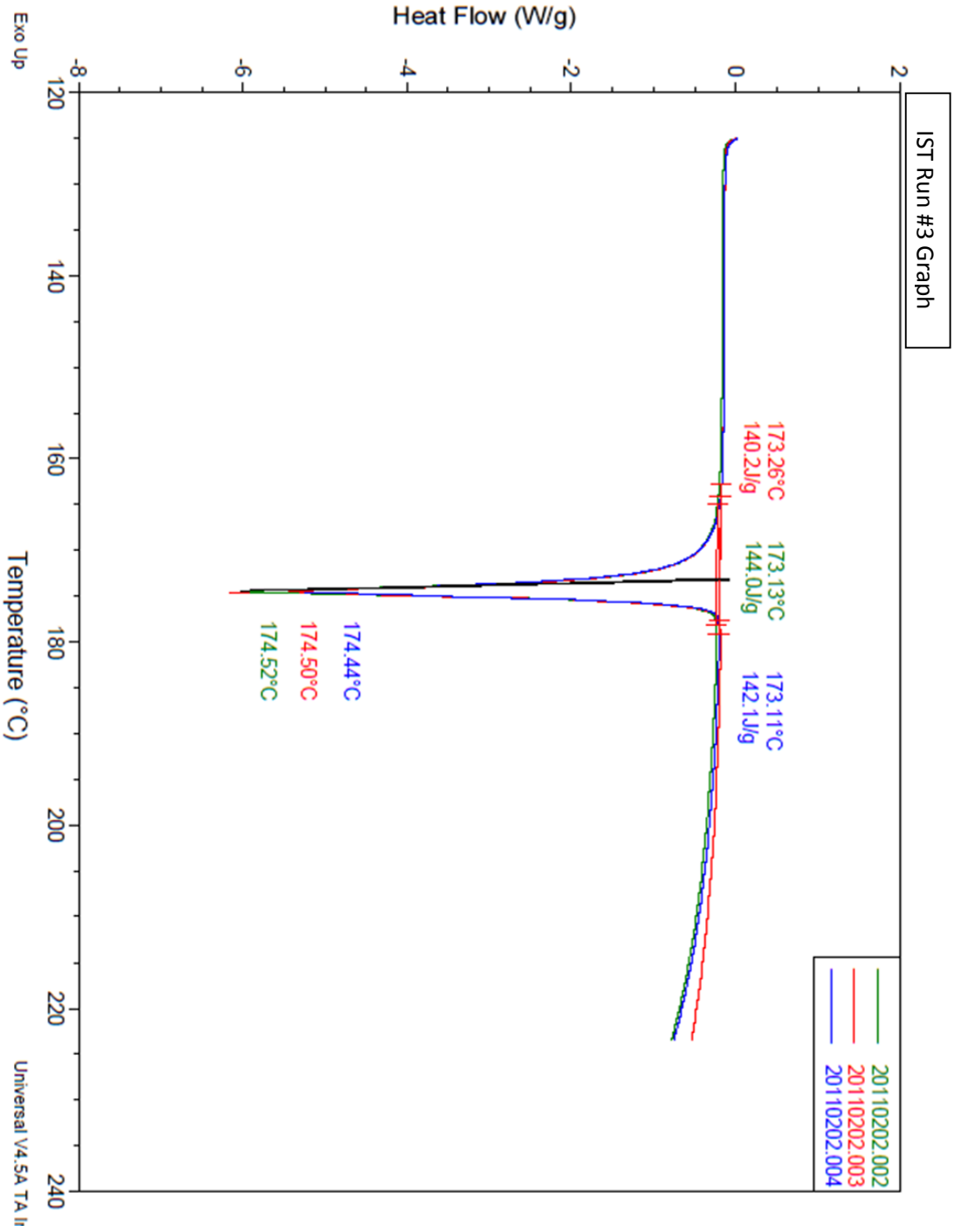

CT011211E Repeat 4 under 63 Graph
CT011211E.R4.under63.S03
CT011211E.R4.under63.S02
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
120
140
160
180
200
220
240
260
Temperature (°C)

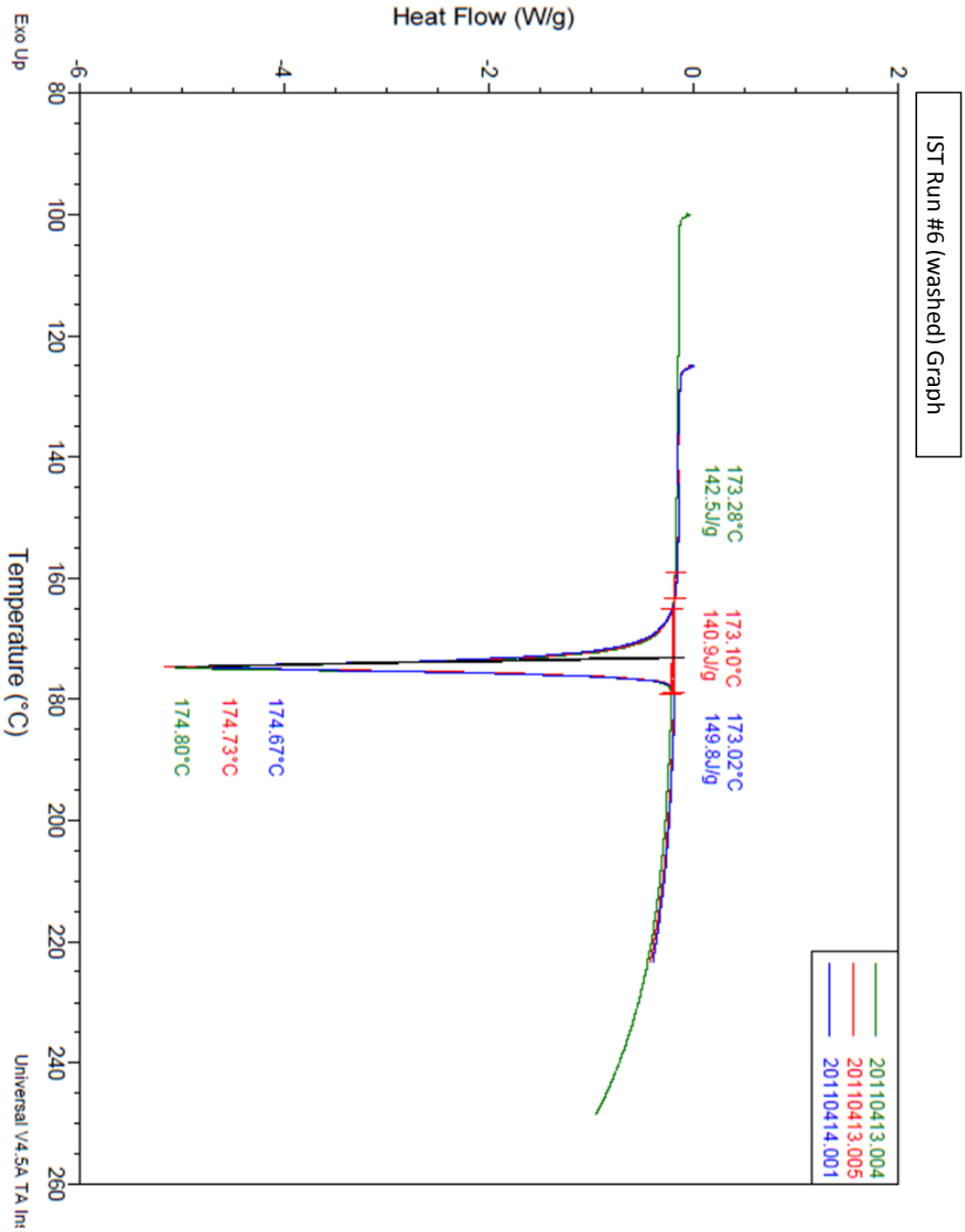

CT011211E Repeat 4 63-106 um Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
120
140
160
180
200
220
240
260
Temperature (°C)
CT011211E.R4.63-106.S02
CT011211E.R4.63-106.S03

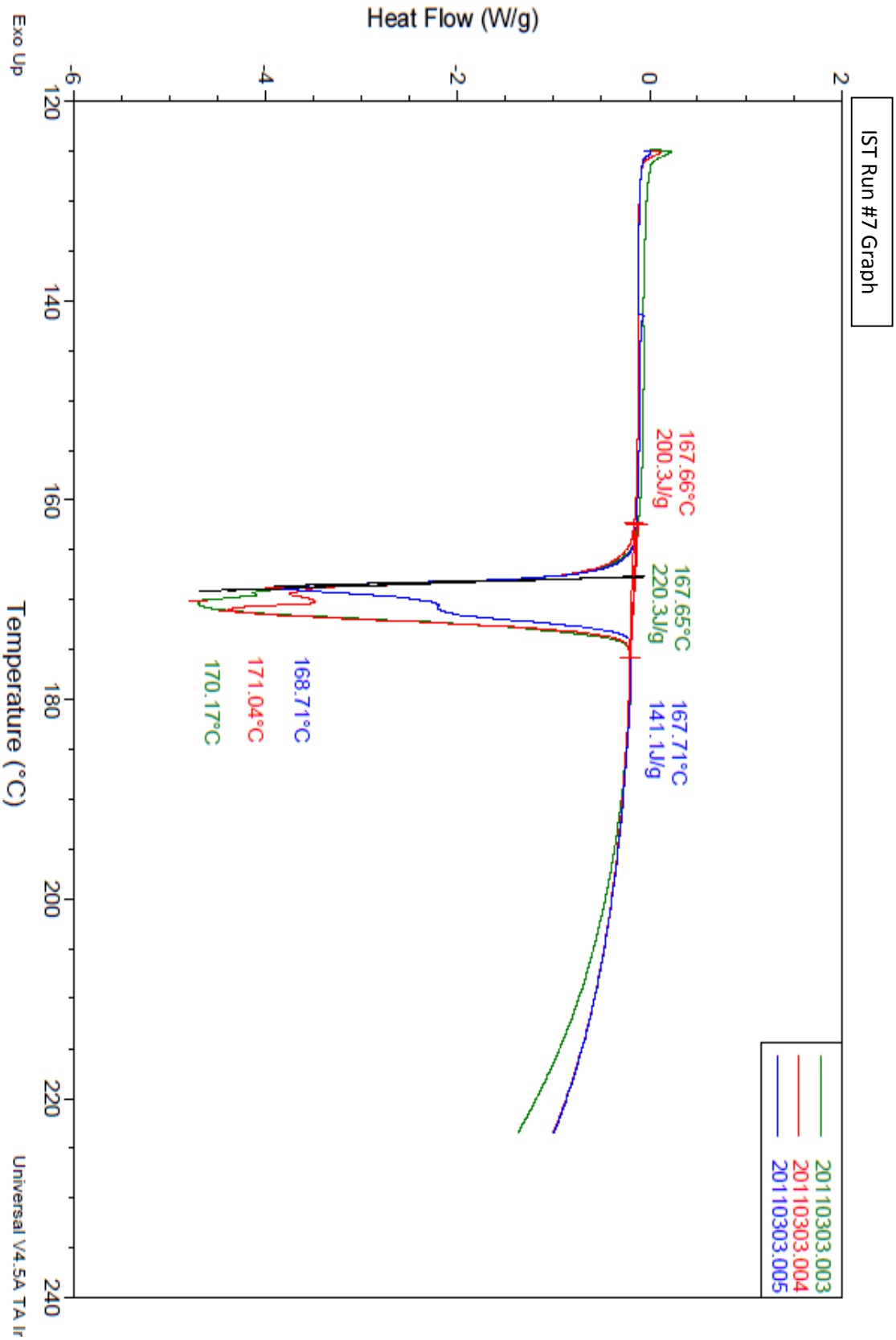

CT012612B Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
-1.4
Exo Up
150
160
170
180
190
200
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA In

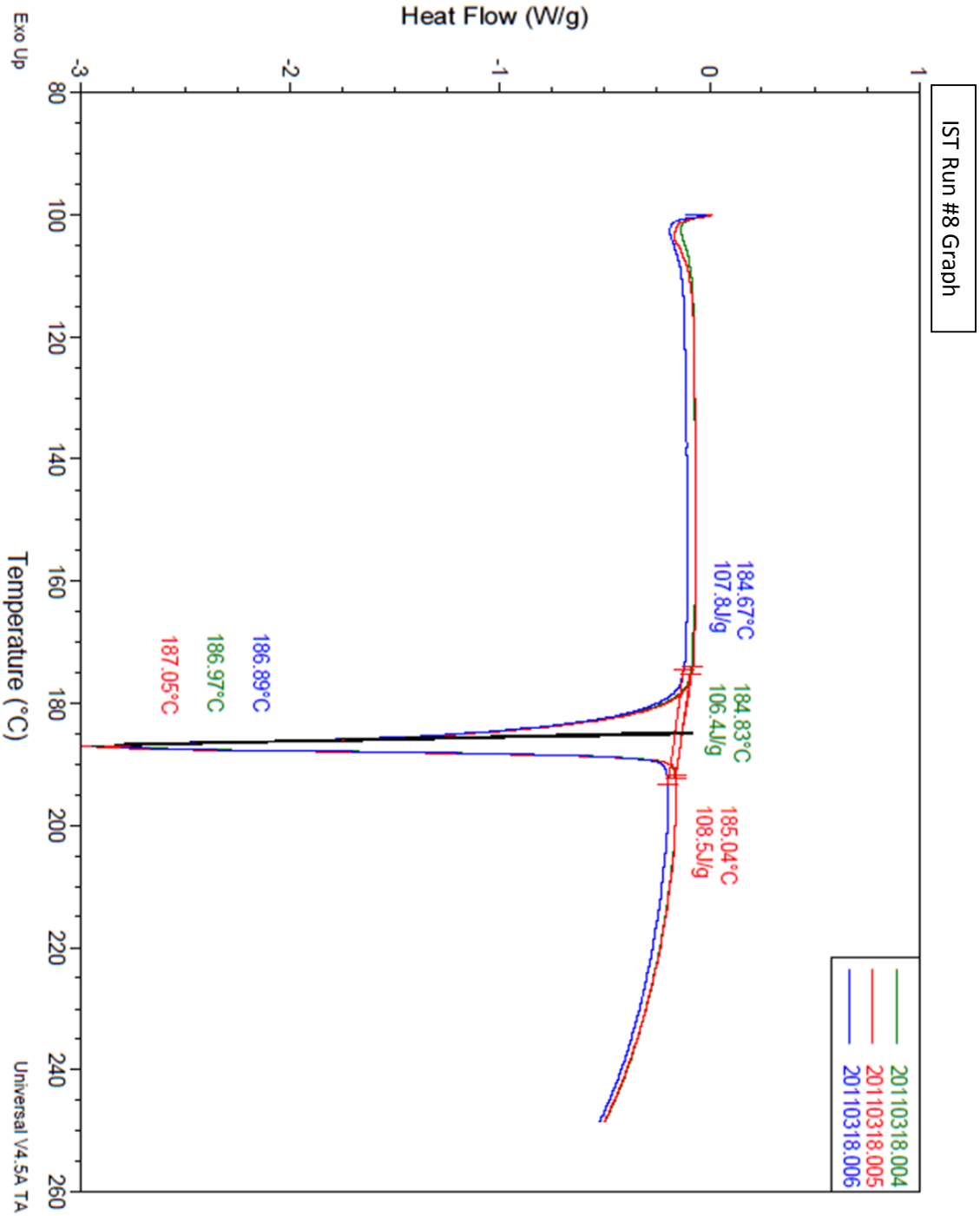

CT012612C Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
150
160
170
180
190
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA I


IST Run #2 Graph
20110128.001
20110128.002
20110131.001
172.81°C
142.1J/g
173.71°C
155.4J/g
172.64°C
179.5J/g
174.15°C
174.10°C
174.50°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.5A TA In

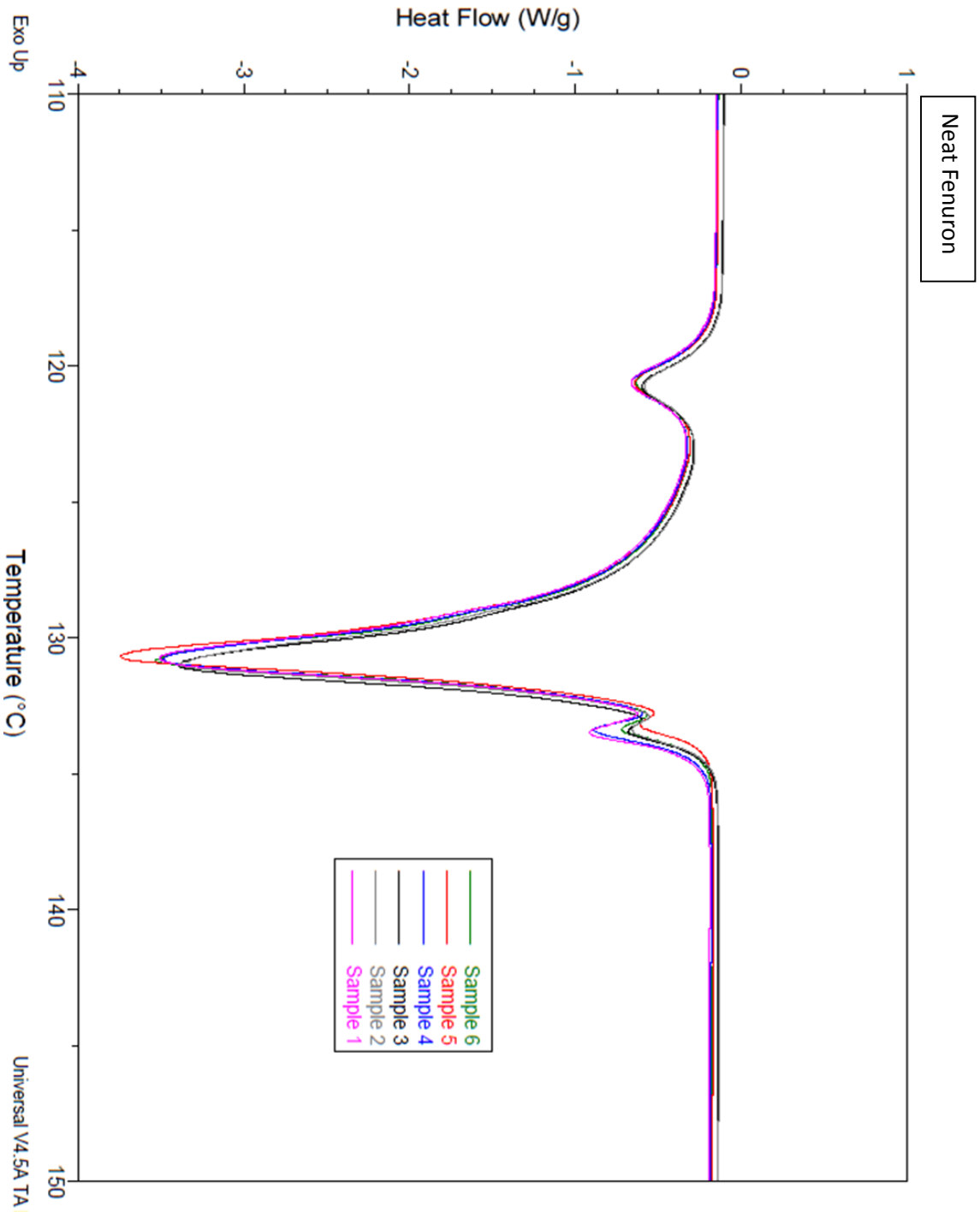

IST Run #3 Graph
20110202.002
20110202.003
20110202.004
173.26°C
140.2J/g
173.13°C
144.0J/g
173.11°C
142.1J/g
174.44°C
174.50°C
174.52°C
Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.5A TA In

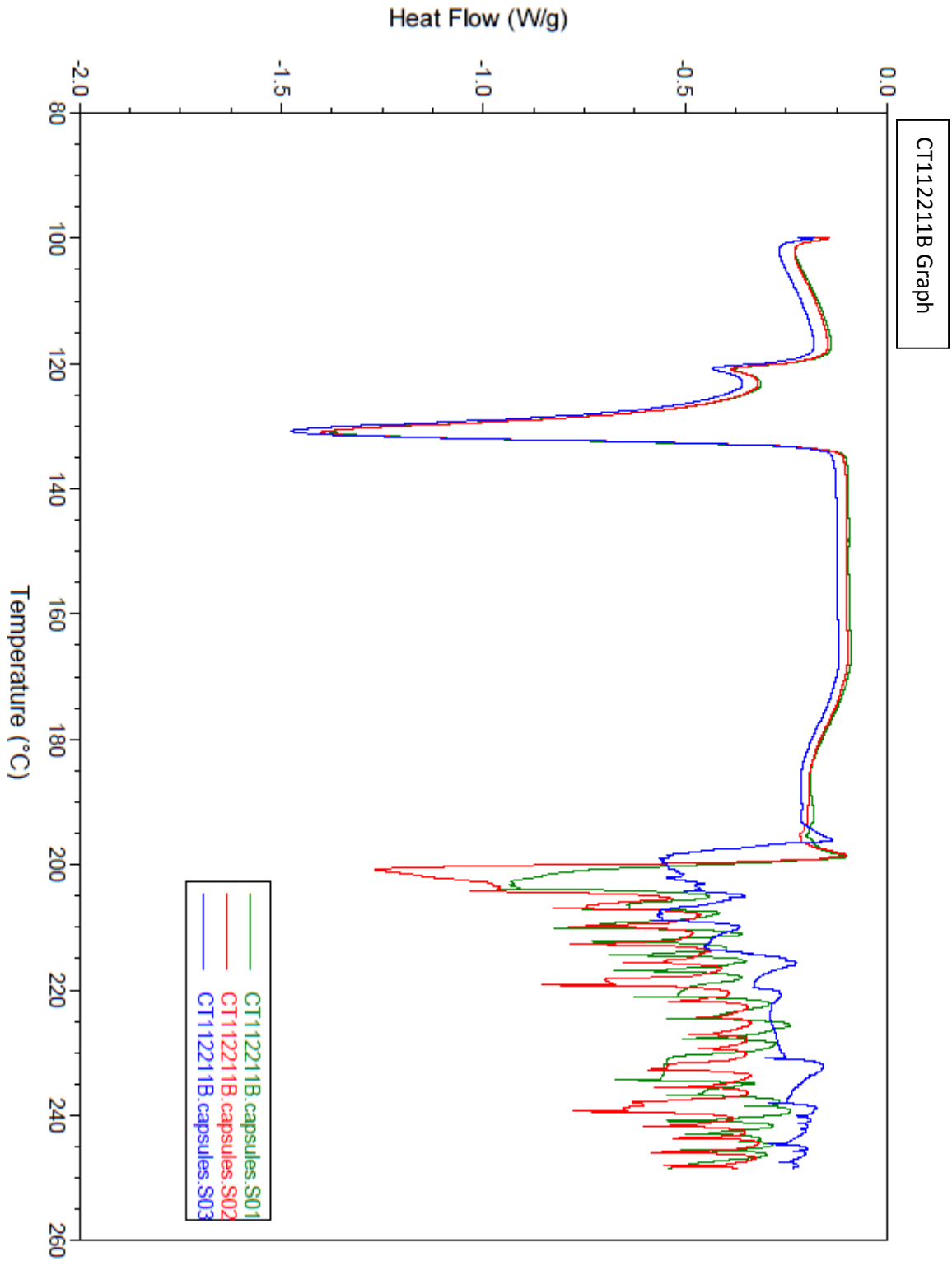

IST Run #6 (washed) Graph
Heat Flow (W/g)
2
0
-2
-4
-6
Exo Up
80
100
120
140
160
180
200
220
240
260
Temperature (°C)
Universal V4.5A TA Ins
20110413.004
20110413.005
20110414.001
173.28°C
142.5J/g
173.10°C
140.9J/g
173.02°C
149.8J/g
174.67°C
174.73°C
174.80°C

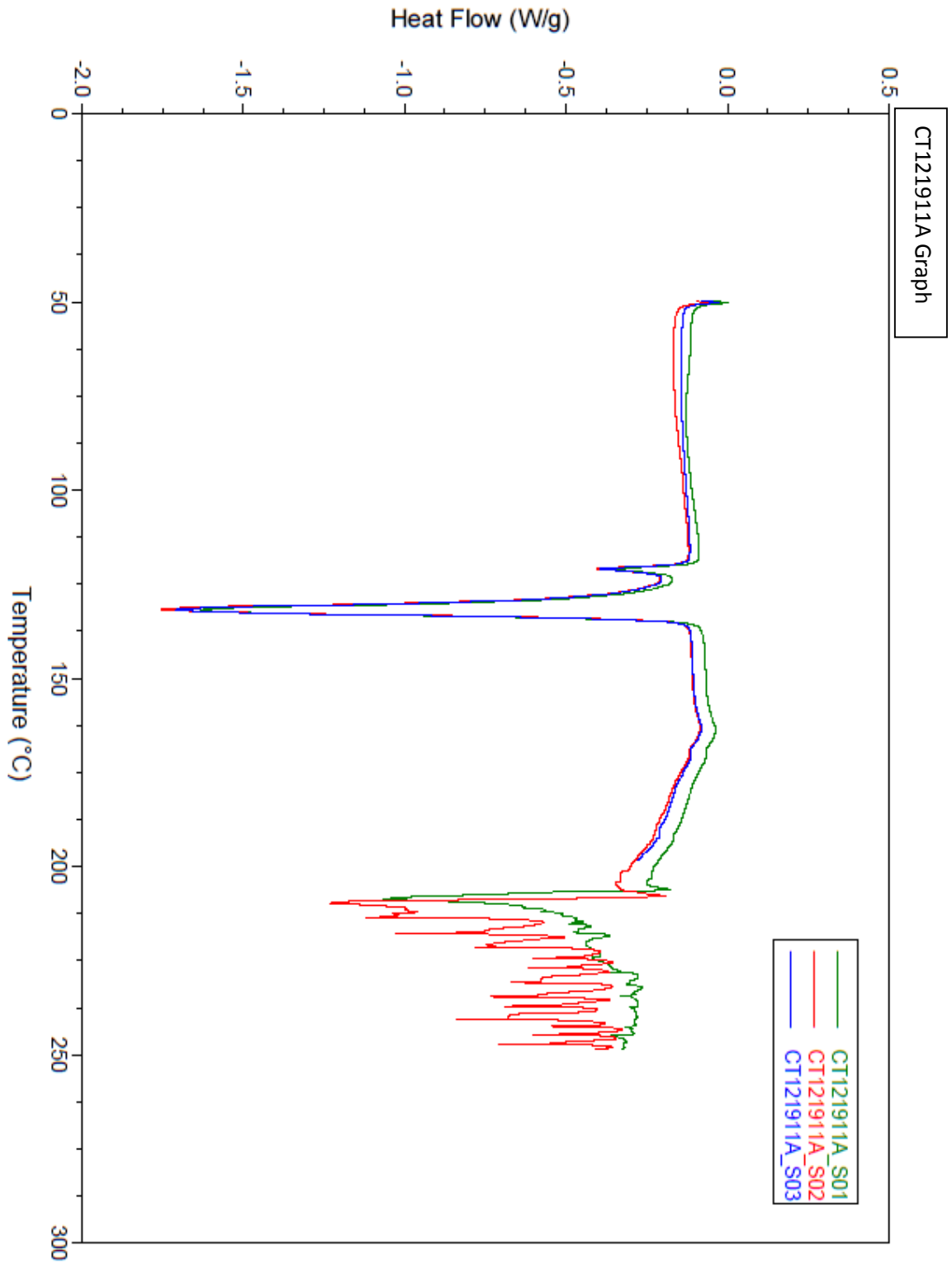

IST Run #7 Graph
Heat Flow (W/g)
2
0
-2
-4
-6
120
140
160
180
200
220
240
Temperature (°C)
Exo Up
Universal V4.5A TA Ir
167.66°C
200.3J/g
167.65°C
220.3J/g
167.71°C
141.1J/g
168.71°C
171.04°C
170.17°C
20110303.003
20110303.004
20110303.005

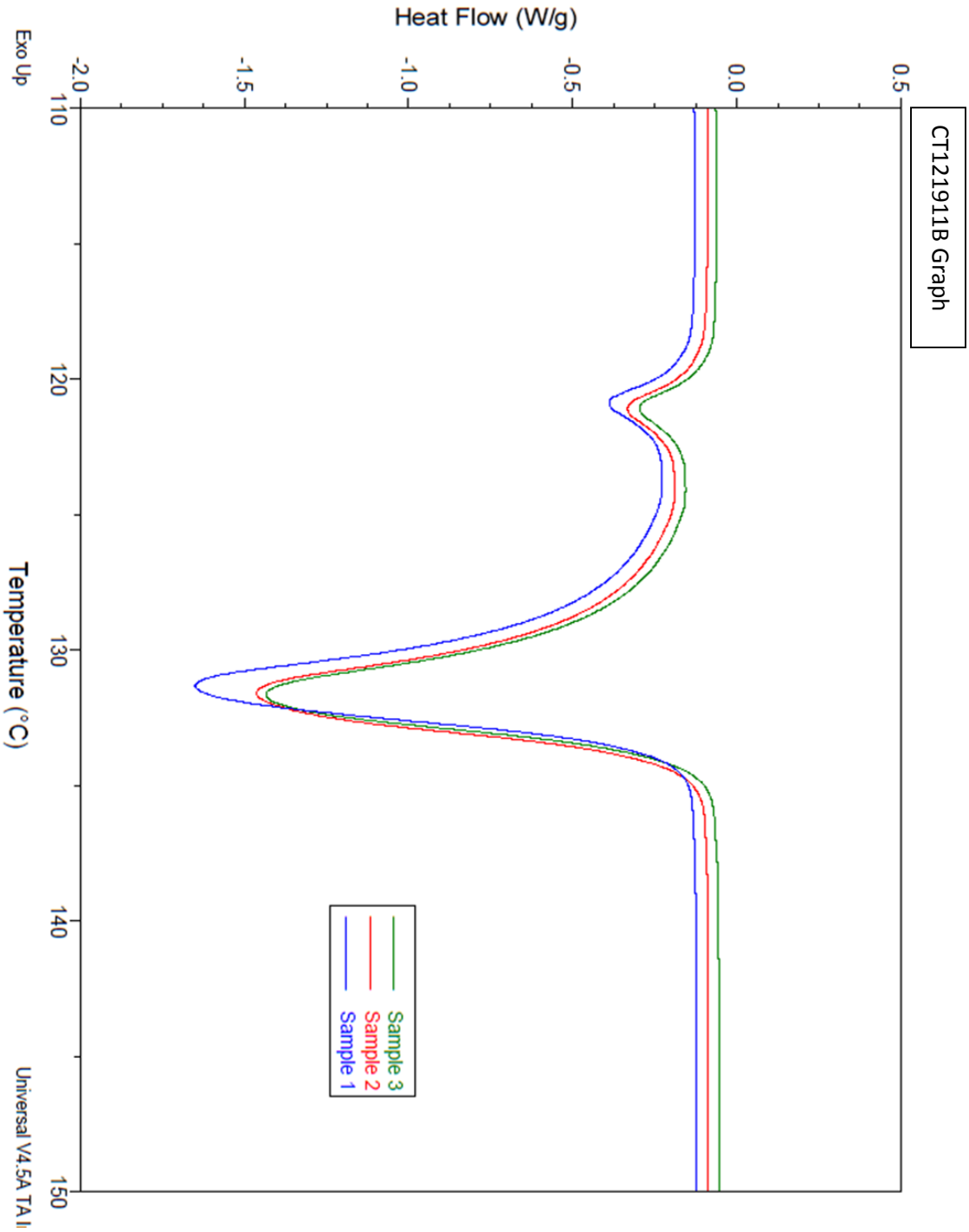

IST Run #8 Graph
Heat Flow (W/g)
1
0
-1
-2
-3
Exo Up
80
100
120
140
160
180
200
220
240
260
Temperature (°C)
Universal V4.5A TA
20110318.004
20110318.005
20110318.006
184.67°C
107.8J/g
184.83°C
106.4J/g
185.04°C
108.5J/g
186.89°C
186.97°C
187.05°C

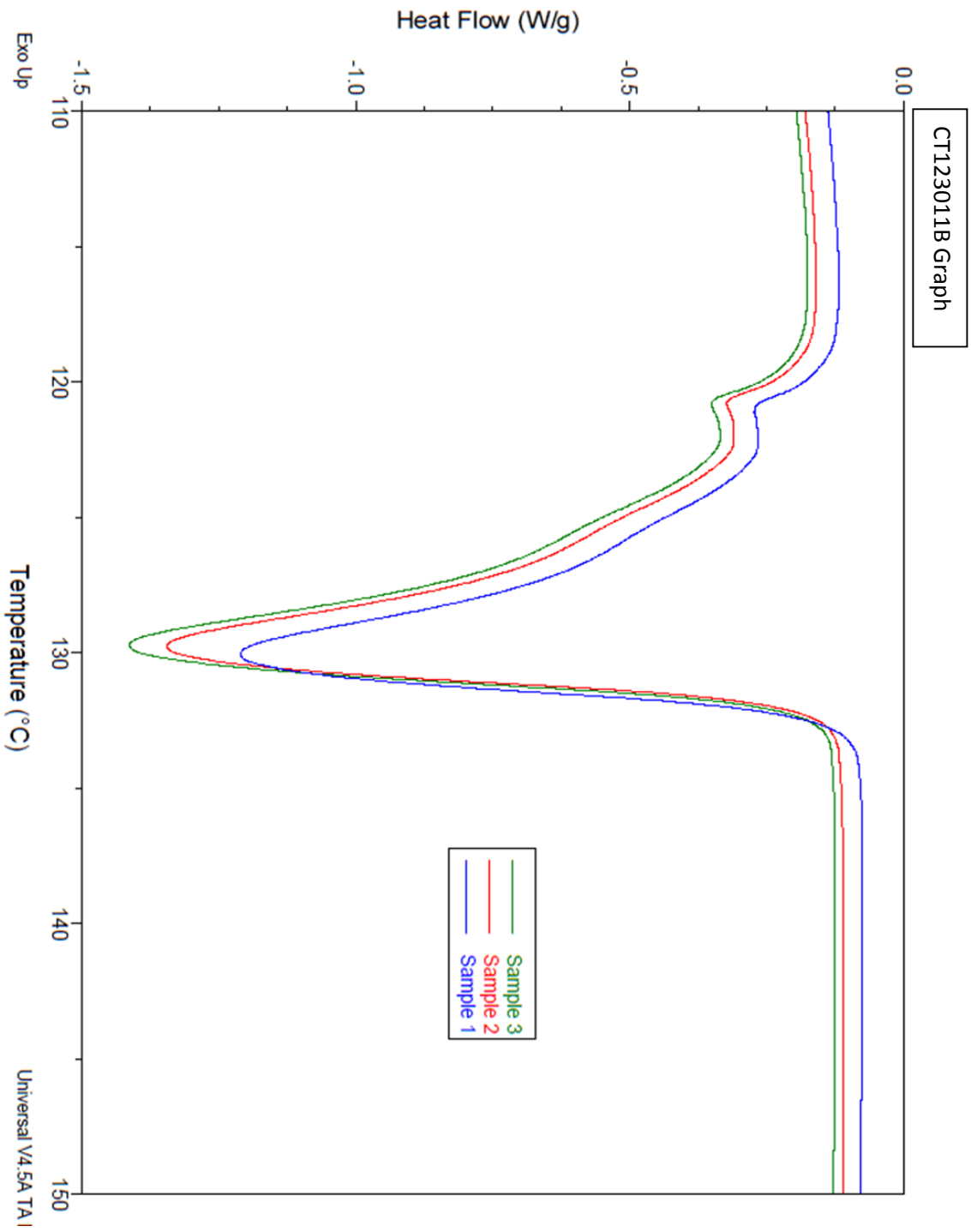
Appendix 3B: DSC Data for Fenuron Based Samples

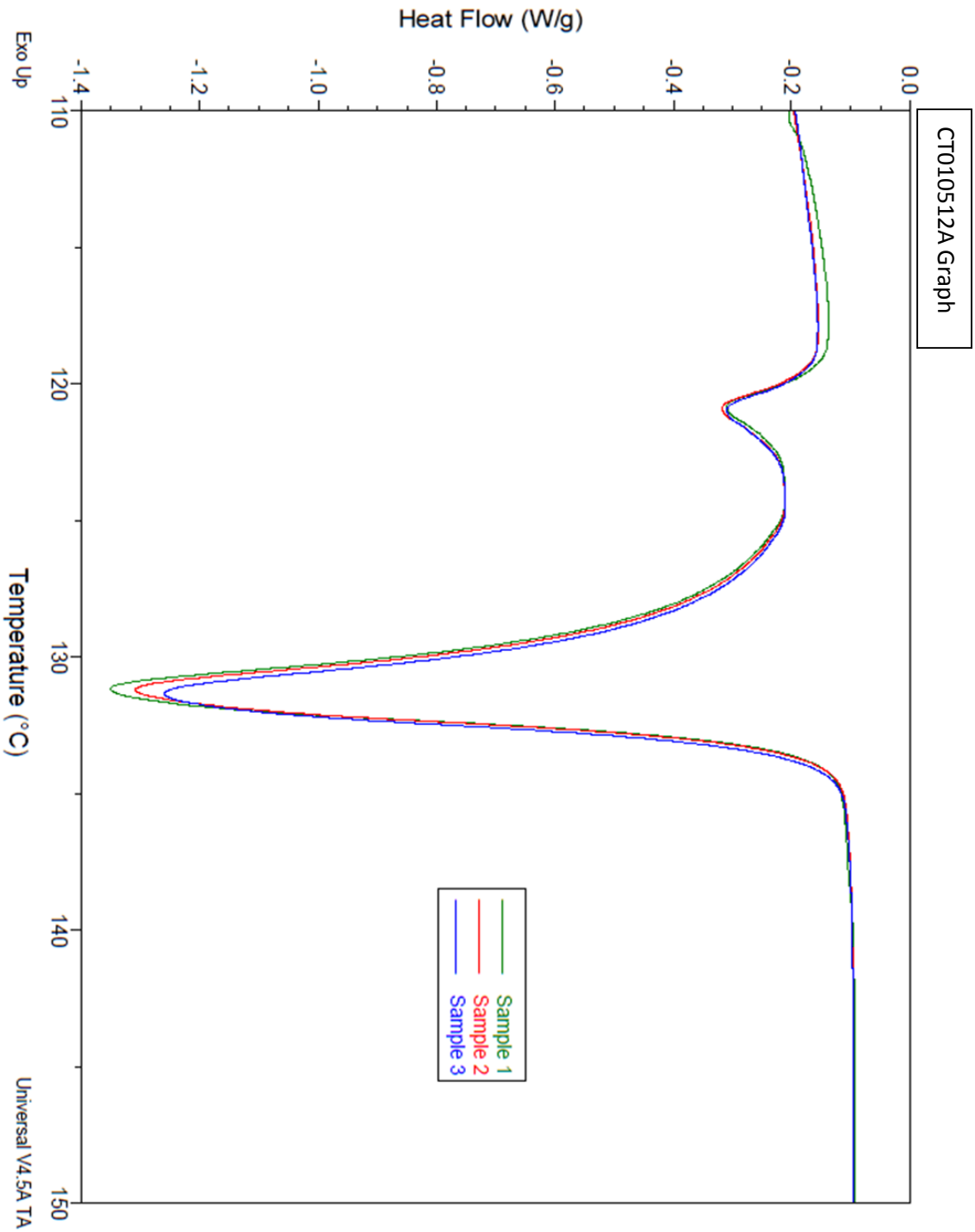


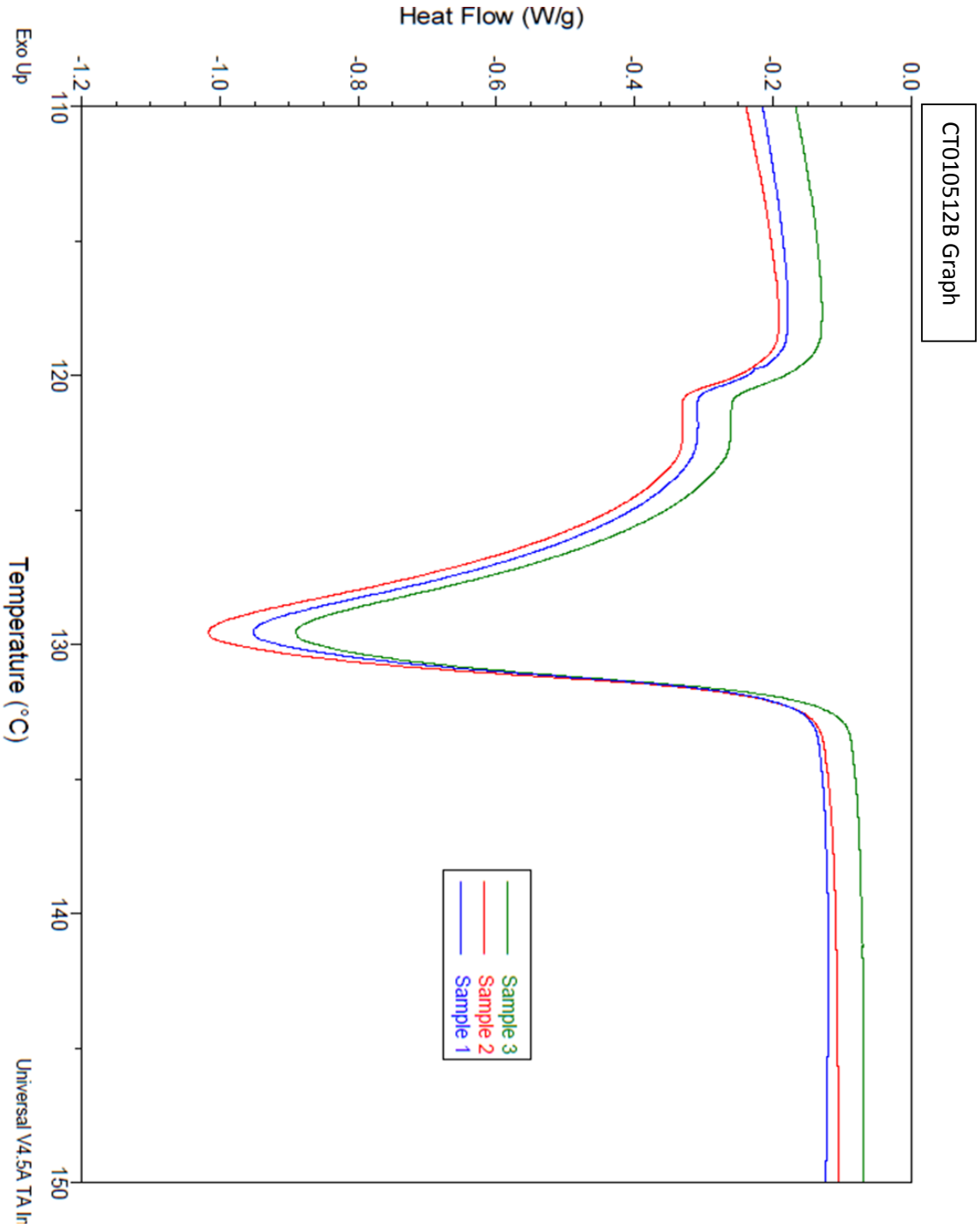

CT112211B Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
80
100
120
140
160
180
200
220
240
260
Temperature (°C)
CT112211B.capsules.S01
CT112211B.capsules.S02
CT112211B.capsules.S03

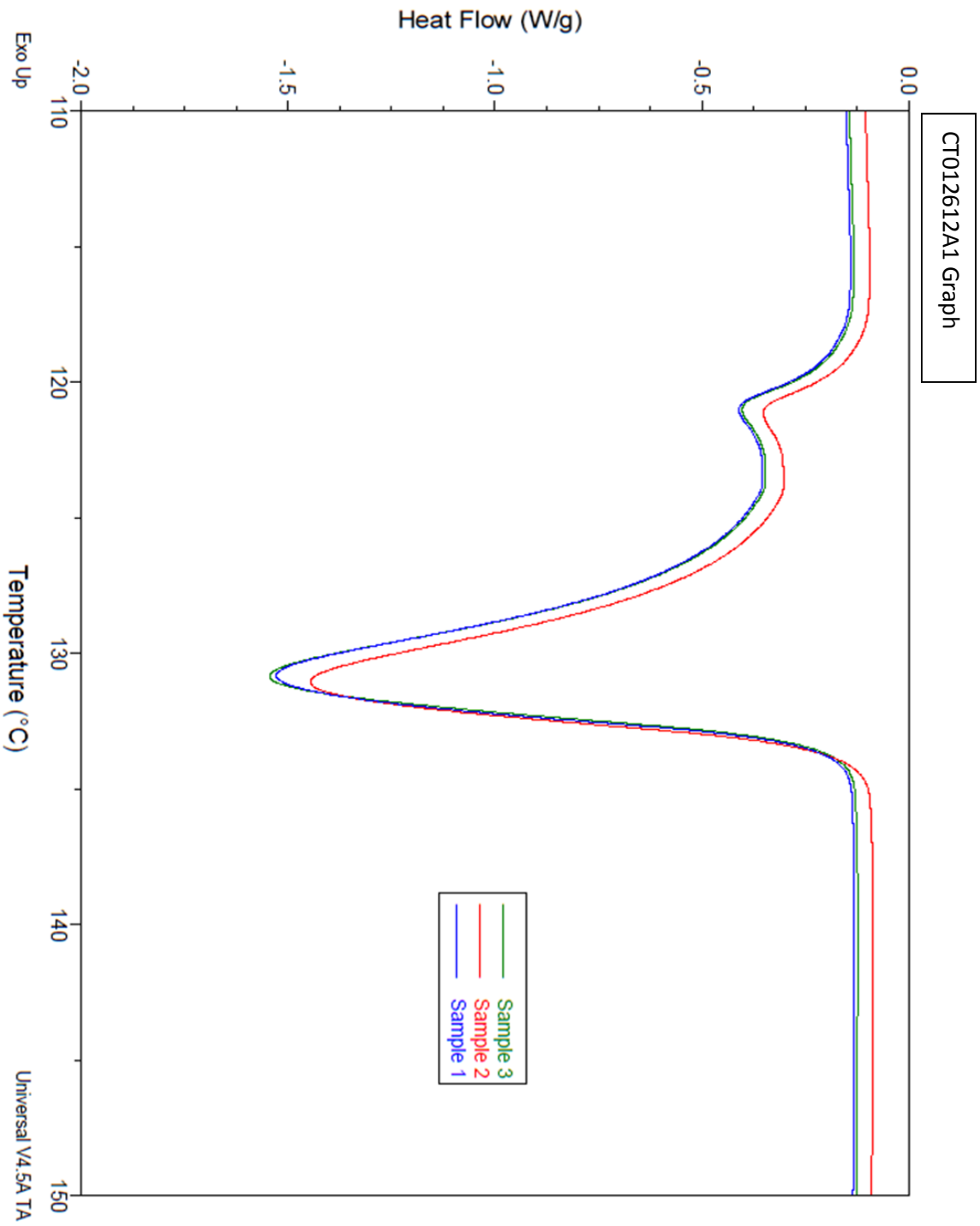

CT121911A Graph
CT121911A_S01
CT121911A_S02
CT121911A_S03
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
Temperature (°C)
0
50
100
150
200
250
300

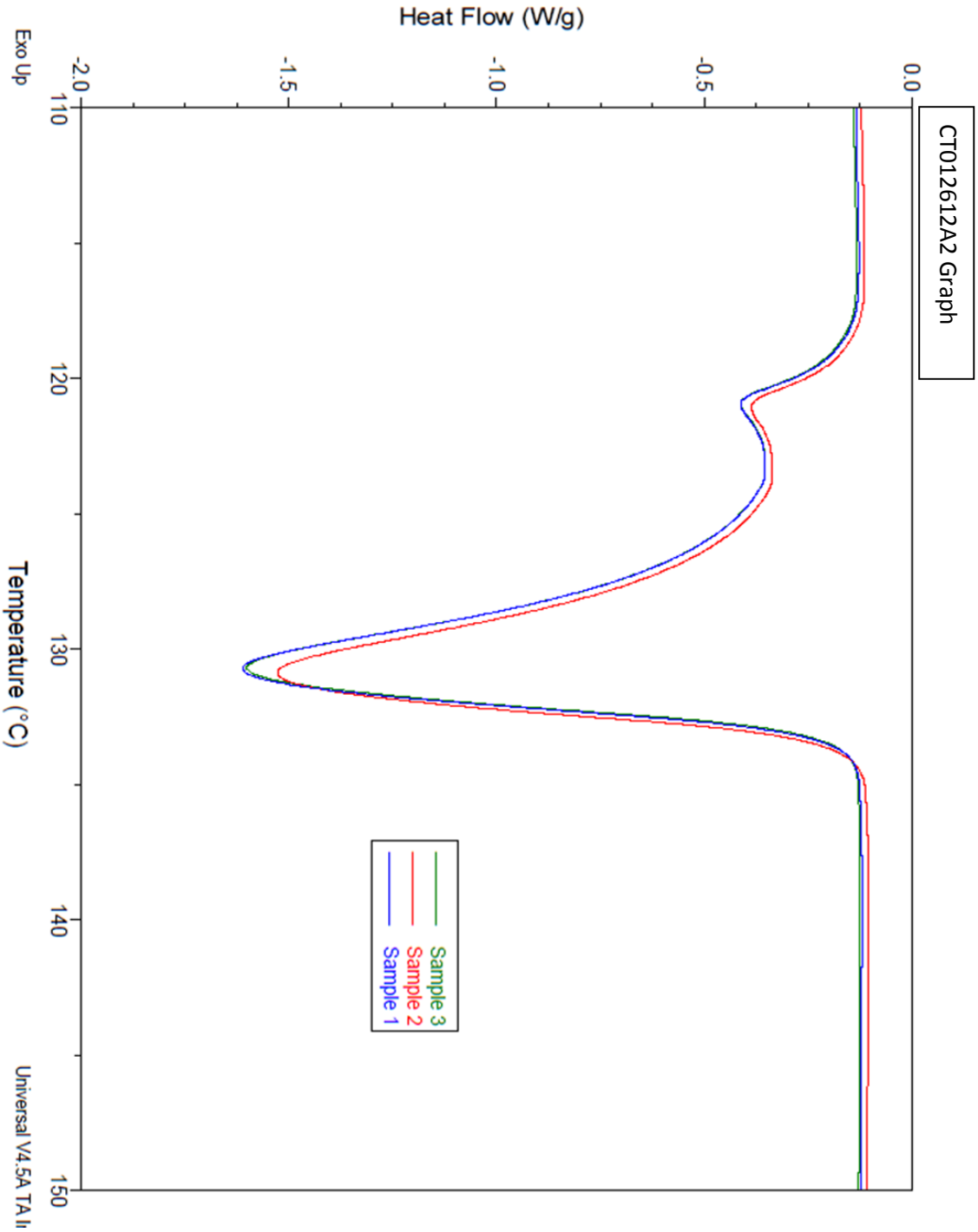

CT121911B Graph
Heat Flow (W/g)
0.5
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA I

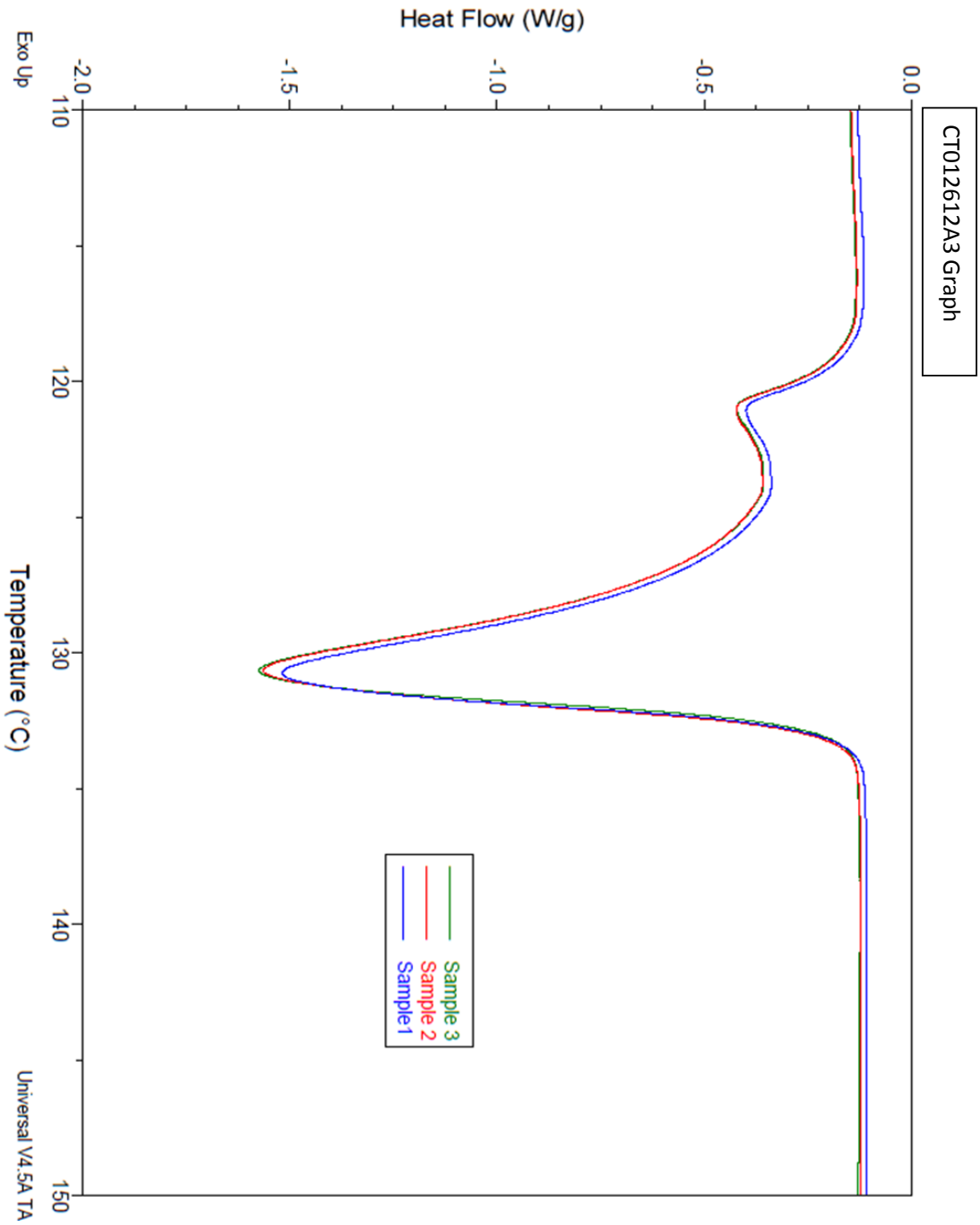

CT123011B Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA I

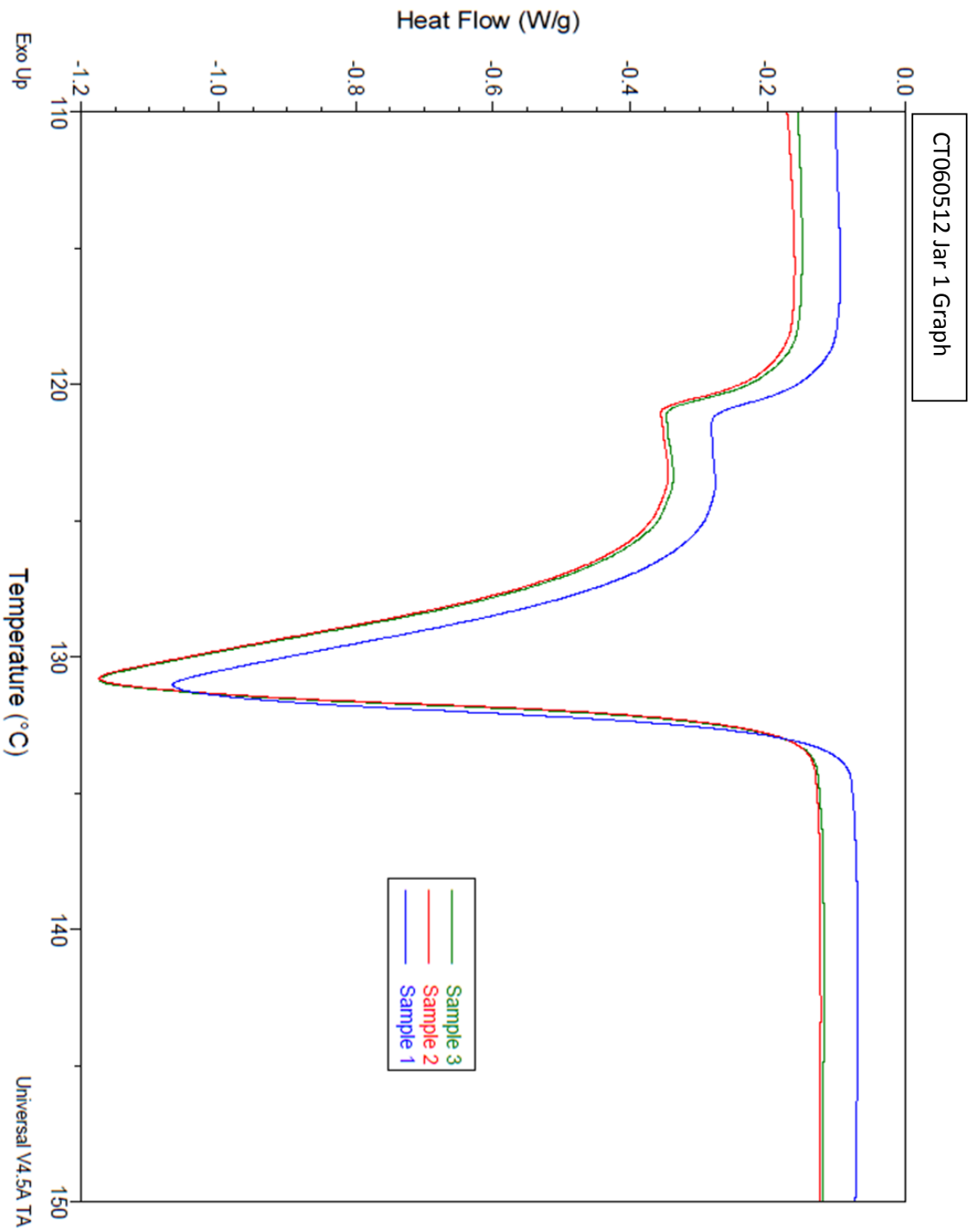

CT010512A Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
-1.4
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 1
Sample 2
Sample 3
Universal V4.5A TA

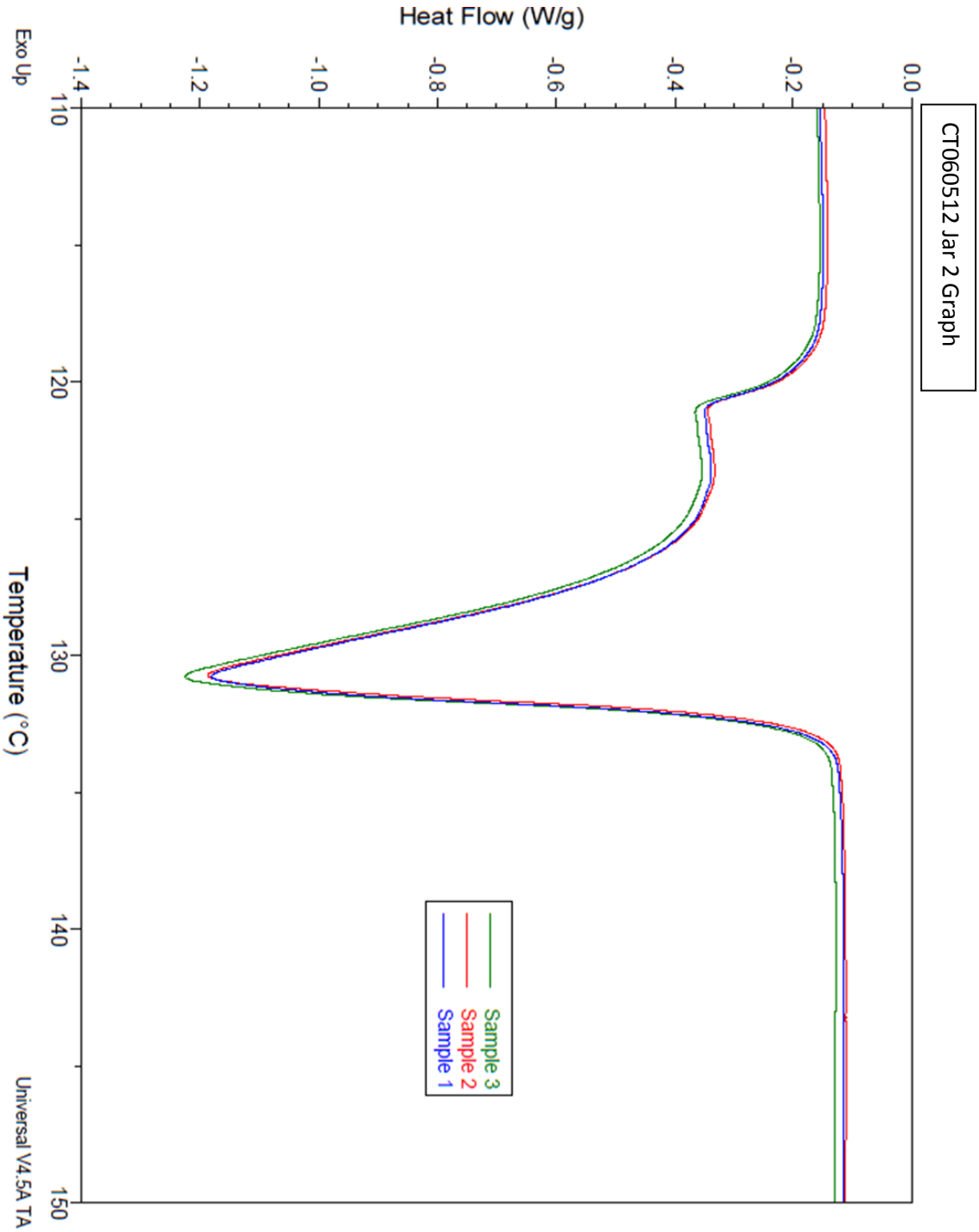

CT010512B Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
110
120
130
140
150
Temperature (°C)
Exo Up
Universal V4.5A TA In
Sample 3
Sample 2
Sample 1

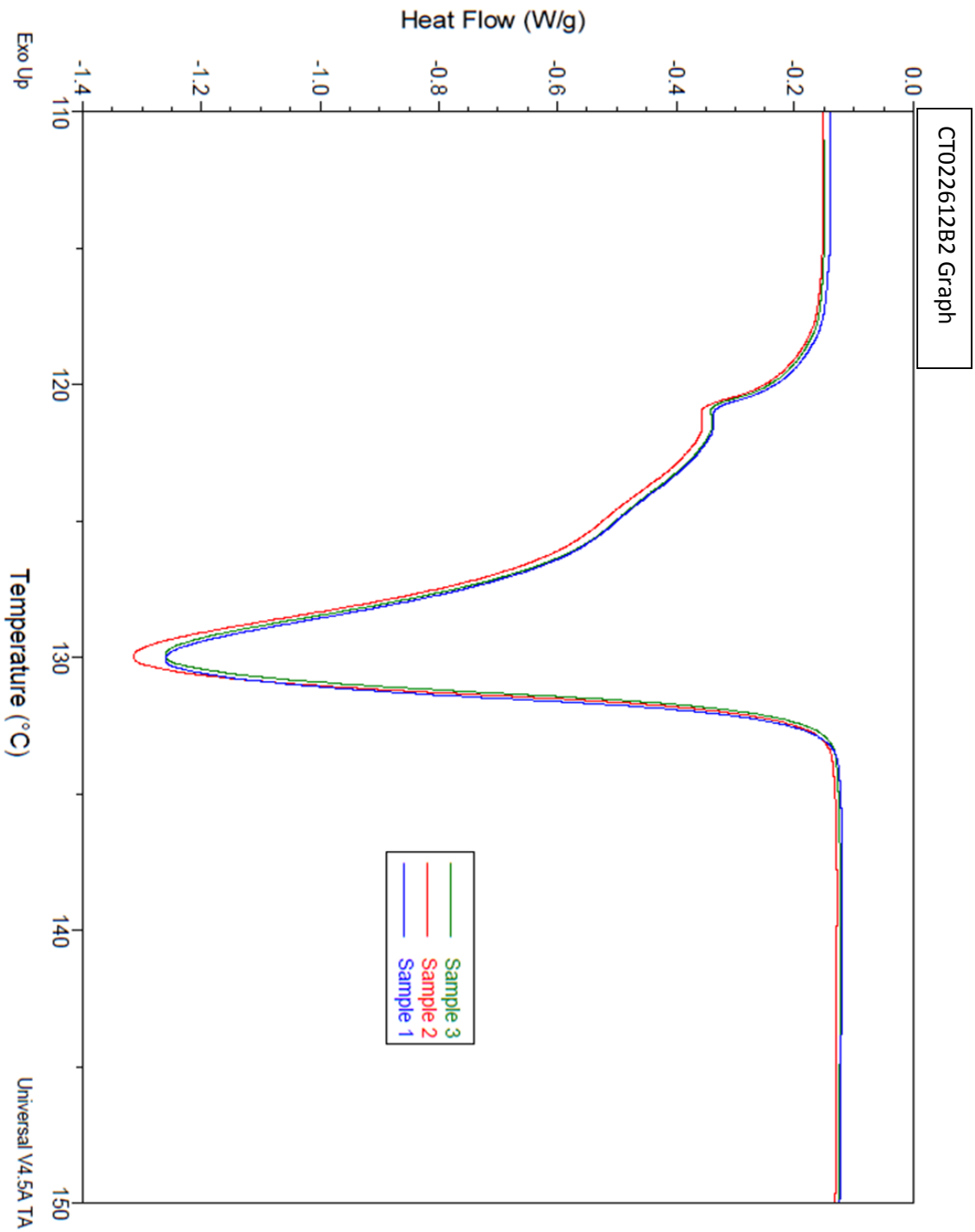

CT012612A1 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

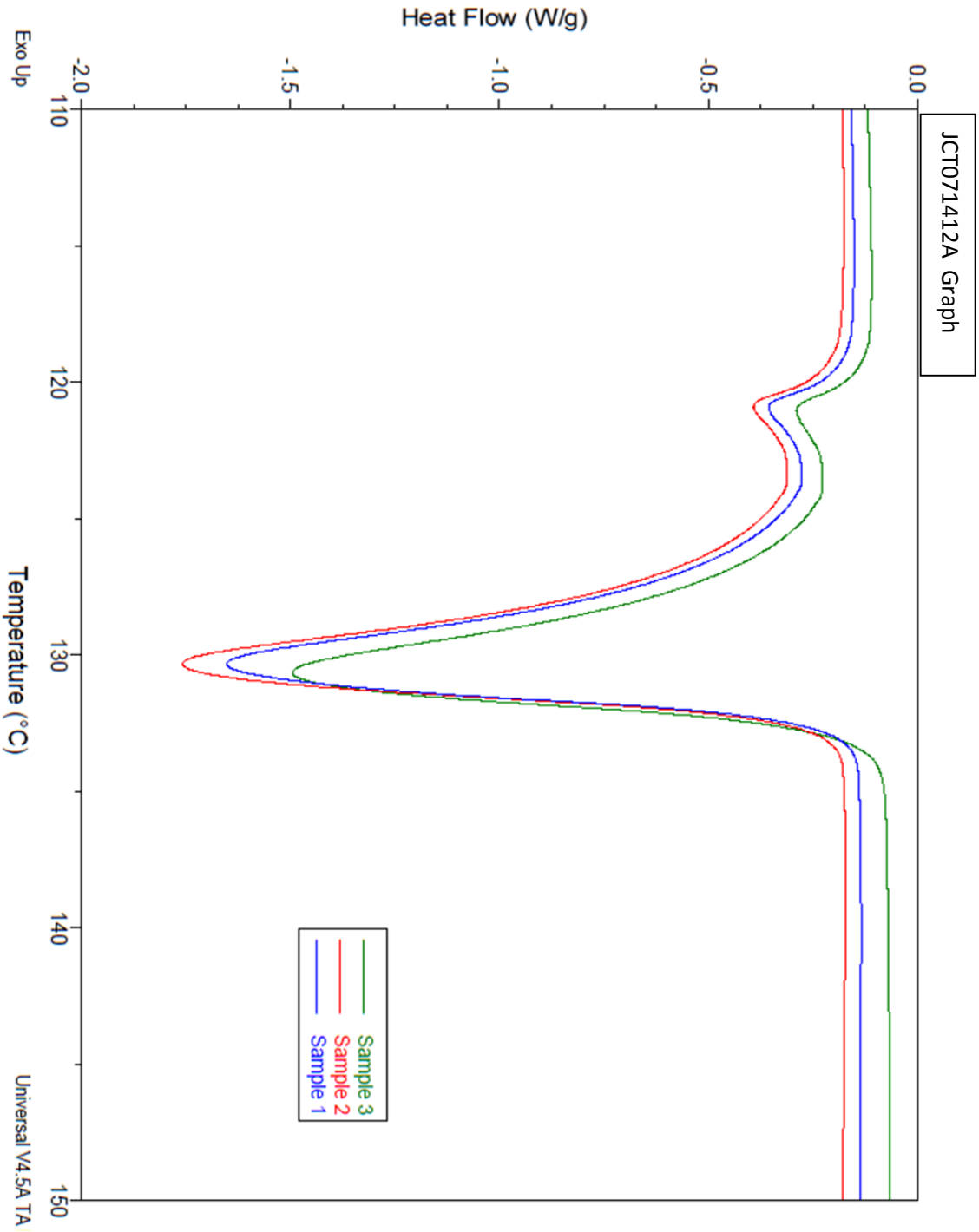

CT012612A2 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA In

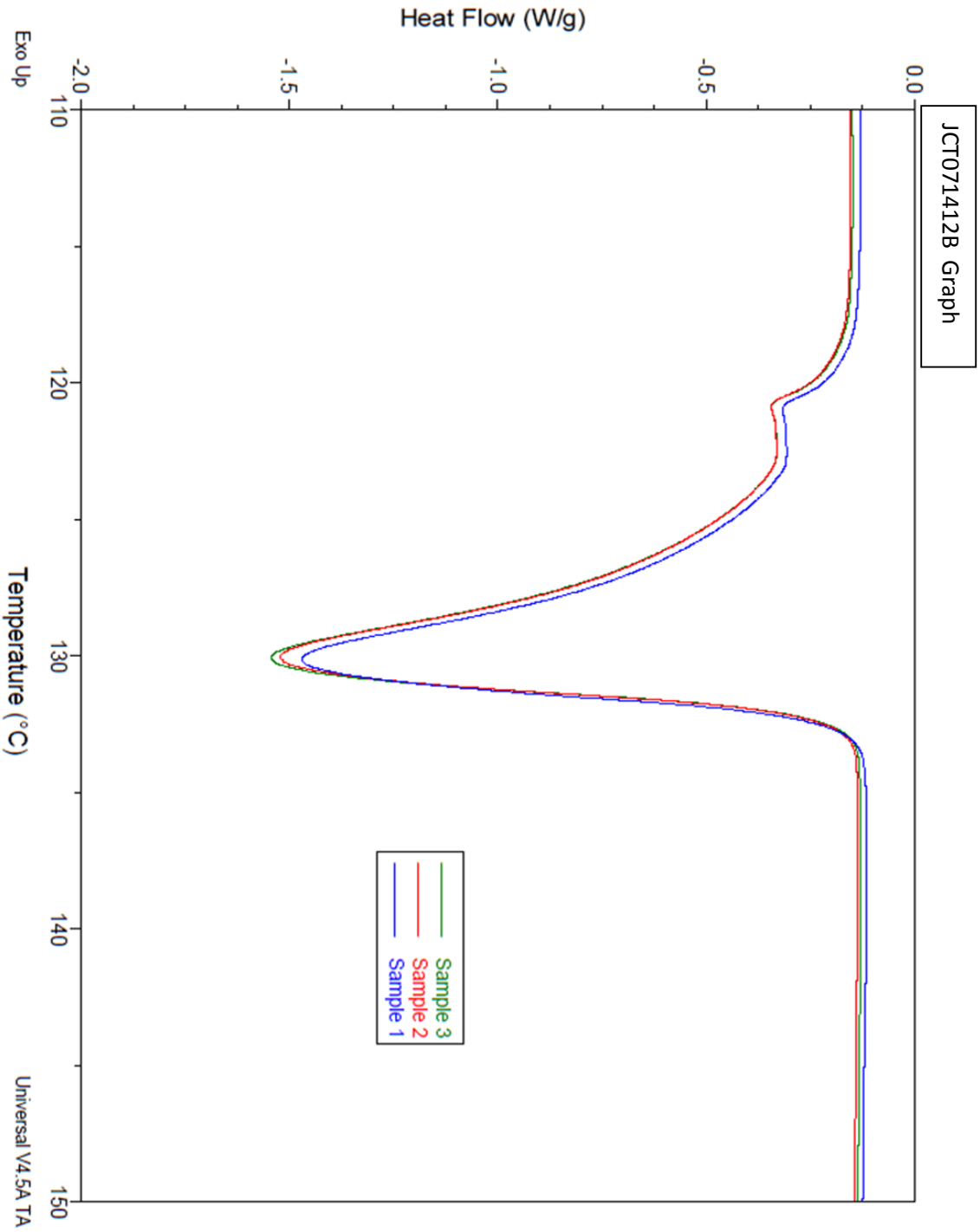

CT012612A3 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample1
Universal V4.5A TA

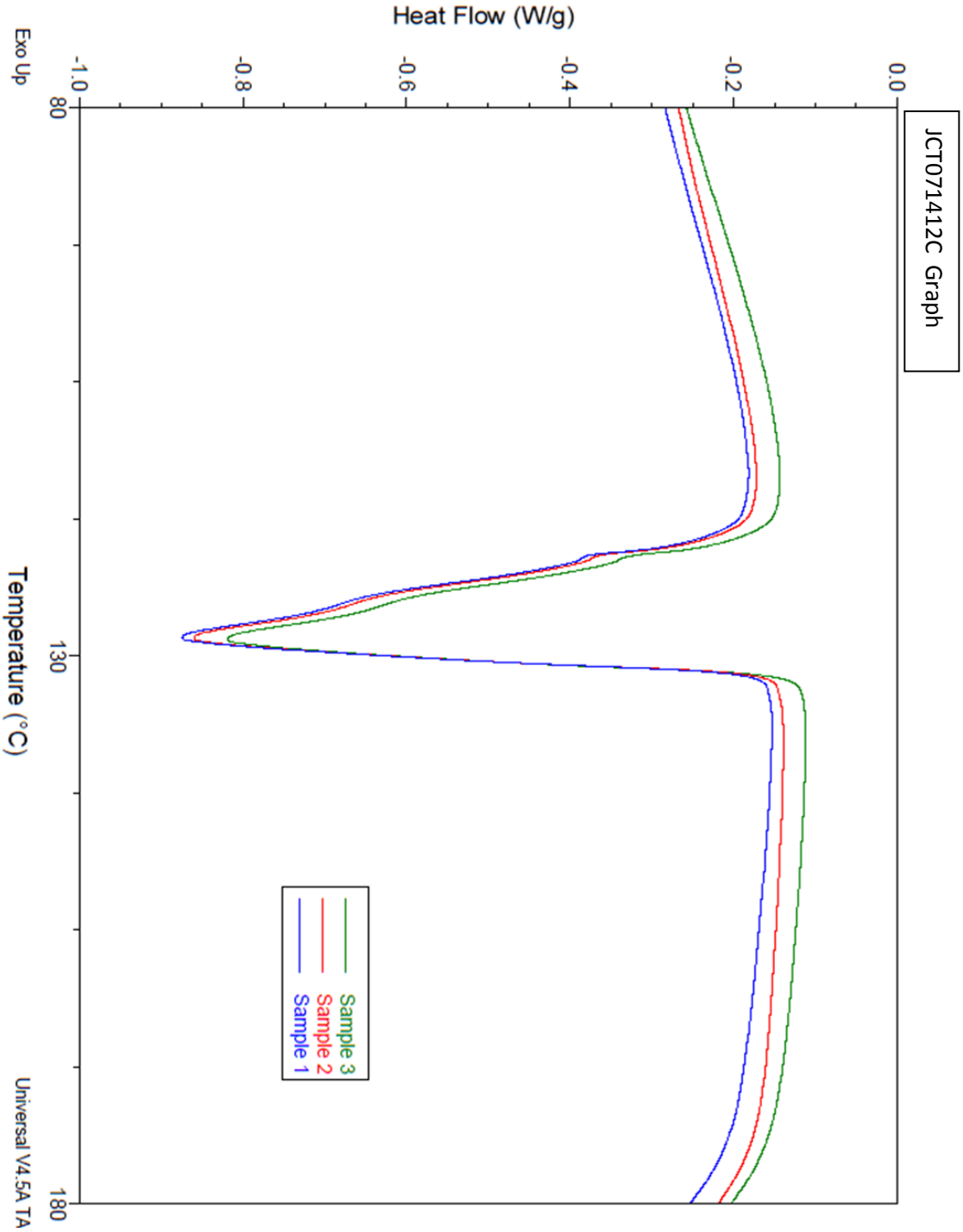

CT060512 Jar 1 Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
110
120
130
140
150
Temperature (°C)
Exo Up
Universal V4.5A TA
Sample 3
Sample 2
Sample 1

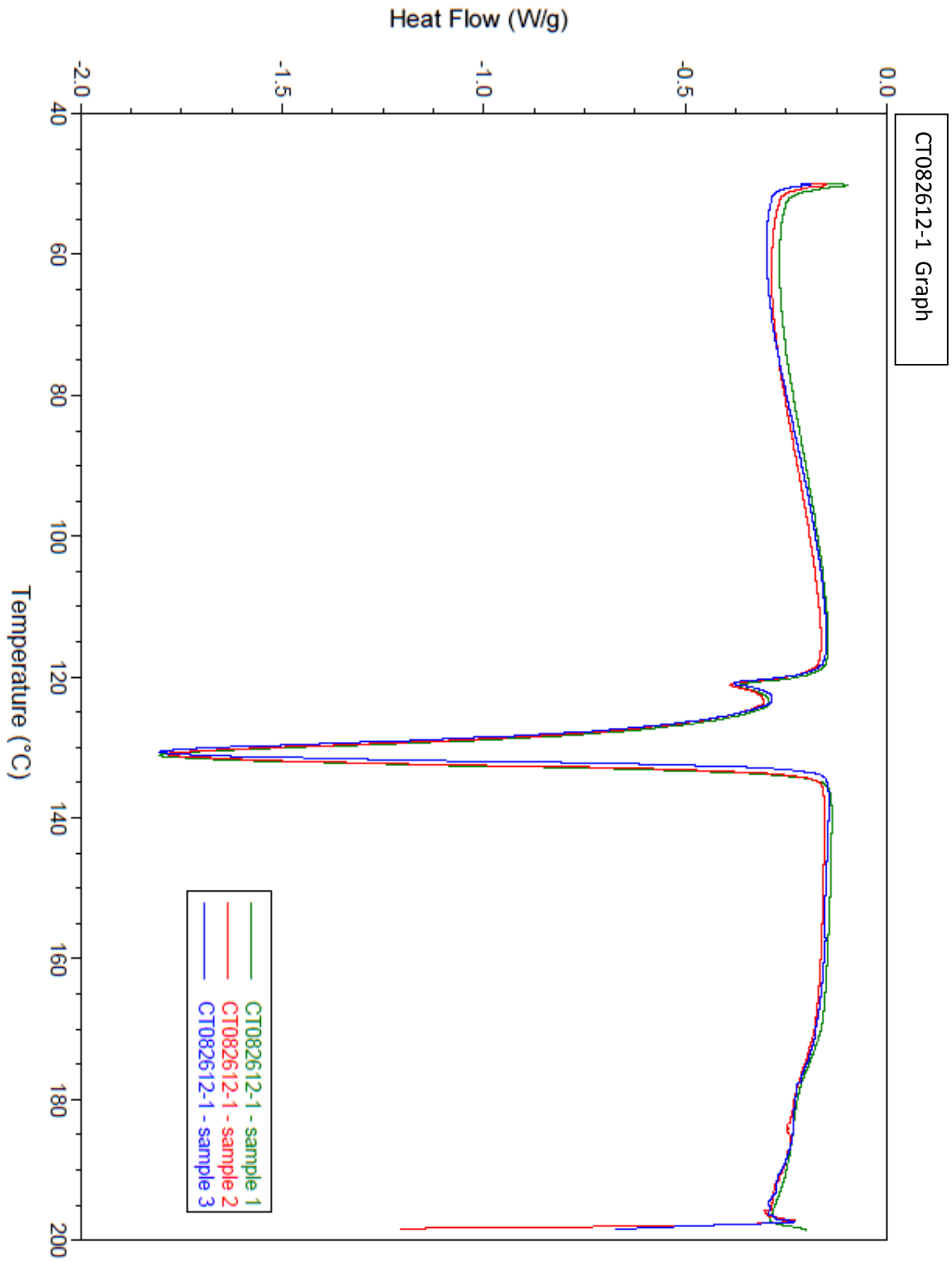

CT060512 Jar 2 Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
-1.4
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

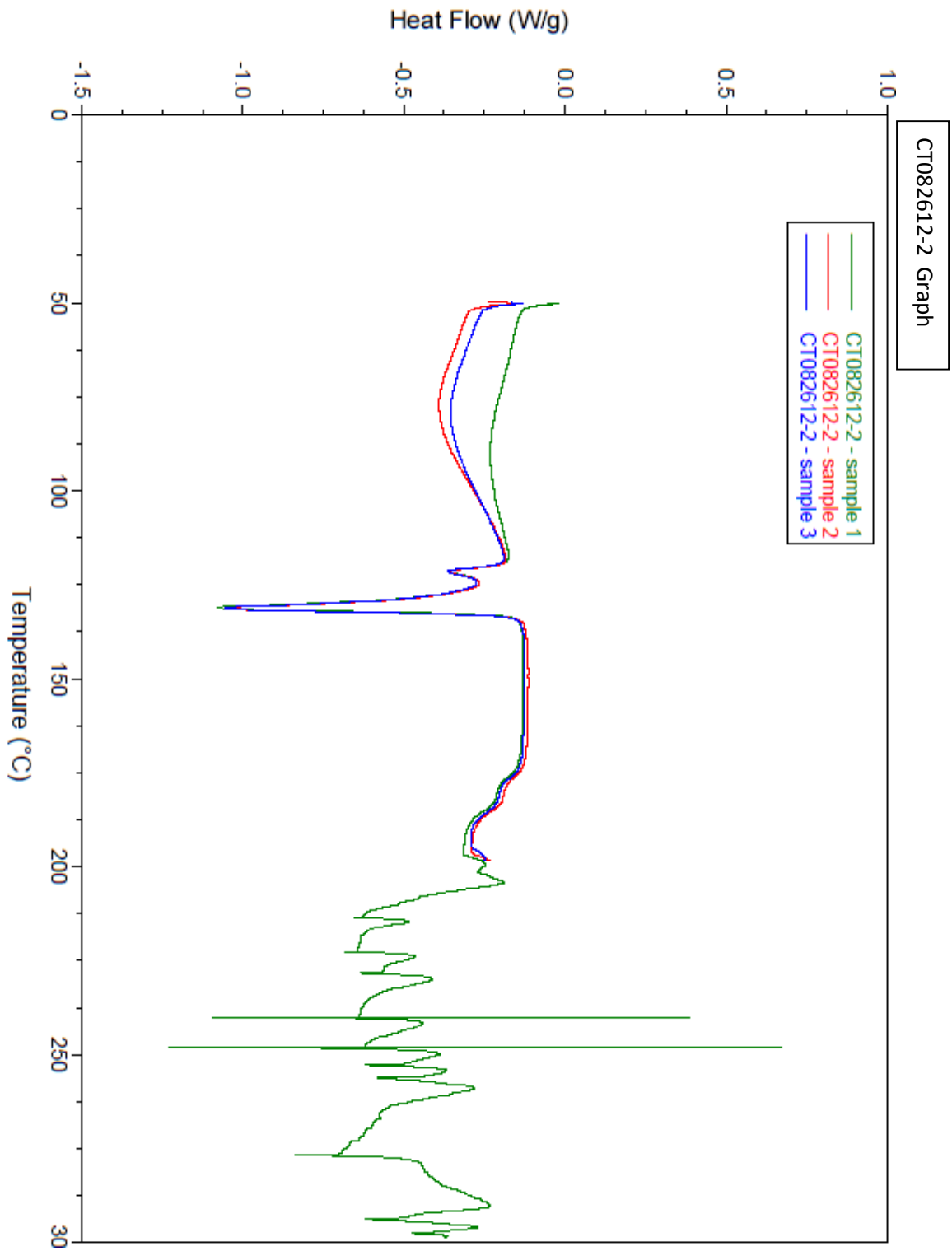

CT022612B2 Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
-1.4
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

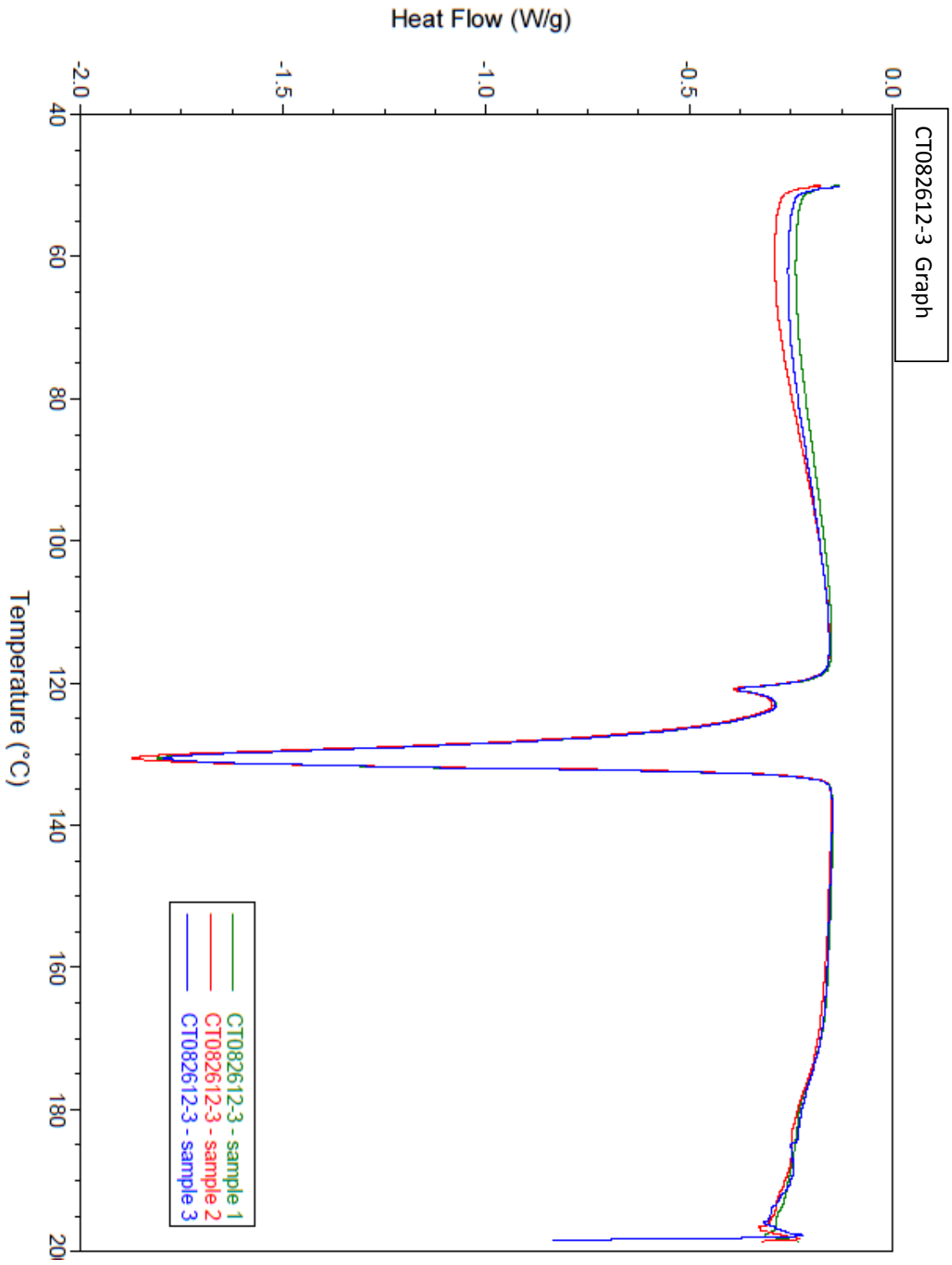

JCT071412A Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

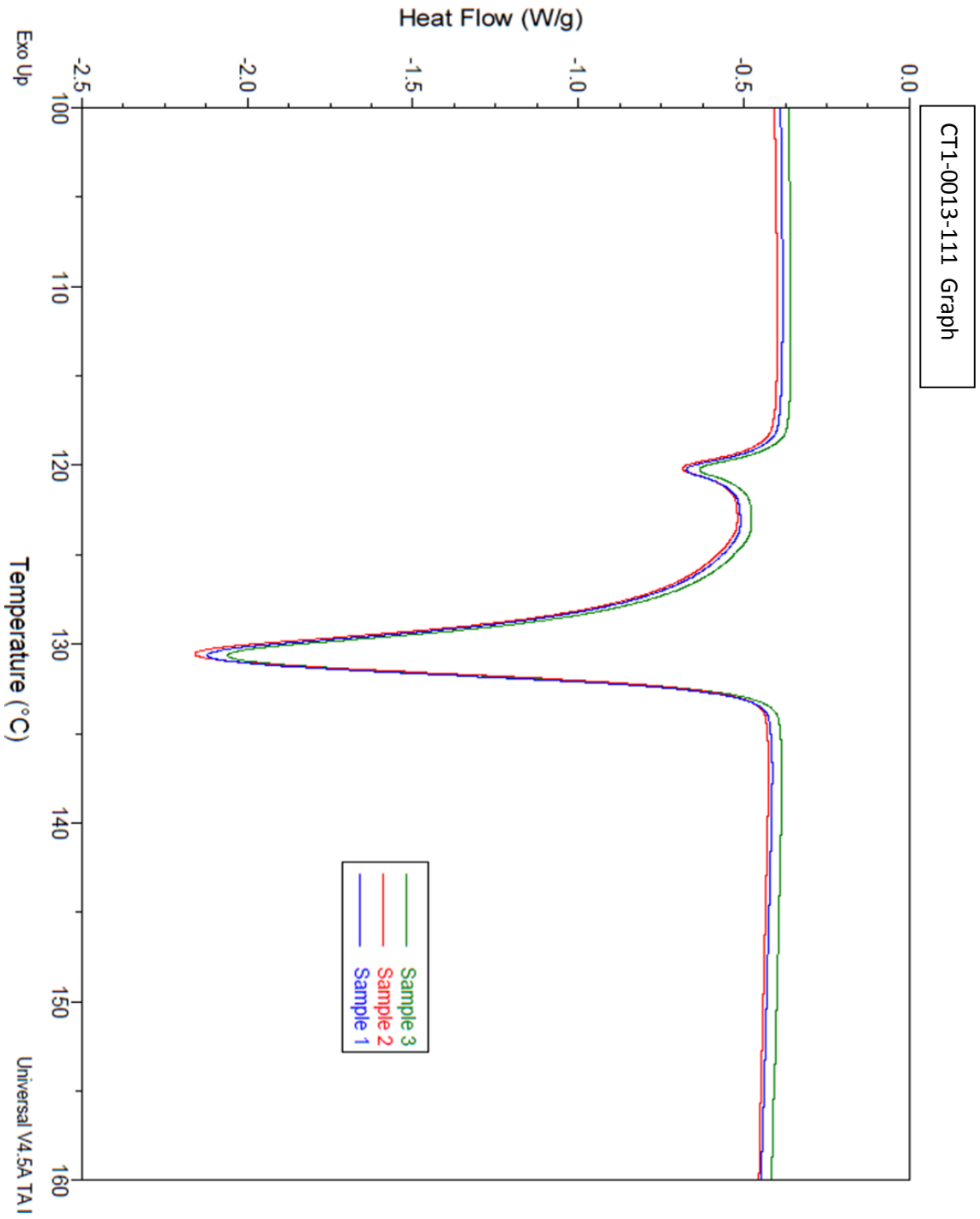

JCT071412B Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

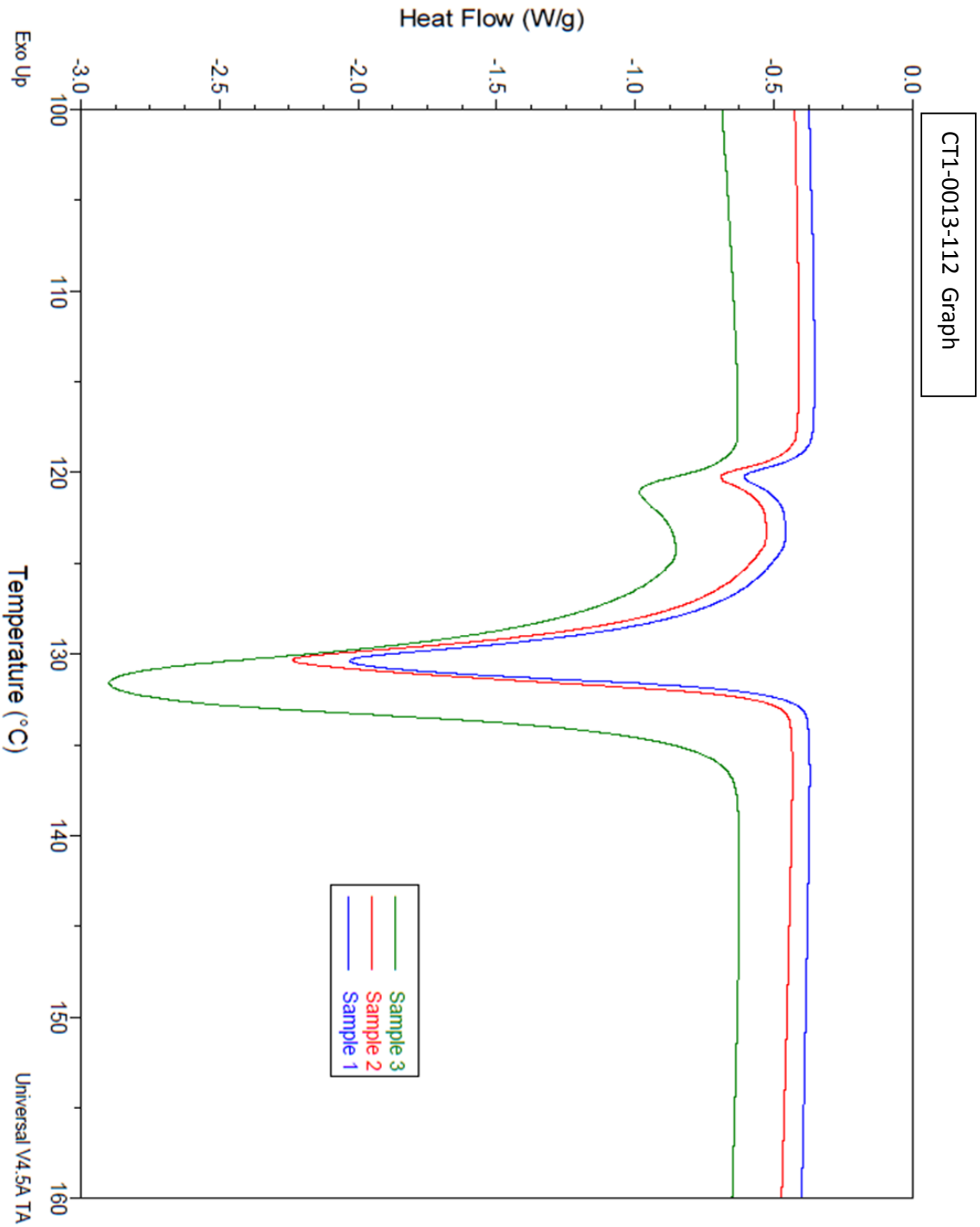

JCT071412C Graph
Heat Flow (W/g)
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
Exo Up
80
130
180
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

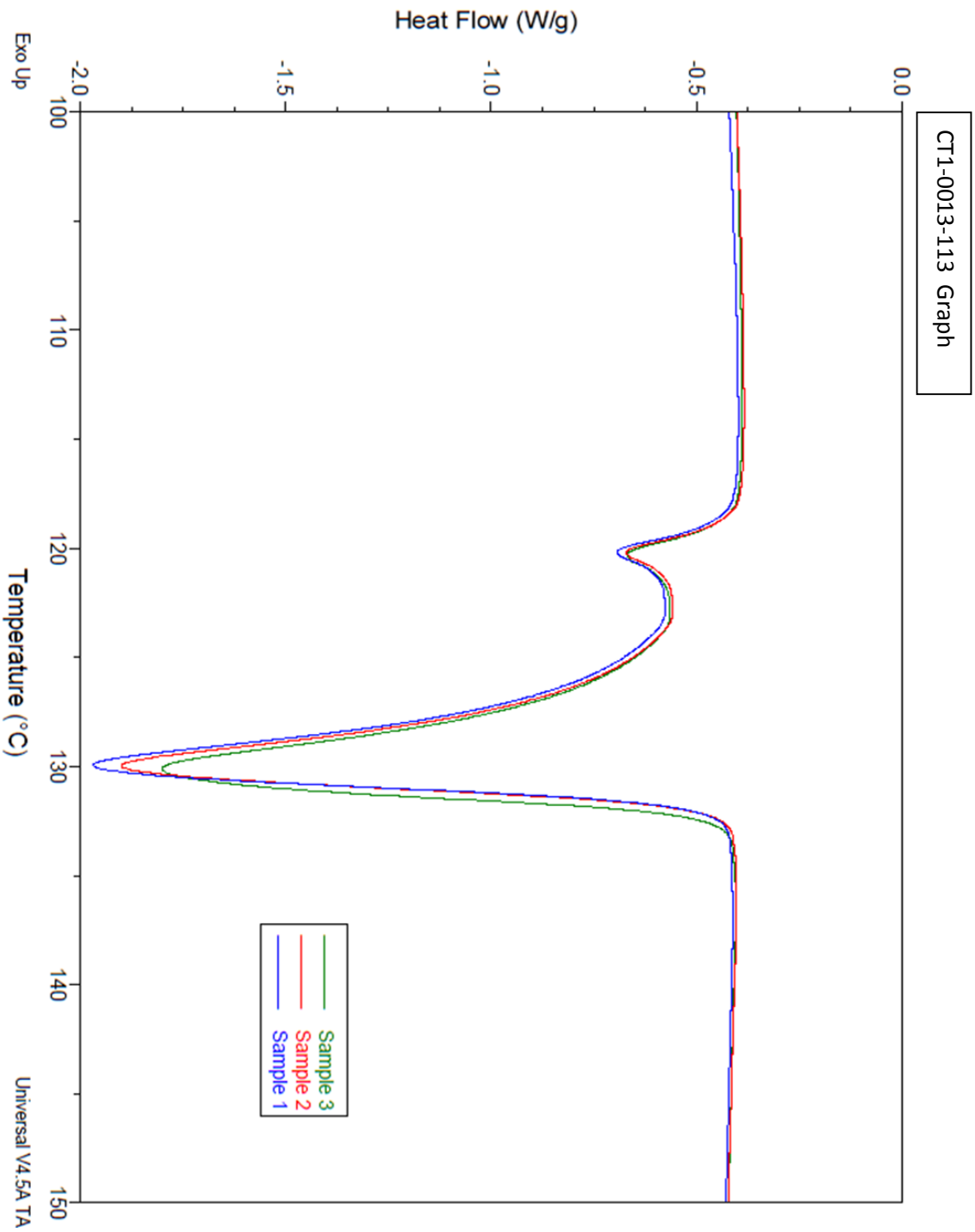

CT082612-1 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
40
60
80
100
120
140
160
180
200
Temperature (°C)
CT082612-1 - sample 1
CT082612-1 - sample 2
CT082612-1 - sample 3

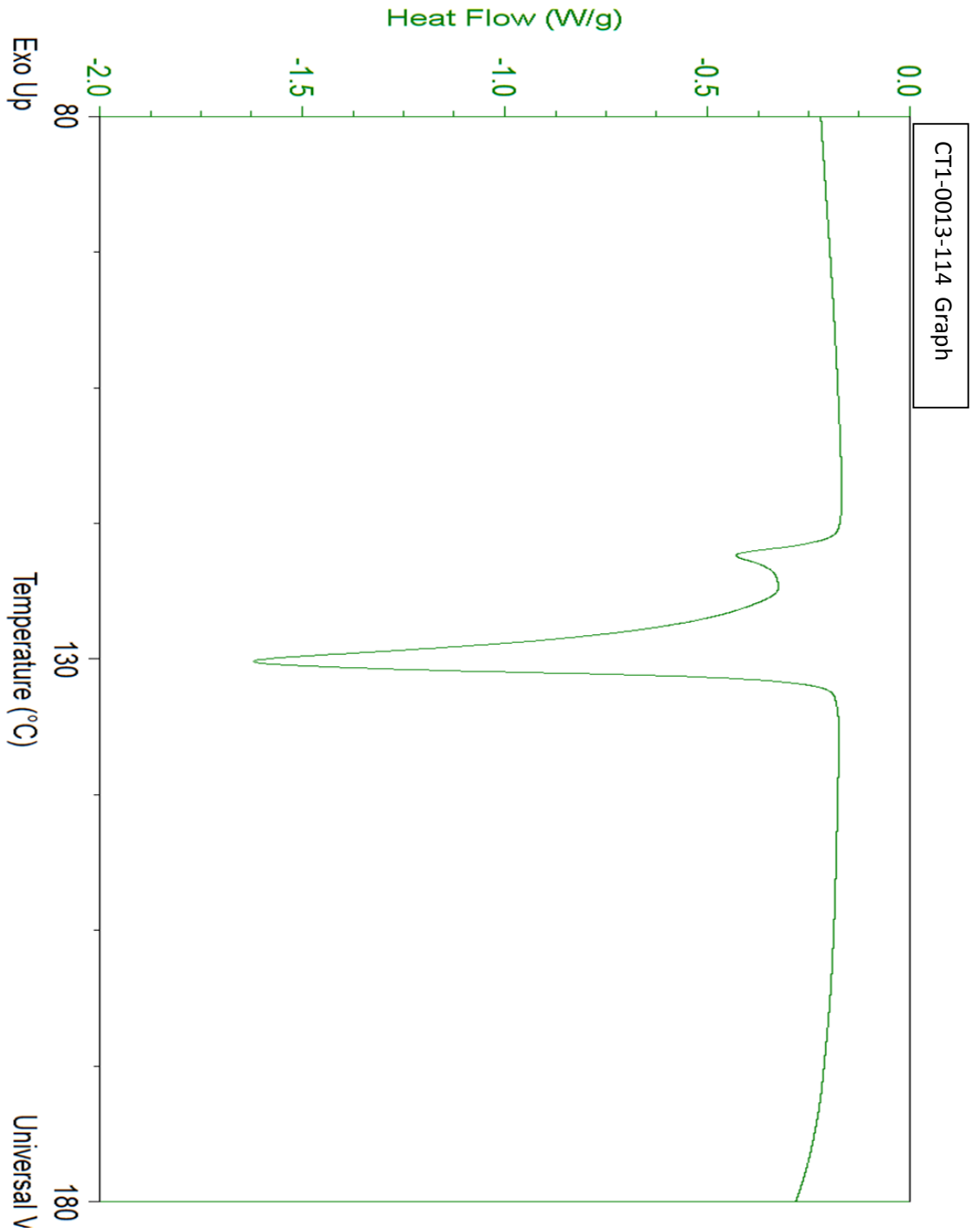

CT082612-2 Graph
CT082612-2 - sample 1
CT082612-2 - sample 2
CT082612-2 - sample 3
Heat Flow (W/g)
1.0
0.5
0.0
-0.5
-1.0
-1.5
Temperature (°C)
0
50
100
150
200
250
300

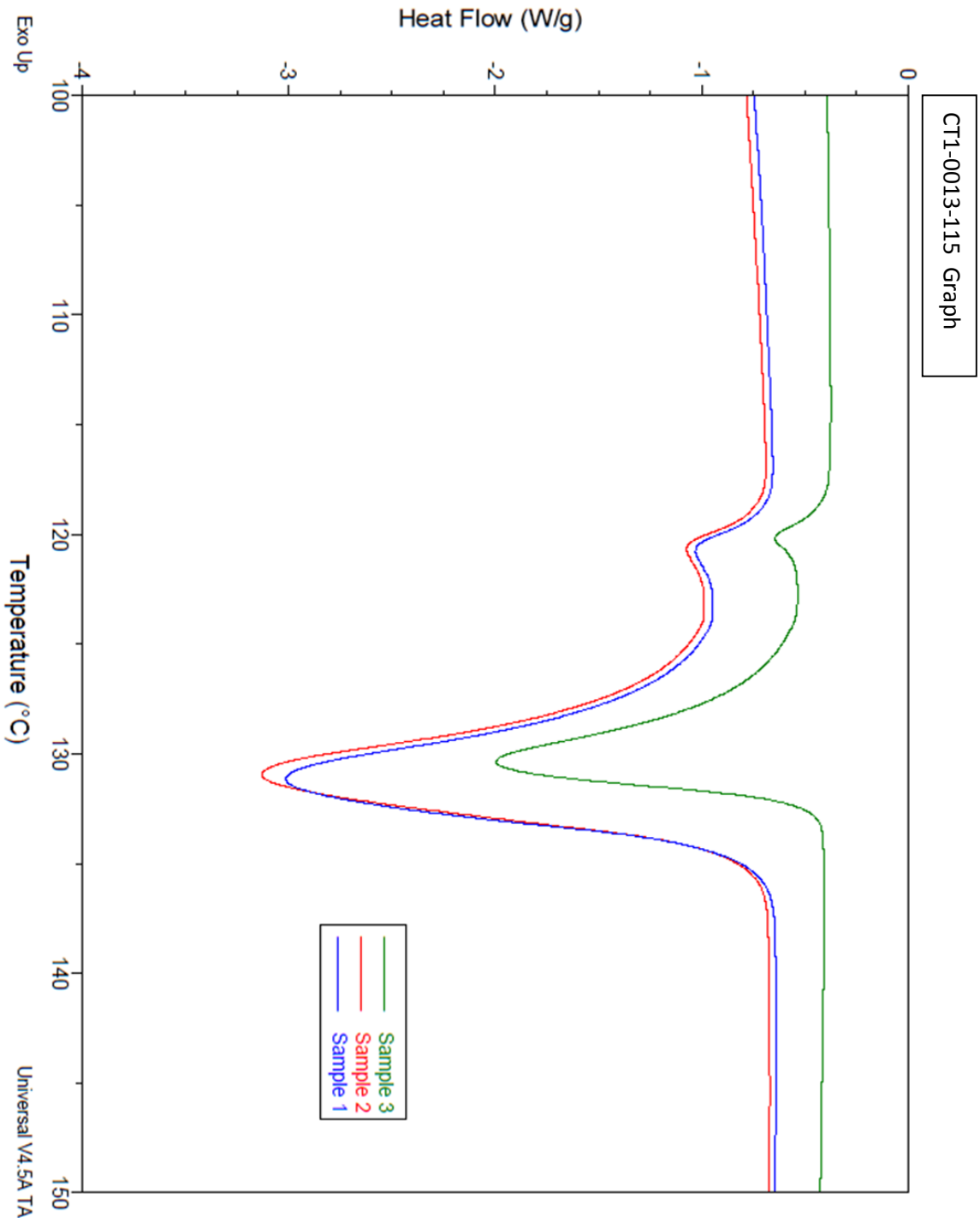

CT082612-3 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
40
60
80
100
120
140
160
180
200
Temperature (°C)
CT082612-3 - sample 1
CT082612-3 - sample 2
CT082612-3 - sample 3

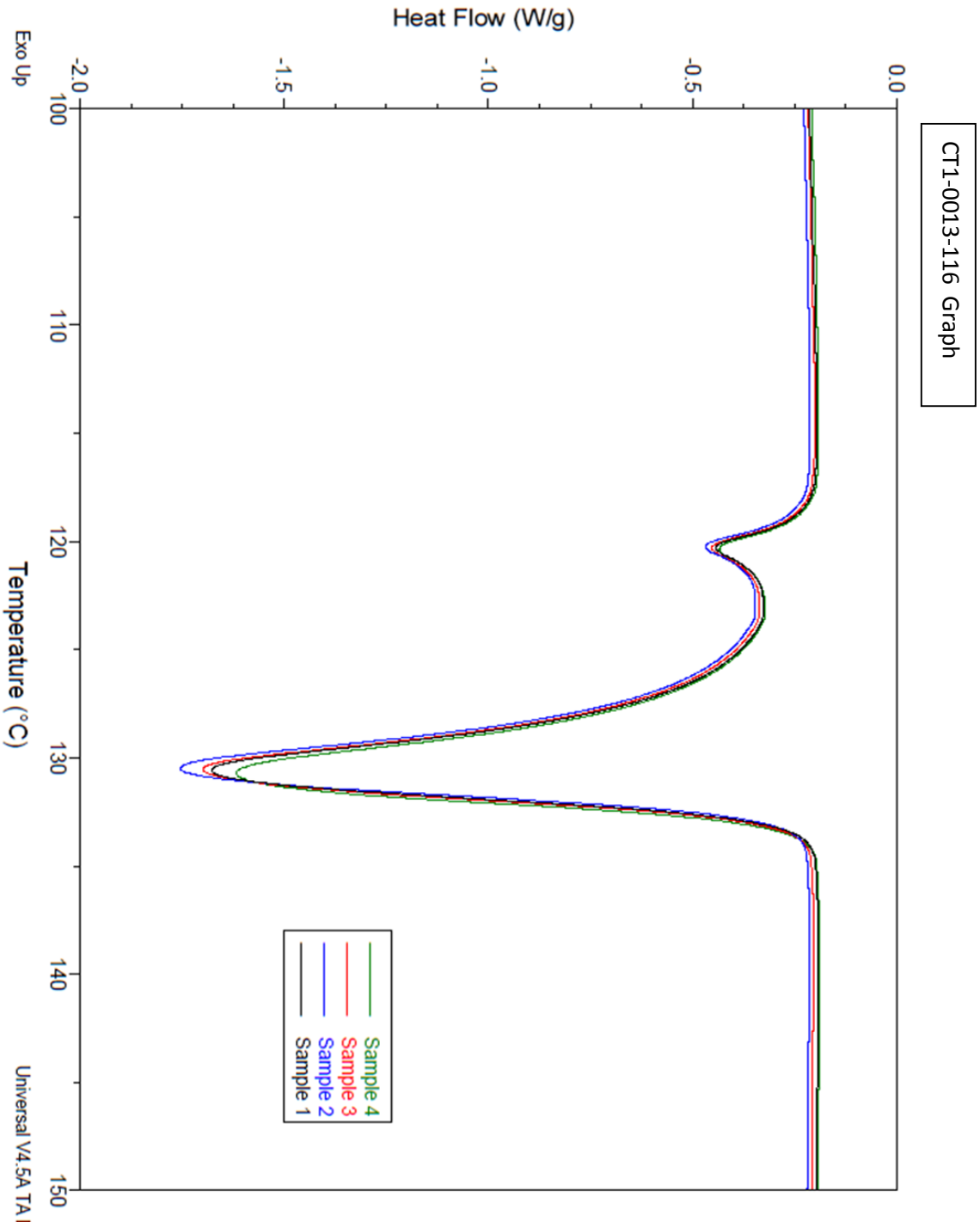

CT1-0013-111 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
-2.5
Exo Up
100
110
120
130
140
150
160
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA I

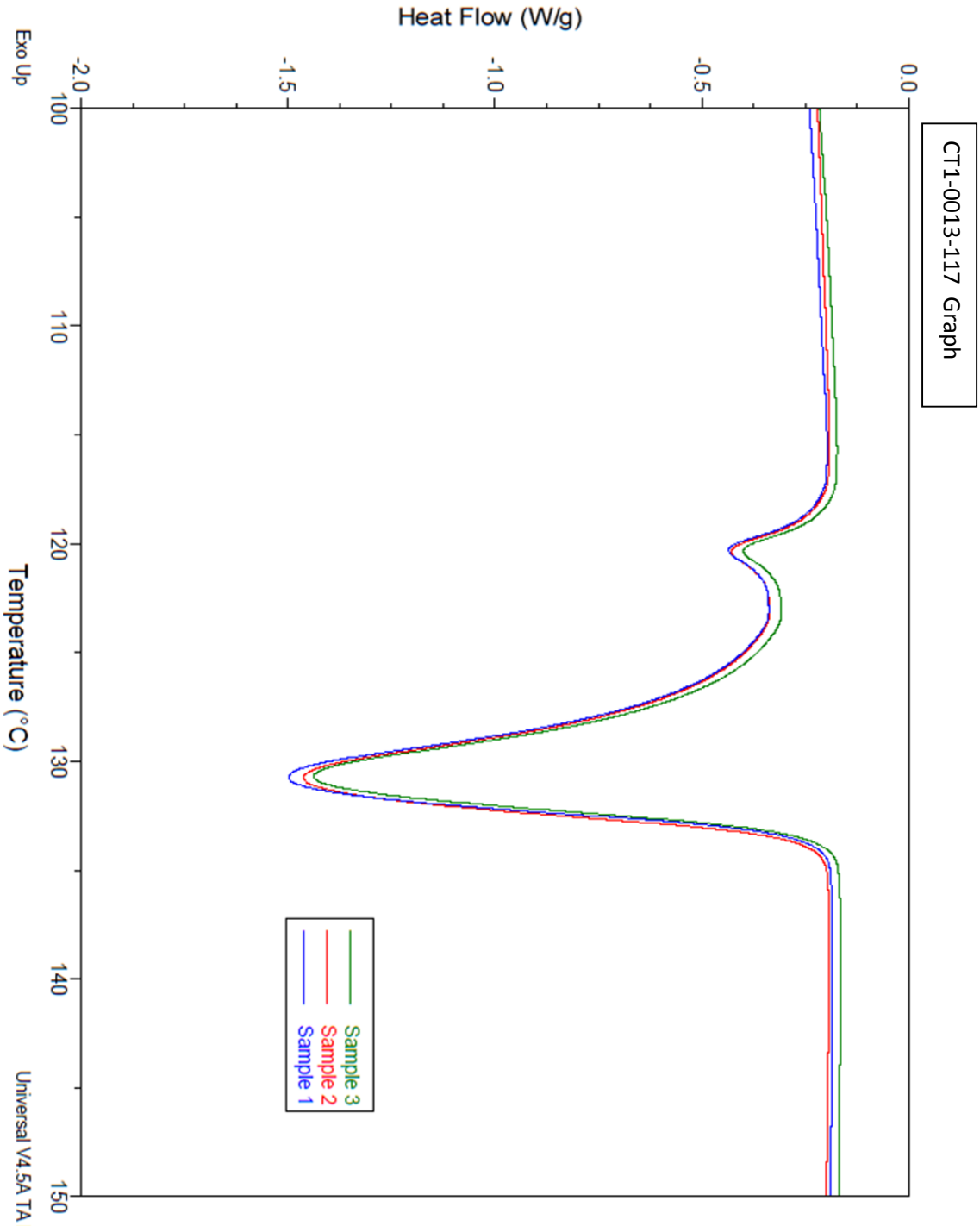

CT1-0013-112 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
-2.5
-3.0
Exo Up
100
110
120
130
140
150
160
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

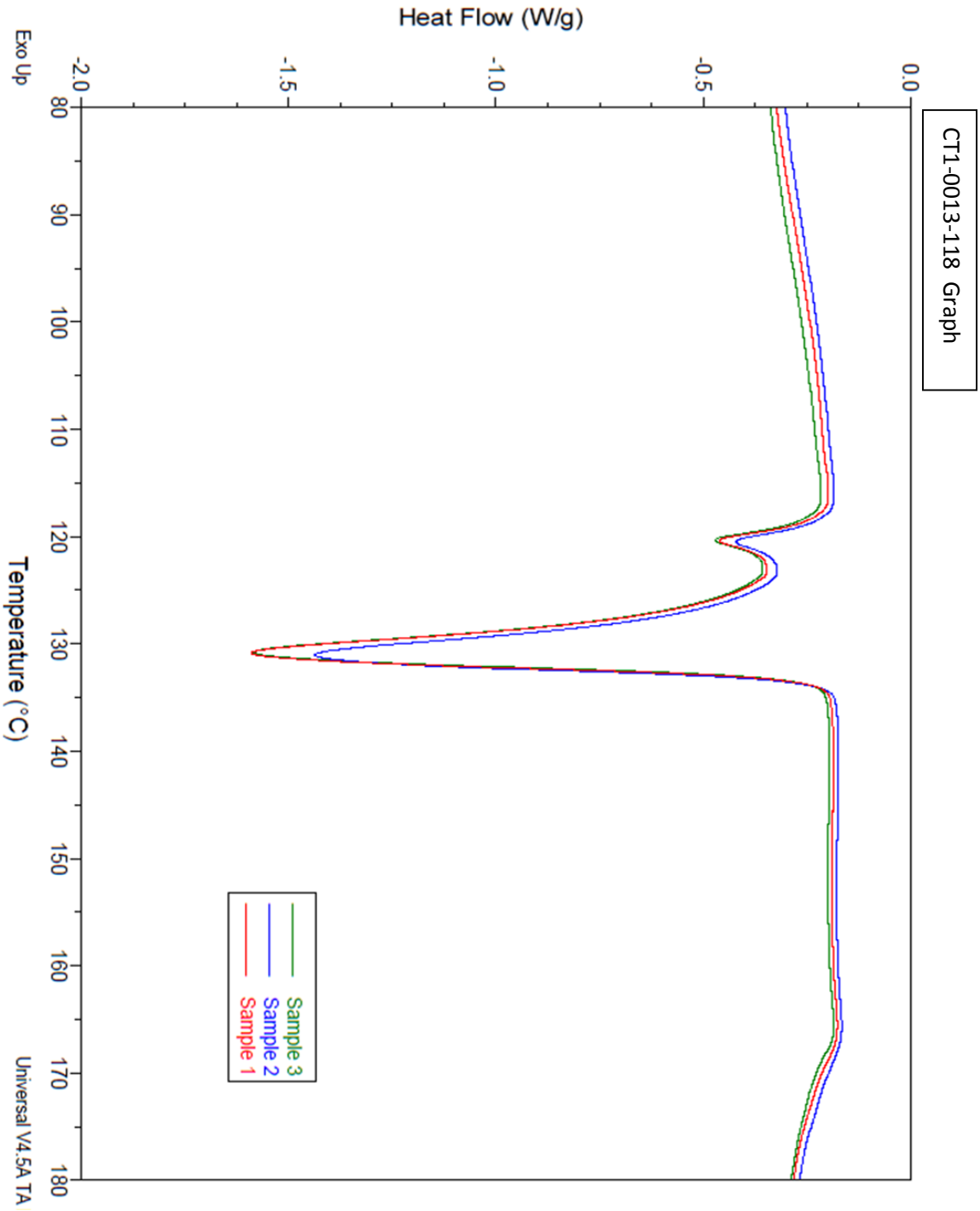

CT1-0013-113 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
100
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

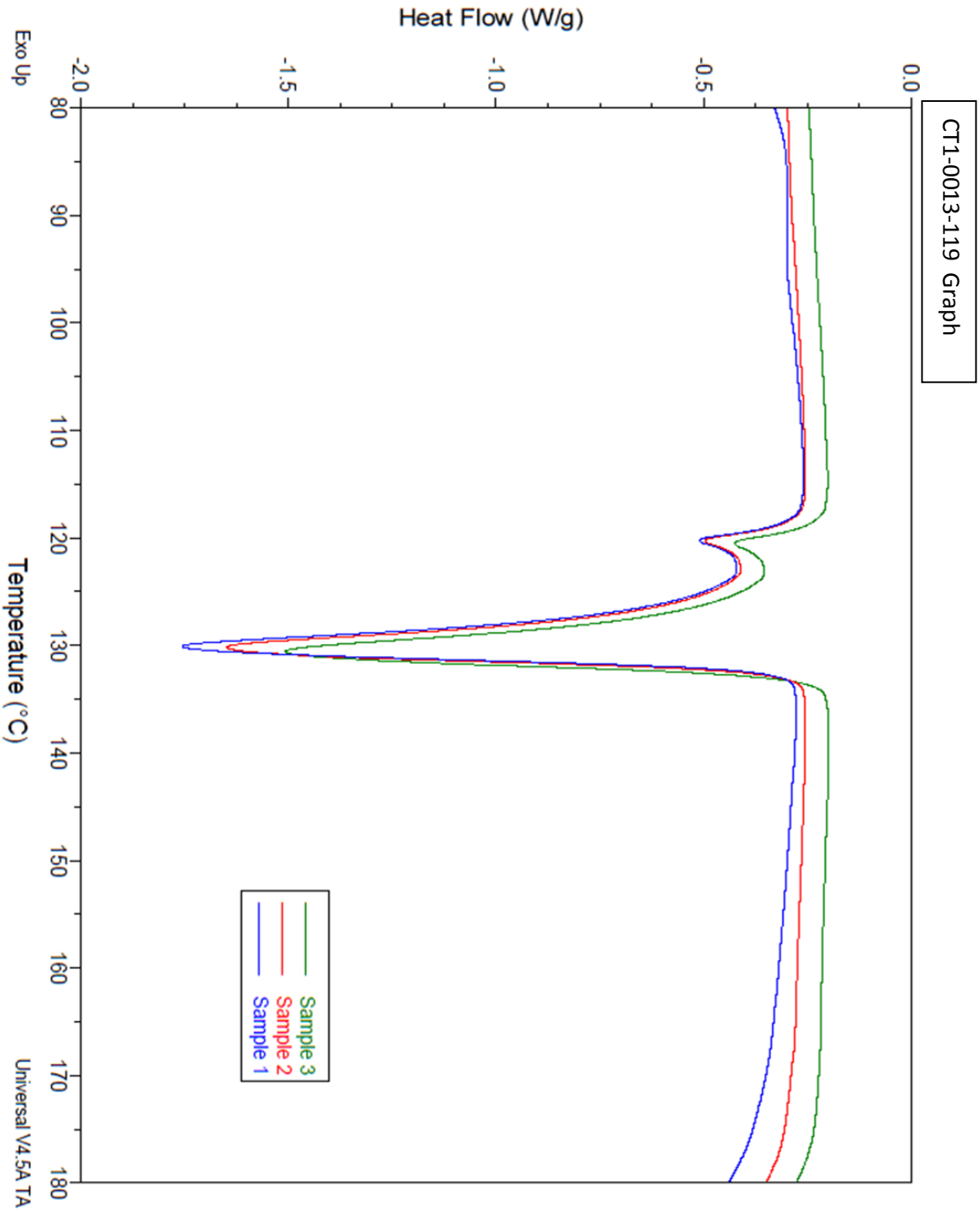

CT1-0013-114 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
80
130
180
Temperature (°C)
Universal V


CT1-0013-115 Graph
Heat Flow (W/g)
0
-1
-2
-3
-4
Exo Up
100
110
120
130
140
150
Temperature (°C)
Universal V4.5A TA
Sample 3
Sample 2
Sample 1


CT1-0013-116 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
100
110
120
130
140
150
Temperature (°C)
Sample 4
Sample 3
Sample 2
Sample 1
Universal V4.5A TA I


CT1-0013-117 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
100
110
120
130
140
150
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA


CT1-0013-118 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
80
90
100
110
120
130
140
150
160
170
180
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA


CT1-0013-119 Graph
Heat Flow (W/g)
0.0
-0.5
-1.0
-1.5
-2.0
Exo Up
80
90
100
110
120
130
140
150
160
170
180
Temperature (°C)
Sample 3
Sample 2
Sample 1
Universal V4.5A TA

Appendix 4A: Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading

Intertek

Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P20134502
Customer	: InfoSciTex Corporation Purchase Order # : 131716
Attention	: Carrie-Ann Ferreira
Analyst	: D. Carter
Date	: December 4, 2013

Test ID	: **Baseline**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.010 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: Unconditioned
Test Conditions	: **23°C ± 2°C / 50% ± 10% RH**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.001.F	1.00	0.54	661	1220	Adhesion to metal	100
P1404.SLS.002.F	1.00	0.51	690	1350	Adhesion to metal	100
P1404.SLS.003.F	1.00	0.51	768	1510	Adhesion / Cohesive	95 / 5
P1404.SLS.004.F	0.99	0.54	853	1600	Adhesion to metal	100
P1404.SLS.005.F	0.99	0.54	889	1660	Adhesion to metal	100
P1404.SLS.006.F	1.00	0.54	725	1340	Adhesion to metal	100
P1404.SLS.007.F	0.99	0.58	916	1600	Adhesion to metal	100
P1404.SLS.008.F	0.99	0.52	690	1340	Adhesion to metal	100
P1404.SLS.009.F	0.99	0.56	793	1430	Adhesion to metal	100
	Average		**776**	**1450**		
	Minimum		**661**	**1220**		
	Maximum		**918**	**1660**		

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Lap Shear Report Page 2 of 2

Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P20134502
Customer	: InfoSciTex Corporation Purchase Order # : 131716
Attention	: Carrie-Ann Ferreira
Analyst	: D. Carter
Date	: December 4, 2013

Test ID	: **Baseline**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.011 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: Unconditioned
Test Conditions	: **23°C ± 2°C / 50% ± 10% RH**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.001.117	0.99	0.53	1050	2000	Adhesion to metal	100
P1404.SLS.002.117	1.00	0.53	1030	1950	Adhesion to metal	100
P1404.SLS.003.117	1.00	0.55	1120	2040	Adhesion to metal	100
P1404.SLS.004.117	1.00	0.53	1080	2040	Adhesion to metal	100
P1404.SLS.005.117	0.99	0.51	998	1980	Adhesion to metal	100
P1404.SLS.006.117	0.99	0.55	892	1640	Adhesion to metal	100
P1404.SLS.007.117	0.99	0.54	1160	2170	Adhesion to metal	100
P1404.SLS.008.117	0.99	0.53	1100	2090	Adhesion to metal	100
P1404.SLS.009.117	0.99	0.51	963	1910	Adhesion to metal	100
	Average		**1040**	**1980**		
	Minimum		**892**	**1640**		
	Maximum		**1160**	**2170**		

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Lap Shear Report Page 1 of 2

Testing	**Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	ASTM D1002-10
Project Number	P201 34602
Customer	InfoSciTex Corporation Purchase Order # 131716
Attention	Carrie-Ann Ferrara
Analyst	D. Carter
Date	December 4, 2013

Test ID	**Dry Extreme Temperature - High**
Adhesive	Unknown
Adhesive Thickness (in)	0.010 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	Aluminum
Adherend Thickness (in)	0.063
Metal Preparation	Unknown
Bonding Conditions	Unknown
Test Speed	0.05 in/min
Sample Conditioning	Unconditioned
Test Conditions	**180°F (82°C)**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.019.F	0.98	0.51	1080	2150	Adhesion to metal	100
P1404.SLS.020.F	0.98	0.55	999	1850	Adhesion/Cohesion	95 / 5
P1404.SLS.021.F	1.00	0.53	1060	1970	Adhesion to metal	100
P1404.SLS.022.F	1.00	0.54	999	1850	Adhesion/Cohesion	95 / 5
P1404.SLS.023.F	0.98	0.51	973	1930	Adhesion/Cohesion	95 / 5
P1404.SLS.024.F	0.99	0.51	742	1470	Adhesion to metal	100
P1404.SLS.025.F	0.98	0.51	874	1750	Adhesion/Cohesion	95 / 5
P1404.SLS.026.F	0.98	0.51	832	1660	Adhesion to metal	100
P1404.SLS.027.F	0.98	0.52	825	1620	Adhesion to metal	100
	Average		919	1780		
	Minimum		742	1470		
	Maximum		1080	2150		

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Lap Shear Report Page 2 of 2

Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P20134602
Customer	: InfoSciTex Corporation Purchase Order # : 131716
Attention	: Carrie-Ann Ferreira
Analyst	: D. Carter
Date	: December 4, 2013

Test ID	: **Dry Extreme Temperature - High**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.010 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: Unconditioned
Test Conditions	: **180°F (82°C)**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.019.117	0.98	0.52	1440	2920	Adhesion/Cohesion	90 / 10
P1404.SLS.020.117	0.99	0.50	1390	2800	Adhesion/Cohesion	90 / 10
P1404.SLS.021.117	1.00	0.56	1460	2610	Adhesion/Cohesion	90 / 10
P1404.SLS.022.117	0.98	0.53	1520	2920	Adhesion/Cohesion	90 / 10
P1404.SLS.023.117	0.98	0.53	1570	3000	Adhesion/Cohesion	90 / 10
P1404.SLS.024.117	0.98	0.55	1760	3250	Adhesion/Cohesion	80 / 20
P1404.SLS.025.117	0.98	0.58	1730	3150	Adhesion/Cohesion	90 / 10
P1404.SLS.026.117	0.99	0.54	1680	3150	Adhesion/Cohesion	80 / 20
P1404.SLS.027.117	1.00	0.55	1800	3280	Adhesion/Cohesion	90 / 10
	Average		1590	3000		
	Minimum		1390	2610		
	Maximum		1800	3280		

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Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P2013/502
Customer	: InfoSc Tex Corporation Purchase Order # : 131718
Attention	: Carrie-Ann Ferreira
Analyst	: D. Carter
Date	: December 4, 2013

Test ID	: **Dry Extreme Temperature - Low**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.011 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: Unconditioned
Test Conditions	: **-67°F (-55°C)**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.010.F	0.99	0.56	649	1170	Adhesion to metal	100
P1404.SLS.011.F	1.00	0.55	971	1760	Adhesion to metal	100
P1404.SLS.012.F	1.00	0.53	670	1260	Adhesion to metal	100
P1404.SLS.013.F	0.99	0.54	592	1110	Adhesion to metal	100
P1404.SLS.014.F	1.00	0.55	905	1640	Adhesion to metal	100
P1404.SLS.015.F	0.98	0.50	743	1520	Adhesion to metal	100
P1404.SLS.016.F	0.97	0.49	870	1830	Adhesion to metal	100
P1404.SLS.017.F	0.99	0.53	856	1630	Adhesion to metal	100
P1404.SLS.018.F	0.99	0.52	824	1600	Adhesion to metal	100
	Average		787	1500		
	Minimum		592	1110		
	Maximum		971	1830		

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Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P20134502
Customer	: InfoScitex Corporation Purchase Order # : 131718
Attention	: Carrie-Ann Ferreira
Analyst	: D. Carter
Date	: December 4, 2013

Test ID	: **Dry Extreme Temperature - Low**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.011 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: Unconditioned
Test Conditions	: **-67°F (-55°C)**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.010.117	0.96	0.50	1290	2690	Adhesion to metal	100
P1404.SLS.011.117	0.99	0.53	886	1690	Adhesion to metal	100
P1404.SLS.012.117	0.98	0.52	1220	2380	Adhesion to metal	100
P1404.SLS.013.117	1.00	0.53	1260	2370	Adhesion to metal	100
P1404.SLS.014.117	0.99	0.55	1130	2080	Adhesion to metal	100
P1404.SLS.015.117	1.00	0.51	1150	2260	Adhesion to metal	100
P1404.SLS.016.117	1.00	0.51	1250	2440	Adhesion to metal	100
P1404.SLS.017.117	0.97	0.51	1130	2260	Adhesion to metal	100
P1404.SLS.018.117	0.97	0.56	1140	2090	Adhesion to metal	100
	Average		**1160**	**2250**		
	Minimum		**886**	**1690**		
	Maximum		**1290**	**2690**		

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Lap Shear Report Page 1 of 2

Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P20134502
Customer	: InfoSciTex Corporation Purchase Order # 131716
Attention	: Carrie Ann Ferreira
Analyst	: Frank Fay
Date	: January 30, 2014

Test ID	: **High Humidity Accelerated Aging**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.010 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: **30 days at 90°F, 95% RH**
Test Conditions	: **23°C ± 2°C / 50% ± 10% RH**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.046.FEN	0.99	0.55	602	1110	Adhesion to metal	100
P1404.SLS.047.FEN	0.96	0.55	651	1230	Adhesion to metal	100
P1404.SLS.048.FEN	0.97	0.55	597	1120	Adhesion to metal	100
P1404.SLS.049.FEN	0.99	0.54	427	799	Adhesion to metal	100
P1404.SLS.050.FEN	0.99	0.55	627	1150	Adhesion to metal	100
P1404.SLS.051.FEN	0.98	0.53	844	1620	Adhesion to metal	100
P1404.SLS.052.FEN	0.99	0.48	445	930	Adhesion to metal	100
P1404.SLS.053.FEN	0.98	0.55	522	970	Adhesion to metal	100
P1404.SLS.054.FEN	0.99	0.54	518	969	Adhesion to metal	100
	Average		**582**	**1100**		
	Minimum		**427**	**799**		
	Maximum		**844**	**1620**		

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Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10
Project Number	: P2C134602
Customer	: InfoSciTex Corporation Purchase Order # : 131716
Attention	: Carrie-Ann Ferreira
Analyst	: Frank Foy
Date	: January 30, 2014

Test ID	: **High Humidity Acclerated Aging**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.011 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.063
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: **30 days at 90°F, 95% RH**
Test Conditions	: **23°C ± 2°C / 50% ± 10% RH**

Sample Name	Wicth (in)	Overlap Length (in)	Maximum Load (lbf)	Aoparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.046.117	0.98	0.52	500	981	Adhesion to metal	100
P1404.SLS.047.117	0.98	0.52	769	1510	Adhesion to metal	100
P1404.SLS.048.117	0.99	0.53	1040	1980	Adhesion to metal	100
P1404.SLS.049.117	0.98	0.51	862	1720	Adhesion to metal	100
P1404.SLS.050.117	0.98	0.52	585	1150	Adhesion to metal	100
P1404.SLS.051.117	1.00	0.56	619	1110	Adhesion to metal	100
P1404.SLS.052.117	0.99	0.55	840	1540	Adhesion to metal	100
P1404.SLS.053.117	1.00	0.56	808	1090	Adhesion to metal	100
P1404.SLS.054.117	0.98	0.52	831	1630	Adhesion to metal	100
	Average		**739**	**1410**		
	Minimum		**500**	**981**		
	Maximum		**1040**	**1980**		

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Lap Shear Report Page 2 of 2

Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10 (modified number of specimens)
Project Number	: P3C134602 Purchase Order # [illegible]
Customer	: InfoSciTex Corporation
Attention	: Carrie Ann Ferreira
Analyst	: M. Brady / G. Sirne
Date	: March 31, 2014

Test ID	: **Accelerated Aging**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.006 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.064
Metal Pretreatment	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: **90 days at 120°F / 50% RH**
Test Conditions	: **23°C ± 2°C / 50% ± 10% RH**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.037.FEN	1.00	0.52	644	1240	Adhesion to metal	100
P1404.SLS.038.FEN	1.02	0.56	595	1100	Adhesion to metal	100
P1404.SLS.039.FEN	1.00	0.54	595	1100	Adhesion to metal	100
	Average		**621**	**1150**		
	Minimum		**595**	**1100**		
	Maximum		**644**	**1240**		

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Lap Shear Report Page 1 of 2

Testing	: **Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	: ASTM D1002-10 (modified number of specimens)
Project Number	: P2013/502 Purchase Order # : 121716
Customer	: InfoSciTex Corporation
Attention	: Carrie-Ann Ferreira
Analyst	: M. Brady / G. Sime
Date	: March 31, 2014

Test ID	: **Accelerated Aging**
Adhesive	: Unknown
Adhesive Thickness (in)	: 0.008 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	: Aluminum
Adherend Thickness (in)	: 0.064
Metal Preparation	: Unknown
Bonding Conditions	: Unknown
Test Speed	: 0.05 in/min
Sample Conditioning	: **90 days at 120°F / 50% RH**
Test Conditions	: **23°C ± 2°C / 50% ± 10% RH**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.037.117	0.97	0.53	580	1130	Adhesion to metal	100
P1404.SLS.038.117	1.01	0.55	777	1410	Adhesion to metal	100
P1404.SLS.039.117	1.00	0.55	863	1570	Adhesion to metal	100
	Average		**740**	**1370**		
	Minimum		**580**	**1130**		
	Maximum		**863**	**1570**		

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Testing	**Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	ASTM D1002-10
Project Number	P20134502 Purchase Order #: 131716
Customer	InfoSciTex Corporation
Attention	Carrie Ann Ferreira
Analyst	M. Brady / G. Sime
Date	March 31, 2014

Test ID	**Accelerated Aging**
Adhesive	Unknown
Adhesive Thickness (in)	0.010 (average) Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	Aluminum
Adherend Thickness (in)	0.064
Metal Preparation	Unknown
Bonding Conditions	Unknown
Test Speed	0.05 in/min
Sample Conditioning	**90 days at 120°F / 50% RH**
Test Conditions	**180°F (82°C)**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.040.FEN	1.00	0.52	808	1550	Adhesion to metal	100
P1404.SLS.041.FEN	0.98	0.56	771	1430	Adhesion to metal	100
P1404.SLS.042.FEN	0.99	0.55	561	1030	Adhesion to metal	100
P1404.SLS.043.FEN	1.00	0.56	744	1350	Adhesion to metal	100
P1404.SLS.044.FEN	0.99	0.56	716	1310	Adhesion to metal	100
P1404.SLS.045.FEN	0.97	0.52	548	1090	Adhesion to metal	100
	Average		**691**	**1290**		
	Minimum		**548**	**1030**		
	Maximum		**808**	**1550**		

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Lap Shear Report Page 1 of 2

Testing	**Apparent Shear Strength Of Single-Lap-Joint Adhesively Bonded Metal Specimens By Tension Loading**
Test Method	ASTM D1002-10
Project Number	P23134502 Purchase Order # 131716
Customer	InfoSciTex Corporation
Attention	Carrie-Ann Ferreira
Analyst	N. Brady / G. Sime
Date	March 31, 2014

Test ID	**Accelerated Aging**
Adhesive	Unknown
Adhesive Thickness (in)	0.010 (average)
	Determined by subtracting adherend thickness from overall lap joint thickness
Adherend Type	Aluminum
Adherend Thickness (in)	0.064
Metal Preparation	Unknown
Bonding Conditions	Unknown
Test Speed	0.05 in/min
Sample Conditioning	**90 days at 120°F / 50% RH**
Test Conditions	**180°F (82°C)**

Sample Name	Width (in)	Overlap Length (in)	Maximum Load (lbf)	Apparent Shear Strength (psi)	Type Of Failure	Amount of Failure (%)
P1404.SLS.040.117	1.00	0.53	781	1560	Adhesion to metal	90
P1404.SLS.041.117	0.99	0.56	1270	2280	Adhesion to metal	90
P1404.SLS.042.117	1.00	0.54	934	1730	Adhesion to metal	90
P1404.SLS.043.117	1.00	0.53	950	1790	Adhesion to metal	90
P1404.SLS.044.117	0.99	0.53	1390	2650	Adhesion to metal	90
P1404.SLS.045.117	0.99	0.54	1020	1910	Adhesion to metal	90
	Average		**1060**	**1990**		
	Minimum		**781**	**1560**		
	Maximum		**1390**	**2650**		

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Appendix 4B: Strength Properties of Double Lap Shear Adhesive Joints

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Testing	:	Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading
Test Method	:	ASTM D3528-96 (Reapproved 2008)
Project Number	:	P20134502
Customer	:	InfoSciTex Corporation Purchase Order # : 131718
Attention	:	Carrie-Ann Ferreira
Analyst	:	D. Carter
Date	:	December 5, 2013

Test ID	:	**Baseline**
Specimen Type	:	A
Adhesive	:	Unknown
Substrate	:	Aluminum
Bonding Conditions	:	Bonded by customer
Sample Conditioning	:	None
Test Conditions	:	23°C ± 2°C / 50% ± 10% RH
Crosshead Speed	:	0.05 in/min
Significance	:	ASTM D3528 specifies that dimensions be measured to the nearest 0.01 in and that load and stress be reported to 3 significant figures

Sample Name	Total Adhesive Thickness (in)	Specimen Width (in)	Average Bond Overlap (in)	Maximum Load (lbf)	Ultimate Strength (PSI)	Type Of Failure
P1404.DLS.001.FEN	0.019	0.98	0.54	2020	1910	Adhesion to metal / 100%
P1404.DLS.002.FEN	0.018	0.93	0.54	2280	2250	Adhesion to metal / 100%
P1404.DLS.003.FEN	0.020	0.97	0.53	2510	2440	Adhesion to metal / 100%
P1404.DLS.004.FEN	0.019	0.97	0.53	2340	2280	Adhesion to metal / 100%
P1404.DLS.005.FEN	0.021	0.98	0.53	2640	2540	Adhesion to metal / 100%
P1404.DLS.006.FEN	0.020	0.92	0.55	2130	2100	Adhesion to metal / 100%
P1404.DLS.007.FEN	0.021	0.96	0.54	2780	2560	Adhesion to metal / 100%
P1404.DLS.008.FEN	0.024	0.98	0.54	2050	1940	Adhesion to metal / 100%
P1404.DLS.009.FEN	0.018	0.98	0.54	3500	3310	Adhesion to metal / 100%
Maximum	0.024	0.98	0.55	3500	3310	
Minimum	0.018	0.92	0.53	2020	1910	
Average	**0.020**	**0.96**	**0.54**	**2470**	**2380**	
Std. Dev.	0.002	0.02	0.01	466	432	
C.O.V. (%)	9	2	1	19	18	

Bond area calculation based on "best effort" measurement of bond dimensions.

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Testing	:	Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading
Test Method	:	ASTM D3528-96 (Reapproved 2008)
Project Number	:	P20134502
Customer	:	InfoSciTex Corporation Purchase Order # : 131718
Attention	:	Carrie-Ann Ferreira
Analyst	:	D. Carter
Date	:	December 5, 2013

Test ID	:	**Baseline**
Specimen Type	:	A
Adhesive	:	Unknown
Substrate	:	Aluminum
Bonding Conditions	:	Bonded by customer
Sample Conditioning	:	None
Test Conditions	:	23°C ± 2°C / 50% ± 10% RH
Crosshead Speed	:	0.05 in/min
Significance	:	ASTM D3528 specifies that dimensions be measured to the nearest 0.01 in and that load and stress be reported to 3 significant figures.

Sample Name	Total Adhesive Thickness (in)	Specimen Width (in)	Average Bond Overlap (in)	Maximum Load (lbf)	Ultimate Strength (PSI)	Type Of Failure
P1404.DLS.001.117	0.021	0.98	0.54	2840	2880	Adhesion to metal / 100%
P1404.DLS.002.117	0.024	0.98	0.54	2890	2730	Adhesion to metal / 100%
P1404.DLS.003.117	0.024	0.97	0.54	3760	3590	Adhesion to metal / 100%
P1404.DLS.004.117	0.027	0.97	0.53	2850	2770	Adhesion to metal / 100%
P1404.DLS.005.117	0.021	0.94	0.53	2330	2310	Adhesion to metal / 100%
P1404.DLS.006.117	0.020	0.96	0.54	3030	2860	Adhesion to metal / 100%
P1404.DLS.007.117	0.021	0.96	0.52	2710	2710	Adhesion to metal / 100%
P1404.DLS.008.117	0.025	0.97	0.52	2620	2800	Adhesion to metal / 100%
P1404.DLS.009.117	0.020	0.98	0.53	2920	2810	Adhesion to metal / 100%
Maximum	0.027	0.96	0.54	3760	3590	
Minimum	0.020	0.94	0.52	2330	2310	
Average	**0.023**	**0.97**	**0.53**	**2880**	**2780**	
Std. Dev.	0.003	0.01	0.01	393	342	
C.O.V. (%)	11	1	2	14	12	

Bond area calculation based on "best effort" measurement of bond dimensions.

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Intertek

Testing	: **Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading**
Test Method	: ASTM D3528-96 (Reapproved 2008)
Project Number	: P20134502
Customer	: InfoSciTex Corporation Purchase Order # : 131716
Attention	: Carrie-Ann Ferreira
Analyst	: Frank Foy
Date	: January 29, 2014

Test ID	: **30 days at 90°F, 95% RH**
Specimen Type	: A
Adhesive	: Unknown
Substrate	: Aluminum
Bonding Conditions	: Bonded by customer
Sample Conditioning	: None
Test Conditions	: 23°C ± 2°C / 50% ± 10% RH
Crosshead Speed	: 0.05 in/min
Significance	: ASTM D3528 specifies that dimensions be measured to the nearest 0.01 in and that load and stress be reported to 3 significant figures.

Sample Name	Total Adhesive Thickness (in)	Specimen Width (in)	Average Bond Overlap (in)	Maximum Load (lbf)	Ultimate Strength (PSI)	Type Of Failure
P1404.DLS.010.FEN	0.023	0.91	0.60	1730	1580	Adhesion to metal / 100%
P1404.DLS.011.FEN	0.022	0.93	0.71	1950	1480	Adhesion to metal / 100%
P1404.DLS.012.FEN	0.017	0.95	0.54	1860	1810	Adhesion to metal / 100%
P1404.DLS.013.FEN	0.021	0.90	0.60	2070	1920	Adhesion to metal / 100%
P1404.DLS.014.FEN	0.025	0.97	0.55	1880	1760	Adhesion to metal / 100%
P1404.DLS.015.FEN	0.019	0.97	0.54	1740	1660	Adhesion to metal / 100%
P1404.DLS.016.FEN	0.020	0.97	0.54	1710	1630	Adhesion to metal / 100%
P1404.DLS.017.FEN	0.021	0.98	0.58	1820	1600	Adhesion to metal / 100%
P1404.DLS.018.FEN	0.025	0.98	0.53	2190	2110	Adhesion to metal / 100%
Maximum	0.025	0.98	0.71	2190	2110	
Minimum	0.017	0.90	0.53	1710	1480	
Average	**0.021**	**0.95**	**0.58**	**1880**	**1730**	
Std. Dev	0.003	0.03	0.06	163	195	
C.O.V. (%)	12	3	10	9	11	

Bond area calculation based on "best effort" measurement of bond dimensions.

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Intertek

Testing	**Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading**
Test Method	ASTM D3528-96 (Reapproved 2008)
Project Number	P2C134602
Customer	InfoSciTex Corporation Purchase Order # 131716
Attention	Carrie-Ann Ferreira
Analyst	Frank Foy
Date	January 29, 2014

Test ID	**30 days at 90°F, 95% RH**
Specimen Type	A
Adhesive	Unknown
Substrate	Aluminum
Bonding Conditions	Bonded by customer
Sample Conditioning	None
Test Conditions	23°C ± 2°C / 50% ± 10% RH
Crosshead Speed	0.05 in/min
Significance	ASTM D3528 specifies that dimensions be measured to the nearest 0.01 in and that load and stress be reported to 3 significant figures.

Sample Name	Total Adhesive Thickness (in)	Specimen Width (in)	Average Bond Overlap (in)	Maximum Load (lbf)	Ultimate Strength (PSI)	Type Of Failure
P1404.DLS.010.117	0.026	0.99	0.52	2390	2320	Adhesion to metal / 100%
P1404.DLS.011.117	0.019	0.97	0.54	2470	2360	Adhesion to metal / 100%
P1404.DLS.012.117	0.021	0.99	0.53	1930	1840	Adhesion to metal / 100%
P1404.DLS.013.117	0.028	0.95	0.54	3190	3110	Adhesion to metal / 100%
P1404.DLS.014.117	0.024	0.97	0.54	2450	2340	Adhesion to metal / 100%
P1404.DLS.015.117	0.021	0.98	0.54	2650	2500	Adhesion to metal / 100%
P1404.DLS.016.117	0.028	0.98	0.52	2530	2480	Adhesion to metal / 100%
P1404.DLS.017.117	0.025	0.98	0.53	2400	2310	Adhesion to metal / 100%
P1404.DLS.018.117	0.023	0.98	0.53	2610	2510	Adhesion to metal / 100%
Maximum	0.028	0.99	0.54	3190	3110	
Minimum	0.019	0.95	0.52	1930	1840	
Average	**0.024**	**0.98**	**0.53**	**2510**	**2420**	
Std. Dev.	0.003	0.01	0.01	326	326	
C.O.V. (%)	13	1	2	13	14	

Bond area calculation based on "best effort" measurement of bond dimensions.

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Appendix 4C: Climbing Drum Peel for Adhesives

Intertek

Peel Resistance Report Page 1 of 2

Testing	: **Climbing Drum Peel for Adhesives**
Test Method	: ASTM D1781 - 98 (Reapproved 2012) - Modified specimen length and peel distance
Project Number	: P201345C2
Customer	: InfoSciTex Corporation Purchase Order # 131716
Attention	: Carrie-Ann Ferreira
Analyst	: D. Midgette / K. Schuman
Date	: December 23, 2013

Adherend Description	: Aluminum
Adherend Thickness (in)	: 0.02
Sample Dimensions (in)	: 3 x 0.5 x 1" (nominal)
Bonding Conditions	: Not Supplied
Sample Preparation	: Remedial machining performed by Intertek PTL
Conditioning	: Unconditioned
Test Machine Type	: Instron 5985
Speed Of Testing	: 1.0 in/min
Flange Radius (in)	: 2.51
Drum Radius (in)	: 2.01
Calibration Method	: Adherend
Torque Compensation (lbf)	: 38.1
Preload (lbf)	: 66.1
Test Conditions	: 23°C ± 2°C / 50% ± 10% RH

Sample ID	Test Number	Average Peel Load (lbf)	Maximum Peel Load (lbf)	Minimum Peel Load (lbf)	Specimen Width (in)	**Average Peel Torque (in-lbf/in) of specimen width**	Failure Type
P1404.CDP.001.FEN	1	70.8	86.2	62.0	3.0	5.34	1
P1404.CDP.002.FEN	2	68.1	86.3	61.2	3.0	4.90	1
P1404.CDP.003.FEN	3	82.6	117	57.8	3.0	4.00	1,4
P1404.CDP.004.FEN	4	73.5	90.1	61.0	3.0	5.78	1,4
P1404.CDP.005.FEN	5	80.3	105	73.7	3.0	6.89	1,4
P1404.CDP.006.FEN	6	86.3	92.2	57.4	3.0	4.61	1
P1404.CDP.007.FEN	7	82.0	126	74.2	3.0	7.17	4
P1404.CDP.008.FEN	8	59.0	70.5	54.7	3.0	3.41	1
P1404.CDP.009.FEN	9	72.4	85.0	62.0	3.0	5.60	1
				Average		**5.30**	
				Std. Dev		1.24	

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Intertek

Testing	**Climbing Drum Peel for Adhesives**
Test Method	ASTM D1781 - 98 (Reapproved 2012) - Modified specimen length, peel distance and number of specimens
Project Number	P23134502
Customer	InfoSciTex Corporation Purchase Order # : 131716
Attention	Carrie-Ann Ferrera
Analyst	D. Widgette / K. Schuman
Date	December 23, 2013

Sample ID	Test Number	Average Peel Load (lbf)	Maximum Peel Load (lbf)	Minimum Peel Load (lbf)	Specimen Width (in)	**Average Peel Torque (in-lbf/in) of specimen width**	Failure Type
P1404.CDP.001.117	1	62.4	102	50.9	3.0	3.97	1,4
P1404.CDP.002.117	2	54.3	86.8	51.5	3.0	2.85	1,4
P1404.CDP.003.117	3	55.0	71.2	51.4	3.0	2.76	1,4
				Average		**3.13**	
				Std. Dev.		0.73	

Failure Types:

1= Cohesive failure within the adhesive
2= Adhesion to the facing
3= Adhesion to the core
4= Failure within the core

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Intertek

Testing	**Climbing Drum Peel for Adhesives**
Test Method	ASTM D1781 - 98 (Reapproved 2012) - Modified specimen length and peel distance
Project Number	P201 34502
Customer	InfoSciTex Corporation Purchase Order # : 131718
Attention	Carrie-Ann Ferreira
Analyst	M. Brady / K. Schuman
Date	January, 28 2014

Adherend Description	Aluminum
Adherend Thickness (in)	0.02
Sample Dimensions (in)	3 x 0.6 x 11 (nominal)
Bonding Conditions	Not Supplied
Sample Preparation	Remedial machining performed by Intertek PTL
Conditioning	**30 days @ 90°F / 95% RH**
Test Machine Type	Instron 5985
Speed Of Testing	1.0 in/min
Flange Radius (in)	2.51
Drum Radius (in)	2.01
Calibration Method	Adherend
Torque Compensation (lbf)	36.7
Preload (lbf)	60.0
Test Conditions	23°C ± 2°C / 50% ± 10% RH

Sample ID	Test Number	Average Peel Load (lbf)	Maximum Peel Load (lbf)	Minimum Peel Load (lbf)	Specimen Width (in)	**Average Peel Torque (in-lbf/in) of specimen width**	Failure Type
P1404.CDP.028.FEN	1	40.2	41.3	38.9	3.0	0.250	2
P1404.CDP.029.FEN	2	41.1	42.9	38.8	3.0	0.390	2
P1404.CDP.030.FEN	3	39.5	40.3	38.9	3.0	0.130	2
P1404.CDP.031.FEN	4	39.5	40.4	36.9	3.0	0.130	2
P1404.CDP.032.FEN	5	37.8	38.0	34.8	3.0	-0.150	2
P1404.CDP.033.FEN	6	did not reach the specified preload in order to obtain data					
P1404.CDP.034.FEN	7	38.6	37.6	35.5	3.0	-0.340	2
P1404.CDP.035.FEN	8	38.3	38.5	38.0	3.0	-0.070	2
P1404.CDP.036.FEN	9	38.2	39.8	37.0	3.0	-0.080	2
				Average		**0.030**	
				Std. Dev.		0.236	

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Intertek

Testing	**Climbing Drum Peel for Adhesives**
Test Method	ASTM D1781 - 98 (Reapproved 2012) Modified specimen length and peel distance
Project Number	P2014-34602
Customer	InfoSciTex Corporation Purchase Order # : 134716
Attention	Carlo-Ann Ferreira
Analyst	D. Midgette / D. Carter / K. Schurman
Date	April 1, 2014

Test ID	**Accelerated Aging**
Adherend Description	Aluminum
Adherend Thickness (in)	0.02
Sample Dimensions (in)	3 x 0.5 x 11 (nominal)
Bonding Conditions	Not Supplied
Sample Preparation	Remedial machining performed by Intertek PTL
Conditioning	**90 days @ 120°F / 60% RH**
Test Machine Type	Instron 5985
Speed Of Testing	10 in/min
Flange Radius (in)	2.51
Drum Radius (in)	2.01
Calibration Method	Adherend
Torque Compensation (in)	34.8
Preload (lbf)	55.0
Test Conditions	23°C ± 2°C / 50% ± 10% RH

Sample ID	Test Number	Average Peel Load (lbf)	Maximum Peel Load (lbf)	Minimum Peel Load (lbf)	Specimen Width (in)	**Average Peel Torque (in-lbf/in) of specimen width**	Failure Type
P1404.CDP.019.FEN	1	51.9	56.6	40.9	3.0	2.79	2
P1404.CDP.020.FEN	2	44.3	48.6	42.7	3.0	1.55	2
P1404.CDP.021.FEN	3	43.3	47.5	40.7	3.0	1.39	2
P1404.CDP.022.FEN	4	52.7	63.1	45.6	3.0	2.92	2
P1404.CDP.023.FEN	5	48.5	51.9	45.4	3.0	2.24	2
P1404.CDP.024.FEN	6	47.3	50.4	45.8	3.0	2.04	2
P1404.CDP.025.FEN	7	57.8	64.4	53.2	3.0	3.76	2
P1404.CDP.026.FEN	8	50.2	58.4	45.2	3.0	2.52	2
P1404.CDP.027.FEN	9	56.7	64.4	51.7	3.0	3.48	2
				Average		**2.52**	
				Std. Dev		0.97	

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Intertek

Peel Resistance Report Page 2 of 2

Testing	: **Climbing Drum Peel for Adhesives**
Test Method	: ASTM D1781 - 98 (Reapproved 2012) Modified specimen length, peel distance and number of specimens
Project Number	: P2013450-2
Customer	: InfoSciTex Corporation Purchase Order # : 131716
Attention	: Carrie-Ann Ferreira
Analyst	: M. Brady / K. Schuman
Date	: January, 29 2014

Sample ID	Test Number	Average Peel Load (lbf)	Maximum Peel Load (lbf)	Minimum Peel Load (lbf)	Specimen Width (in)	**Average Peel Torque (in-lbf/in) of specimen width**	Failure Type
P1404.CDP.010.117	1	45.7	48.9	42.8	3.0	1.14	1,2
P1404.CDP.011.117	2	47.1	52.3	43.2	3.0	1.37	1,2
P1404.CDP.012.117	3	45.9	51.4	39.8	3.0	1.18	1,2
				Average		1.23	
				Std. Dev.		0.12	

Failure Types:
1= Cohesive failure within the adhesive
2= Adhesion to the facing
3= Adhesion to the core
4= Failure within the core

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Intertek

Testing : **Climbing Drum Peel for Adhesives**
Test Method : ASTM D1781 - 98 (Reapproved 2012) - Modified specimen length, peel distance and number of specimens
Project Number : P2013-4503
Customer : InfoSciTex Corporation Purchase Order # : 131713
Attention : Carrie Ann Ferreira
Analyst : D. Mogotto / S. Carter / K. Schuman
Date : April 1, 2014

Sample ID	Test Number	Average Peel Load (lbf)	Maximum Peel Load (lbf)	Minimum Peel Load (lbf)	Specimen Width (in)	**Average Peel Torque (in-lbf/in) of specimen width**	Failure type
P1404.CDP.007.117	1	74.0	90.5	64.8	3.0	3.40	4
P1404.CDP.008.117	2	62.6	56.8	48.2	3.0	2.89	2
P1404.CDP.009.117	3	84.1	73.2	54.5	3.0	4.79	2, 4
				Average		**4.69**	

Failure Types
1= Cohesive failure within the adhesive
2= Adhesion to the facing
3= Adhesion to the core
4= Failure within the core

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Appendix 4D: Core Shear Properties of Sandwich Constructions by Beam Flexure

Intertek

Testing	:	Core Shear Properties of Sandwich Constructions by Beam Flexure
Test Method	:	ASTM C393/C393M - 11[e1]
Project Number	:	P20134502 Purchase Order # : 131716
Customer	:	InfoSciTex Corporation
Attention	:	Carrie-Ann Ferreira
Analyst	:	M. Bracy / C. Sima
Date	:	December 12, 2013
Material	:	Baseline
Sample Preparation	:	Tested as received
Facing Thickness (in, nominal)	:	0.063
Instron Model Number	:	4201 Last Calibration Date : January 2013
Measurement Equipment	:	PTL 020 Last Calibration Date : January 2013
Sampling Rate (cals points/s)	:	10
Core Conditioning	:	Unconditioned
Test Conditions	:	23°C ± 2°C / 50% ± 10% RH
Moisture Content	:	Unknown
Configuration	:	3-point
Span Length (in)	:	6
Supports	:	1 in x 5 in steel bars on cylindrical pivot, 1 in x 4 in 60 Shore A rubber pressure pads centered on supports
Loading Nose	:	1 in x 4 in steel bars on cylindrical pivots with 50 Shore A rubber pressure pads
Cross-Head Speed (in/min)	:	0.2

Specimen label	Average Sandwich Width (in)	Average Sandwich Thickness (in)	Average Sandwich Length (in)	Maximum Load (lbf)	Maximum Core Shear Strength (PSI)	Maximum Facing Stress (PSI)	Failure Mode
P1404.3PF.001 FEN	3.05	0.644	8.08	788	222	10600	CUU
P1404.3PF.002 FEN	3.03	0.636	8.08	778	224	10700	CUU
P1404.3PF.003 FEN	3.03	0.641	8.08	793	226	10800	CUU
P1404.3PF.004 FEN	3.03	0.639	8.08	781	224	10700	CUU
P1404.3PF.005 FEN	3.03	0.636	8.07	777	224	10700	CUU
P1404.3PF.006 FEN	3.04	0.644	8.06	783	222	10500	CUU
P1404.3PF.007 FEN	3.02	0.641	8.07	753	219	10400	CUU
P1404.3PF.008 FEN	3.03	0.642	8.07	773	220	10500	CUU
P1404.3PF.009 FEN	3.02	0.628	8.07	781	229	10900	CUU
			Average	780	223	10700	
			Std. Dev.	8	2	151	
			C.O.V. (%)	1	1	1	

First Character		Second Character		Third Character	
Failure Type	Code	Failure Area	Code	Failure Location	Code
Core Crushing	C	At Load Bar	A	Core	C
Skin to Core Delamination	D	Gage	G	Core-facing Bond	A
Facing Failure	F	Multiple Areas	M	Bottom Facing	B
Multi-mode	M (type)	Outside Gage	O	Top Facing	T
Transverse Shear	S	Various	V	Both Facings	F
Explosive	X	Unknown	U	Various	V
Other	O			Unknown	U

Specimens did not show any apparent failures regardless of the extension at which the test was ended at

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Intertek

Testing	:	Core Shear Properties of Sandwich Constructions by Beam Flexure	
Test Method	:	ASTM C393/C393M - 11[illegible]	
Project Number	:	P20134502	Purchase Order # : 131716
Customer	:	InfoSciTex Corporation	
Attention	:	Carre-Ann Ferreira	
Analyst	:	M. Brady / G. Sirie	Attachments : 2 graphs
Date	:	December 12, 2013	
Material	:	Baseline	
Sample Preparation	:	Tested as received	
Facing Thickness (in, nominal)	:	0.063	
Instron Model Number	:	4204	Last Calibration Date : January 2013
Measurement Equipment	:	IPTL 020	Last Calibration Date : January 2013
Sampling Rate (data points/s)	:	10	
Conditioning	:	Unconditioned	
Test Conditions	:	23°C ± 2°C / 50% ± 10% RH	
Moisture Content	:	Unknown	
Configuration	:	3-point	
Span Length (in)	:	6	
Supports	:	1 in x 5 in steel bars on cylindrical pivot 1 in x 4 in 60 Shore A rubber pressure pads centered on supports	
Loading Nose	:	1 in x 4 in steel bars on cylindrical pivots with 60 Shore A rubber pressure pads	
Cross-Head Speed (in/min)	:	0.12	

Specimen Label	Average Sandwich Width (in)	Average Sandwich Thickness (in)	Average Sandwich Length (in)	Maximum Load (lbf)	Maximum Core Shear Strength (PSI)	Maximum Facing Stress (PSI)	Failure Mode
P1424.3PF.001.117	3.03	0.832	6.04	762	223	10600	OUU
P1424.3PF.002.117	3.03	0.843	6.06	609	174	9530	M(S)(M)V
P1424.3PF.004.117	3.04	0.831	6.06	767	222	10600	OUU
P1424.3PF.005.117	3.02	0.838	6.07	718	207	9830	OUU
P1424.3PF.006.117	3.04	0.832	6.06	738	213	10100	OUU
P1424.3PF.007.117	3.05	0.841	6.11	742	211	10000	OUU
P1424.3PF.008.117	3.03	0.836	6.05	735	212	10100	OUU
P1424.3PF.009.117	3.04	0.836	6.06	751	213	10300	OUU
			Average	727	209	9960	
			Std. Dev.	51	15	721	
			C.O.V. (%)	7	7	7	

First Character		Second Character		Third Character	
Failure Type	Code	Failure Area	Code	Failure Location	Code
Core Crushing	C	At Load Bar	A	Core	C
Skin to Core Delamination	D	Gage	G	Core-facing Bond	A
Facing Failure	F	Middle Areas	M	Bottom Facing	B
Multimode	M (sys)	Outside Gage	O	Top Facing	T
Transverse Shear	S	Various	V	Both Facings	F
Explosive	X	Unknown	U	Various	V
Other	O			Unknown	U

Note: Specimen P1424.3PF.003.117 was not included in results due to considerable face delamination prior to receiving the specimen

Besides .002.117, specimens did not show any apparent failures regardless of the extension at which the test was ended at.

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Intertek

Testing	:	Core Shear Properties of Sandwich Constructions by Beam Flexure
Test Method	:	ASTM C393/C393M - 11[illegible]
Project Number	:	P201[illegible]402 Purchase Order # : 131[illegible]18
Customer	:	InfoSc Tex Corporation
Attention	:	Carrie-Ann Ferrera
Analyst	:	J. Storie / G. Sims
Date	:	January 29, 2014

Sample Preparation	:	Tested as received
Facing Thickness (in. nominal)	:	0.063
Instron Model Number	:	4204 Last Calibration Date : January 2014
Measurement Equipment	:	IPTL [illegible] and IPTL 484 Last Calibration Date : January 2014
Sampling Rate (data points/s)	:	10
Conditioning	:	30 Days at 90°F / 95% RH
Test Conditions	:	23°C ± 2°C / 50% ± 10% RH
Moisture Content	:	Unknown
Configuration	:	3 point
Span Length (in)	:	6
Supports	:	1 in x 1 in steel bars on cylindrical pivot, 1 in x 4 in 60 Shore A rubber pressure pads centered on supports
Loading Nose	:	1 in x 4 in steel bars on cylindrical pivots with 60 Shore A rubber pressure pads
Cross-Head Speed (in/min)	:	0.[illegible]2

Specimen Label	Average Sandwich Width (in)	Average Sandwich Thickness (in)	Average Sandwich Length (in)	Maximum Load (lbf)	Maximum Core Shear Strength (PSI)	Maximum Facing Stress (PSI)	Failure Mode
P1404.3PF.019.FEN	3.02	0.647	8.03	546	140	6850	DOT
P1404.3PF.020.FEN	3.03	0.653	8.08	621	157	7470	M(DS)OB
P1404.3PF.021.FEN	3.04	0.641	8.05	531	136	6490	DOB
P1404.3PF.022.FEN	3.01	0.639	8.09	711	177	8410	DOT
P1404.3PF.023.FEN	3.03	0.638	8.05	670	173	8250	DOT
P1404.3PF.024.FEN	3.03	0.644	8.04	654	168	7880	DOT
P1404.3PF.025.FEN	3.05	0.642	8.05	730	186	8860	DOT
P1404.3PF.026.FEN	3.03	0.645	8.04	729	187	8860	DOT
P1404.3PF.027.FEN	3.02	0.651	8.07	656	177	8450	DOT
			Average	654	167	7940	
			Std. Dev.	75	18	888	
			C.O.V. (%)	1[illegible]	1[illegible]	11	

First Character		Second Character		Third Character	
Failure Type	Code	Failure Area	Code	Failure Location	Code
Core Crushing	C	All Load Bar	A	Core	C
Skin to Core Delamination	D	Gage	G	Core-facing Bond	A
Facing Failure	F	Multiple Areas	M	Bottom Facing	B
Multi mode	M (sys)	Outside Gage	O	Top Facing	T
Transverse Shear	S	Various	V	Both Facings	F
Explosive	X	Unknown	L	Various	V
Other	O			Unknown	U

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Flexural Report Page 1 of 4

Testing	Core Shear Properties of Sandwich Constructions by Beam Flexure
Test Method	ASTM C393/C393M - 11
Project Number	P2013 4502 Purchase Order # : 131716
Customer	InfoSciTex Corporation
Attention	Carrie-Ann Ferreira
Analyst	J. Stone / G. Stone Attachments : 2 graphs
Date	January 29, 2014

Sample Preparation	Tested as received	
Facing Thickness (in, nominal)	0.030	
Instron Model Number	4204	Last Calibration Date : January 2014
Measurement Equipment	IPTL 020 and IPTL 484	Last Calibration Date : January 2014
Sampling Rate (data points/s)	10	
Conditioning	30 Days at 90°F / 95% RH	
Test Conditions	23°C ± 2°C / 50% ± 10% RH	
Moisture Content	Unknown	
Configuration	3-point	
Span Length (in)	5	
Supports	1 in x 6 in steel bars on cylindrical pivot, 1 in x 4 in 60 Shore A rubber pressure pads centered on supports	
Loading Nose	1 in x 4 in steel bars on cylindrical pivots with 60 Shore A rubber pressure pads	
Cross-Head Speed (in/min)	0.12	

Specimen Lase	Average Sandwich Width (in)	Average Sandwich Thickness (in)	Average Sandwich Length (in)	Maximum Load (lbf)	Maximum Core Shear Strength (PSI)	Maximum Facing Stress (PSI)	Failure Mode
P1404.3PF.019.117	3.04	0.640	8.07	432	123	5850	DOT
P1404.3PF.020.117	3.05	0.646	8.08	747	210	10300	DOL
P1404.3PF.021.117	3.04	0.656	8.06	737	221	9670	DOT
P1404.3PF.022.117	3.04	0.649	8.08	737	217	9850	DOL
P1404.3PF.023.117	3.05	0.640	8.07	746	212	10100	DOT
P1404.3PF.024.117	3.04	0.651	8.06	741	224	9700	DOT
P1404.3PF.025.117	3.04	0.646	8.11	719	223	9680	DOT
P1404.3PF.026.117	3.02	0.645	8.05	714	223	9670	DOL
P1404.3PF.027.117	3.04	0.645	8.05	717	223	9650	DOT
			Average	698	196	9340	
			Std. Dev	120	26	1320	
			C.O.V. (%)	14	14	14	

First Character		Second Character		Third Character	
Failure Type	Code	Failure Area	Code	Failure Location	Code
Core Crushing	C	At Load Bar	A	Core	C
Skin to Core Delamination	D	Gage	G	Core-facing Bond	A
Facing Failure	F	Multiple Areas	M	Bottom Facing	B
Multi-mode	M (xyz)	Outside Gage	O	Top Facing	T
Transverse Shear	S	Various	V	Both Facings	F
Explosive	X	Unknown	U	Various	V
Other	O			Unknown	U

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Appendix 4E: Flatwise Tensile Strength of Sandwich Constructions

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Flatwise Tensile Report Page 1 of 2

Testing	:	**Flatwise Tensile Strength of Sandwich Constructions**
Test Method	:	ASTM C 297-04 (Reapproved 2010)
Project Number	:	P22134502
Customer	:	InfoSciTex Corporation
Attention	:	Carne-Ann Ferreira
Analyst	:	D. Midgette / M. Brady / G. Sirre — Attachments: 3 Graphs
Date	:	December 30, 2013

Ply Orientation / Stacking Sequence	:	N/A
Sample Preparation	:	Machined and bonded by Intertek PTL
Sample Dimensions	:	1.0" x 1.0" x Thickness (nominal)
Loading Block Material	:	Aluminum
Loading Block Dimensions	:	1.0" x 1.0" x 2.0" (nominal)
Instron Model Number	:	4204 — Calibration Date : Jan-2013
Measurement Equipment	:	Intertek PTL part number 500 — Calibration Date : Aug-2013
Alignment Results	:	Self-aligning grips used.
Cross-Head Speed	:	0.02"/min
Sampling Rate (data points/sec)	:	20
Conditioning	:	Unconditioned

Test Conditions 23°C Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.001.FEN	0.994	1.003	0.561	314	315	Cohesive 100%
P1404.FWT.002.FEN	0.995	0.959	0.573	340	342	Core/Cohesive 20%/80%
P1404.FWT.003.FEN	0.990	1.001	0.555	180	182	Core/Cohesive 50%/50%
P1404.FWT.004.FEN	1.002	1.001	0.568	250	248	Adhesive 100%
P1404.FWT.005.FEN	1.003	1.002	0.561	240	238	Adhesive 100%
P1404.FWT.006.FEN	0.992	1.001	0.565	241	243	Adhesive 100%
P1404.FWT.007.FEN	1.000	0.999	0.582	322	322	Adhesive 100%
P1404.FWT.008.FEN	1.001	0.987	0.556	235	233	Core/Cohesive 60%/40%
P1404.FWT.009.FEN	1.001	1.002	0.571	202	201	Adhesive 100%
			Average	258	258	
			Std Dev	55	56	
			C.O.V. (%)	21	22	

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Testing	:	Flatwise Tensile Strength of Sandwich Constructions
Test Method	:	ASTM C 297-04 (Reapproved 2010)
Project Number	:	P201 34502
Customer	:	InfoScitex Corporation
Attention	:	Carrie Ann Ferreira
Analyst	:	D. Midgette / M. Brady / G. Sime — Attachments: 3 Graphs
Date	:	December 30, 2013

Ply Orientation / Stacking Sequence	:	N/A
Sample Preparation	:	Machined and bonded by Intertek PTL
Sample Dimensions	:	1.0" x 1.0" x Thickness (nominal)
Loading Block Material	:	Aluminum
Loading Block Dimensions	:	1.0" x 1.0" x 2.0" (nominal)
Instron Model Number	:	4204 — Calibration Date : Jan-2013
Measurement Equipment	:	Intertek PTL part number 500 — Calibration Date : Aug-2013
Alignment Results		Self-aligning grips used
Cross-Head Speed		0.02"/min
Sampling Rate (data points/s)		20
Conditioning		Unconditioned

Test Conditions 23°C Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.001.117	0.994	1.003	0.576	322	333	Core/Cohesive 50%/50%
P1404.FWT.002.117	0.998	1.000	0.562	277	278	Core 100%
P1404.FWT.003.117	0.995	1.000	0.561	206	208	Core 100%
P1404.FWT.004.117	1.004	1.000	0.570	255	264	Core/Cohesive 30%/70%
P1404.FWT.005.117	1.003	1.000	0.560	160	160	Cohesive 100%
P1404.FWT.006.117	1.003	1.001	0.557	234	>233	Bonding to facing *
P1404.FWT.007.117	1.003	1.002	0.558	409	407	Core/Cohesive 50%/50%
P1404.FWT.008.117	0.996	0.995	0.552	381	383	Core/Cohesive 75%/25%
P1404.FWT.009.117	0.999	1.000	0.555	201	201	Adhesive 100%
			Average	279	279	
			Std Dev	88	88	
			C.O.V. (%)	32	32	

* Bond failure of black to facing excluded from statistics

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Testing	:	**Flatwise Tensile Strength of Sandwich Constructions**
Test Method	:	ASTM C 297-04 (Reapproved 2010)
Project Number	:	P20134602
Customer	:	InfoSciTex Corporation
Attention	:	Carrie-Ann Ferreira
Analyst	:	D. Wiegele / M. Bracy / G. Sirne
Date	:	December 30, 2013

Test Conditions -67°F (-55°C) Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.010.FEN	0.992	0.994	0.558	157	>159	Bond to Facing *
P1404.FWT.011.FEN	0.998	1.021	0.564	274	>274	Bond to Facing *
P1404.FWT.012.FEN	1.000	1.021	0.573	291	291	Core/Cohesive 20%/80%
P1404.FWT.013.FEN	1.000	1.021	0.574	309	309	Core/Cohesive 30%/70%
P1404.FWT.014.FEN	0.999	0.989	0.562	345	345	Core/Cohesive 50%/70%
P1404.FWT.015.FEN	1.000	1.021	0.572	298	298	Core/Cohesive 60%/40%
P1404.FWT.016.FEN	0.998	1.021	0.565	231	>231	Bond to Facing *
P1404.FWT.017.FEN	0.992	1.021	0.577	233	>235	Bond to Facing *
P1404.FWT.018.FEN	1.000	1.020	0.568	325	325	Core/Cohesive 40%/60%
			Average	314	314	
			Std Dev	22	22	
			C.O.V. (%)	7	7	

Test Conditions 180°F (82°C) Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.019.FEN	1.000	0.989	0.563	103	>103	Bond to Facing *
P1404.FWT.020.FEN	1.008	1.017	0.577	185	>180	Bond to Facing *
P1404.FWT.021.FEN	1.002	1.002	0.564	139	>138	Bond to Facing *
P1404.FWT.022.FEN	1.001	0.986	0.563	77.0	>77.0	Bond to Facing *
P1404.FWT.023.FEN	1.002	1.002	0.575	115	>115	Bond to Facing *
P1404.FWT.024.FEN	0.994	1.001	0.561	120	120	Adhesive 100%
P1404.FWT.025.FEN	1.013	0.997	0.573	87.5	>87.0	Bond to Facing *
P1404.FWT.026.FEN	1.008	1.017	0.567	130	>127	Bond to Facing *
P1404.FWT.027.FEN	0.999	1.010	0.558	70.3	>70.0	Bond to Facing *
			Average	---	---	
			Std Dev	---	---	
			C.O.V. (%)	---	---	

* Bond failure of block to facing excluded from statistics

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Testing	:	Flatwise Tensile Strength of Sandwich Constructions
Test Method	:	ASTM C 297-04 (Reapproved 2010)
Project Number	:	P20134502
Customer	:	InfoSciTex Corporation
Attention	:	Carrie-Ann Ferreira
Analyst	:	D. Miugette / M. Brady / G. Sims
Date	:	December 30, 2013

Test Conditions -67°F (-55°C) Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.010.117	1.001	0.998	0.549	238	238	Core/Cohesive 70%/30%
P1404.FWT.011.117	0.996	0.998	0.557	412	412	Core/Cohesive 70%/30%
P1404.FWT.012.117	1.003	1.001	0.556	101	>101	Bond to Facing *
P1404.FWT.013.117	0.995	1.000	0.553	188	>188	Bond to Facing *
P1404.FWT.014.117	0.980	0.998	0.563	91.5	93.0	Core/Cohesive 40%/60%
P1404.FWT.015.117	1.000	1.000	0.556	156	156	Core/Cohesive 50%/50%
P1404.FWT.016.117	0.996	0.998	0.560	53.4	54.0	Core/Cohesive 20%/80%
P1404.FWT.017.117	0.996	0.998	0.562	217	>218	Bond to Facing *
P1404.FWT.018.117	0.993	1.002	0.560	200	>201	Bond to Facing *
			Average	190	191	
			Std Dev	142	142	
			C.O.V. (%)	75	74	

Test Conditions 180°F (82°C) Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.019.117	1.005	1.003	0.553	210	208	Core/Cohesive 50%/50%
P1404.FWT.020.117	1.002	0.997	0.559	101	>102	Bond to Facing *
P1404.FWT.021.117	1.000	1.003	0.556	106	106	Core/Cohesive 20%/80%
P1404.FWT.022.117	1.001	1.003	0.557	73.8	>74.0	Bond to Facing *
P1404.FWT.023.117	1.001	1.003	0.563	158	>157	Bond to Facing *
P1404.FWT.024.117	1.001	1.009	0.555	244	242	Core/Cohesive 50%/50%
P1404.FWT.025.117	1.004	0.999	0.565	191	190	Core/Cohesive 60%/40%
P1404.FWT.026.117	1.004	1.000	0.563	130	>129	Bond to Facing *
P1404.FWT.027.117	1.002	1.004	0.556	126	>125	Bond to Facing *
			Average	188	187	
			Std Dev	59	58	
			C.O.V. (%)	31	31	

* Bond failure of block to facing excluded from statistics

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Flatwise Tensile Report Page 1 of 1

Testing	: Flatwise Tensile Strength of Sandwich Constructions	
Test Method	: ASTM C 297-04 (Reapproved 2010) - Modified time to failure	
Project Number	: P2013 4502	
Customer	: InfoSciTex Corporation	
Attention	: Carrie Ann Ferreira	
Analyst	: Frank Foy / G. Sims	Attachment: 1 Graph
Date	: January 29, 2014	

ACCREDITED Cert. No. 0512.01 TESTING LABORATORY

Ply Orientation / Stacking Sequence	: N/A	
Sample Preparation	: Machined and bonded by Intertek PTL	
Sample Dimensions	: 1.0" x 1.0" x Thickness (nominal)	
Loading Block Material	: Aluminum	
Loading Block Dimensions	: 1.0" x 1.0" x 2.0" (nominal)	
Instron Model Number	: 4204	Calibration Date : Jan-2014
Measurement Equipment	: Intertek PTL part number 493	Calibration Date : Jan 2014
Alignment Results	: Self-aligning grips used.	
Cross Head Speed	: 0.02"/min	
Sampling Rate (data points/s)	: 20	
Conditioning	: 30 days at 90°F, 95% RH	

Test Conditions 23°C Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.046.FEN	0.987	0.980	0.557	9.21	9.43	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.047.FEN	0.991	0.986	0.947	5.96	6.10	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.048.FEN	0.992	0.992	0.556	17.1	17.4	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.049.FEN	0.985	0.996	0.556	23.5	23.7	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.050.FEN	0.992	0.996	0.963	25.6	25.8	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.051.FEN	0.989	0.984	0.553	13.7	14.1	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.052.FEN	0.986	0.991	0.557	14.0	14.3	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.053.FEN	0.973	0.983	0.555	14.1	14.6	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.054.FEN	0.985	0.992	0.558	5.42	5.55	Adhesive failure of core-facing adhesive / 100%
			Average	14.3	14.6	
			Std Dev	7.03	7.1	
			C.O.V. (%)	49	49	

Per ASTM C297, specimens are to be tested at a speed to produce a failure within 3 to 6 minutes. All specimens failed in less than 3 minutes.

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Testing	: Flatwise Tensile Strength of Sandwich Constructions
Test Method	: ASTM C 297-04 (Reapproved 2010) - Modified time to failure
Project Number	: P20134b02
Customer	: InfoSciTex Corporation
Attention	: Carrie-Ann Ferreira
Analyst	: Frank Foy / C. Sime Attachment: 1 Graph
Date	: January 29, 2014

ACCREDITED Cert. No. 0819.01 TESTING LABORATORY

Ply Orientation / Stacking Sequence	: N/A
Sample Preparation	: Machined and bonded by Intertek PTL
Sample Dimensions	: 1.0" x 1.0" x Thickness (nominal)
Loading Block Material	: Aluminum
Loading Block Dimensions	: 1.0" x 1.0" x 2.0" (nominal)
Instron Model Number	: 4204 Calibration Date : Jan 2014
Measurement Equipment	: Intertek PTL asset number 450 Calibration Date : Jan 2014
Alignment Results	: Self-aligning grips used.
Cross-Head Speed	: 0.02"/min
Sampling Rate (data points/s)	: 20
Conditioning	: 30 days at 90°F, 95% RH

Test Conditions 23°C Sample Name	Sample Length (in)	Sample Width (in)	Sample Height (in)	Maximum Load (lbs)	Flatwise Tensile Strength (PSI)	Failure Type / Failure Area (%)
P1404.FWT.046.117	0.995	0.994	0.568	4.02	4.10	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.047.117	0.996	0.997	0.564	7.19	7.24	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.048.117		Failed on removal from chamber				Adhesive failure of core-facing adhesive / 100%
P1404.FWT.049.117	0.995	0.996	0.564	12.2	12.3	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.050.117	0.999	0.998	0.568	9.98	10.0	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.051.117		Failed on removal from chamber				Adhesive failure of core-facing adhesive / 100%
P1404.FWT.052.117	0.995	0.996	0.534	7.55	7.72	Adhesive failure of core-facing adhesive / 100%
P1404.FWT.053.117		Failed on removal from chamber				Adhesive failure of core-facing adhesive / 100%
P1404.FWT.054.117	0.995	0.997	0.555	6.36	6.40	Adhesive failure of core-facing adhesive / 100%
			Average	7.88	7.96	
			Std Dev	2.84	2.86	
			C.O.V. (%)	35	35	

Per ASTM C297, specimens are to be tested at a speed to produce a failure within 3 to 6 minutes. All specimens failed in less than 3 minutes.

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Appendix 5: List of Scientific/Technical Publications

1. C.-A.M. Ferreira, M.M. Reed, J.W. Belcher, M. Cushman (2012, March). Shelf-Stable Epoxy Resin Adhesive. Paper presented at SAMPE Tech 2012, North Charleston, SC.