ALBERTA INNOVATES

CLEAN RESOURCES FINAL REPORT PACKAGE

Project proponents are required to submit a Final Report Package, consisting of a Final Public Report and a Final Financial Report. These reports are to be provided under separate cover at the conclusion of projects for review and approval by Alberta Innovates (AI) Clean Resources Division. Proponents will use the two templates that follow to report key results and outcomes achieved during the project and financial details. The information requested in the templates should be considered the minimum necessary to meet AI reporting requirements; proponents are highly encouraged to include other information that may provide additional value, including more detailed appendices. Proponents must work with the AI Project Advisor during preparation of the Final Report Package to ensure submissions are of the highest possible quality and thus reduce the time and effort necessary to address issues that may emerge through the review and approval process.

Final Public Report

The Final Public Report shall outline what the project achieved and provide conclusions and recommendations for further research inquiry or technology development, together with an overview of the performance of the project in terms of process, output, outcomes and impact measures. The report must delineate all project knowledge and/or technology developed and must be in sufficient detail to permit readers to use or adapt the results for research and analysis purposes and to understand how conclusions were arrived at. It is incumbent upon the proponent to ensure that the Final Public Report **is free of any confidential information or intellectual property requiring protection**. The Final Public Report will be released by Alberta Innovates after the confidentiality period has expired as described in the Investment Agreement.

Final Financial Report

The Final Financial Report shall provide complete and accurate accounting of all project expenditures and contributions over the life of the project pertaining to Alberta Innovates, the proponent, and any project partners. The Final Financial Report will not be publicly released.

ALBERTA INNOVATES

CLEAN RESOURCES FINAL PUBLIC REPORT TEMPLATE

1. PROJECT INFORMATION:

Project Title:	Development of an Asphaltene Mesophase Pre-cursor for Future Production of Carbon Fibre
Alberta Innovates Project Number:	G2020000347
Submission Date:	February 26, 2021
Total Project Cost:	$44,240
Alberta Innovates Funding:	$44,240
AI Project Advisor:	Shunlan Liu

2. APPLICANT INFORMATION:

Applicant (Organization):	University of Alberta
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Applicant Representative Name:	Kevin Hodder
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A. EXECUTIVE SUMMARY

Provide a high-level description of the project, including the objective, key results, learnings, outcomes and benefits.

RESPOND BELOW

A sample of asphaltenes of unknown origin was subjected to pyrolysis at various temperatures and pressures, along with the additions of solvents and additives to induce mesophase formation. Microscopy was used to measure the mesophase yield optically, while the softening point was measured via differential scanning calorimetry. It was found that lower pressure reactions provided a mesophase yield of up to 48 ± 4.3%. It is suggested that the addition of tetralin, feedstock of C5 and lower initial pressure led to the large increase in mesophase formation. The previous variables were found via an optimization design of experiments, where thirty experimental runs helped us determine the parameters with the largest effects on mesophase formation. The knowledge created includes three methodologies: 1) filtering of C5 asphaltenes from raw feedstock; 2) mesophase pre-cursor formation via pyrolysis; and 3) an indirect method of calculating the softening point. The glass transition temperature is a critical variable that determines the feasibility of melt spinning into CF.

B. INTRODUCTION

Please provide a narrative introducing the project using the following sub-headings.

- **Sector introduction:** Include a high-level discussion of the sector or area that the project contributes to and provide any relevant background information or context for the project.
- **Knowledge or Technology Gaps:** Explain the knowledge or technology gap that is being addressed along with the context and scope of the technical problem.

RESPOND BELOW

SECTOR INTRODUCTION

Carbon fiber (CF) is currently categorized as either a PAN-based (Polyacrylonitrile) or a pitch-based material. For the former, a polymer is chemically-treated and then spun into fibers, followed by the traditional oxidation and carbonization process. Alternatively, different pitch types including petroleum and coal tar are used as feedstock for major manufacturers of CF. Both anisotropic and isotropic pitch (a mesophase precursor) is needed to produce CF. Anisotropic precursors result in higher quality and stronger CF with applications in aerospace and cement fortification, while isotropic pitch is used to manufacture CF's to be used in production of electrodes and insulators (Park, 2018).

Although, pitch-based CF is an established product within the market, the majority of CF produced remains PAN-based (Table 1). Significant literature exists on the preparation and properties of a pitch-based precursor (both petroleum and coal-tar), which could offer valuable insights into developing a viable process for converting asphaltenes to a CF product.

Table 1. 2013 statistics on PAN and pitch-based Carbon fibre, reproduced based on data reported in (Park, 2018)

CF Type	Companies (Worldwide)	Total Estimated Production (unit: ton)
PAN-based	Toray, Toho Tenax, Mitsubishi Rayon, Formosa, Hexcel, SGL, Aksa, Cytec, Hyosung, Taekwang	82950
Pitch-based	Mitsubishi, Nippon Graphite, Cytec	201
Pitch-based	Petoca Materials, Kureha Chemicals, Donac	441

KNOWLEDGE OR TECHNOLOGY GAPS:

There is currently no known method to produce the mesophase precursor required for proper CF production from asphaltenes. Spinning asphaltenes that have not been treated to include a mesophase component will result in poor mechanical properties. We have explored several strategies to convert asphaltenes into a precursor for CF production with a high mesophase content. Asphaltenes are mainly treated as a waste stream, thus, there is a considerable lack of knowledge available that would allow the conversion of asphaltenes into a value-added product such as CF. Below are some key knowledge gaps identified by our research team:

A standardized method that produces a consistent feedstock for pyrolysis.

The feedstock should have an acceptable tolerance/variance in its molecular make up and aggregation behavior. If the feedstock molecular makeup varies, then consistent CF production with homogenous mechanical properties is not possible.

The physical and chemical parameter relationships that affect the formation of a mesophase precursor are unknown.

Such parameters include, but are not limited to:

- Feedstock aromaticity and alkyl side chains.
- Process variables such as temperature, pressure, purging gas, hold time, de-pressurization, mixing rate, volatiles, solvents, and other additives.
- Uncatalyzed treatment options such as co-pyrolysis with liquid solvents and/or a solid dilutant.

- Softening point (SP) is a measurement that is frequently employed as an indicator for the suitability of a CF precursor for spinning (spinnability). However, this does not fill the void of rheological data of the feedstock as it undergoes pyrolysis.
- Role of heteroatoms in the mesophase formation and their potential removal prior to oxidization and carbonization.

Lack of existing research infrastructure in Alberta.

The Carbon Fibre Grand Challenge: Phase I is a vital first step towards creating an eco-system that forges/fosters intergroup collaboration. Also, the equipment needed to perform quality control on feedstock, precursor and spun fibre is currently nonexistent in Alberta labs. The traditional "ball and cup" method apparatus, commonly used for standard determination of softening point, cannot be used in environments above 160 ^{o}C.

C. PROJECT DESCRIPTION

Please provide a narrative describing the project using the following sub-headings.

- **Knowledge or Technology Description:** Include a discussion of the project objectives.
- **Updates to Project Objectives:** Describe any changes that have occurred compared to the original objectives of the project.
- **Performance Metrics:** Discuss the project specific metrics that will be used to measure the success of the project.

RESPOND BELOW

KNOWLEDGE OR TECHNOLOGY DESCRIPTION

A sample of asphaltenes of unknown origin was subjected to thermal processes using a specialized reactor system. The experimental parameters were altered using a Design of Experiments (DOE) methodology based off of the Taguchi method, where two levels for each parameter are modified to reach optimization of a process. This allowed us to determine the parameters that had the largest effect on mesophase yield during pyrolysis, as well as screen parameters that had little effect on the mesophase content. The mesophase content of the samples were measured via optical cross polarized microscopy, while softening points were calculated using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA).

UPDATE TO PROJECT OBJECTIVES

The original objectives were to take the optimized sample with the highest mesophase content and subject it to melt spinning to produce CF. Although a plan for melt spinning was developed, due to delays in equipment along with access restrictions imposed by the University, melt spinning was not achievable within the time frame.

PERFORMANCE METRICS

Table 2 outlines the performance metric that were used to determine the success of the mesophase pre-cursor project.

Table 2: Performance metrics of mesophase pre-cursor project

Performance Metric	Objective	Rationale
Mesophase Content	Maximized	The mesophase content is the main variable measured to determine success of the project. Through varying parameters during pyrolysis
Softening Point	Minimized	The softening point will be measured via DSC and success would be measured through lower values. A lower softening point is ideal for CF production, since if the softening point is too high the mesophase will not be viable for melt spinning.
Elemental Analysis	Tracking removal of heteroatoms	After pyrolysis the samples will be assessed via elemental analysis to check the presence of heteroatoms that are detrimental to CF production.

D. METHODOLOGY

Please provide a narrative describing the methodology and facilities that were used to execute and complete the project. Use subheadings as appropriate.

RESPOND BELOW

MATERIALS AND EQUIPMENT

Sample S2 from the asphaltene sample bank was obtained from Alberta Innovates (Fig. 1). A document containing the elemental composition information as well as other analytical properties was also obtained, but held confidential due to a non-disclosure agreement. There is no known history of the sample regarding thermal or chemical treatment. However, the sample contains a high amount of C5 asphaltene, ash and micro carbon residue, suggesting that the sample was exposed to temperature above 350 ^{o}C.

Figure 1: Sample S2 as received from Alberta Innovates. The sample arrived in a sealed container, which was then opened to reveal the dark, powdery material.

The following chemicals were purchased from Fisher Scientific: toluene, pentane, tetralin, anthracene, pyrene and iso-propanol. Diesel was purchased at a Mobil gas station in Edmonton. N_2 and CO_2 gas cylinders were purchased from Praxair. Pyrex tubes (made by Corning) were sourced from Fisher Scietific (10×75 mm).

A high temperature and pressure testing reactor was purchased from Parr Instruments to perform the pyrolysis experiments for the project (Series 4560 Mini Reactors, 300 mL) with a maximum design temperature of 500 °C and operating pressure of 14 MPa. To accommodate the high operating temperature, graphite gaskets were used to seal the reactor. A controller was used to maintain the heating rate, temperature, and pressure of the reactor. The data was logged on Specview software provided with the reactor. Mass mesaurments were performed using a Torbal AGCN120 scale (120g x 0.0001g w/ Automatic Internal Calibration).

EXTRACTION OF C5 ASPHALTENES

In order to facilitate an increase chance of project success, we decided to extract the C5 asphaltenes from the feedstock as it more prone to mesophase formation. Our procedure for asphaltene washing and extraction was as follows:

- Sample is mixed with toluene at a ratio of 31 mL of solvent for each gram of sample.
- The mixture is then heated to reflux for 3 h while being stirred with a magnetic hot plate. The condenser ensures that material (such as maltenes and asphaltenes) do not evaporate and the

asphaltene dissolved in the toluene returns to the flask. Any solid material is filtered, such as sand or particulates not soluble in toluene (Fig. 2).

- The mixture is filtered through a D5 frit that is subjected to vacuum of -90 kPa and has a pore size 10-20 µm. This last step ensures that any unwanted particulates are captured. This step can be replaced with Soxhlet extraction, where the sample is placed in a glass microfiber Soxhlet extraction thimble and the asphaltene fraction is extracted and collected at the bottom flask.
- Pentane (C5) is added to the residue with the same ratio as the toluene and sonicated for three hours.
- Extra C5 is added to the solution at a ratio of 6 mL solvent for each gram of sample to wash any residue asphaltene that is still stuck to the bottom of the flask. The solution is then sonicated for 30 min followed by filtering through the same frit (Fig. 3).
- The filtered cake is then left to dry under ambient conditions overnight.
- The following day a vacuum of 10^{-6} Torr is placed over the sample to remove any remaining solvent (Fig. 4).

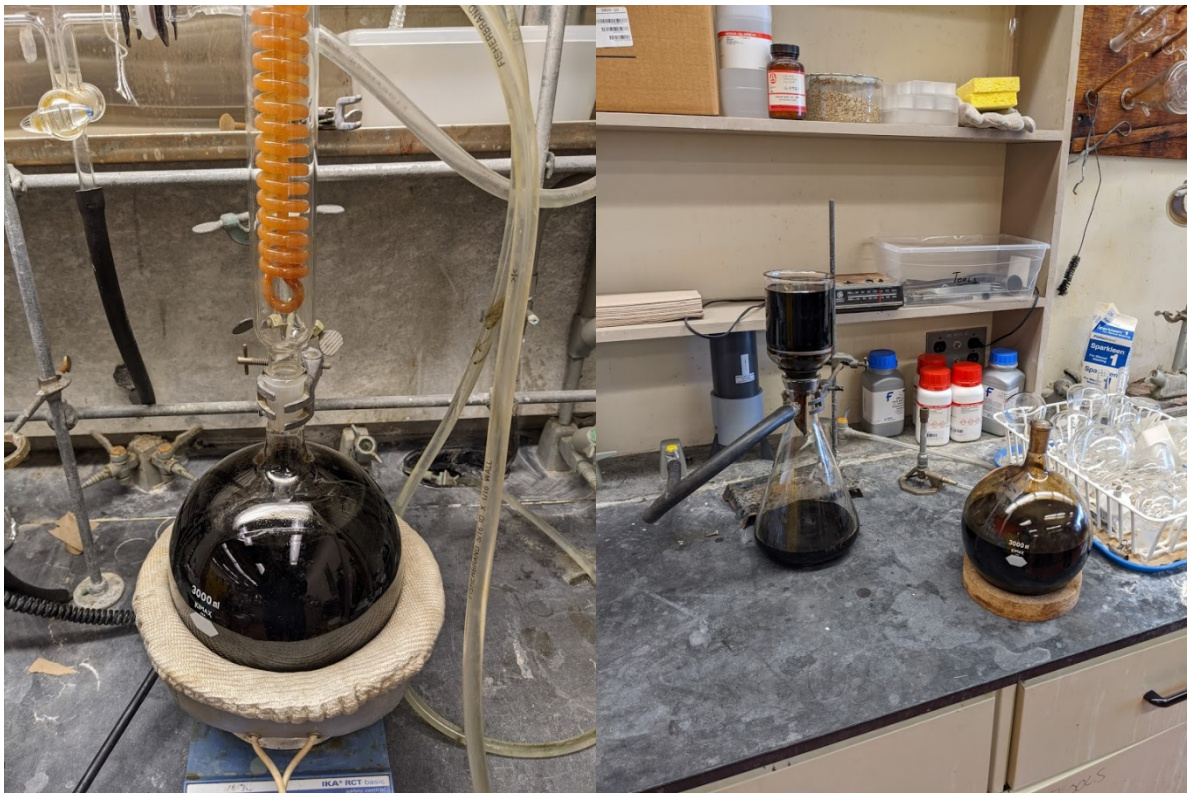


Figure 2: The asphaltene extraction procedure showing the heating and filtering steps, respectively.

Figure 3: Sonication of toluene soluble residue and C5 mixture to precipitate C5 asphaltenes

Figure 4: The dried C5 asphaltene after step six.

PYROLYSIS OF ASPHALTENE SAMPLE

A total of 30 experiments were run using the reactor at six hours per experiment. This required the most significant time commitment for the project at a minimum 180 hours of experimentation. Figure 5 shows the placement of the reactor and the Soxhlet extraction apparatus used to separate the asphaltenes. Figure 6 shows an example of asphaltene placement in the glass vial, along with the result after heat and pressure are applied for several hours. A general description of the steps involved in the pyrolysis test are as follows:

1. The weight of the C5 asphaltene sample (the feedstock set to undergo pyrolysis) is weighed and then placed in Pyrex sample tube.
2. Sample tubes were placed in the sample holder and then in the reactor vessel.
3. Inspect the gasket and replace with a new one if needed (ensure the sealing surfaces are clean).
4. Assemble the reactor (follow the torquing recommendation to ensure proper sealing).
5. Place the reactor in its holder and cover it with the heating jacket.
6. Open the cooling water tap.
7. Purge the assembled system with N_2 at 4 MPa twice (for the CO_2 test, this was done three times at 2.25 MPa).
8. Setup the recorder on the ParrCom software and start logging (15 second intervals).
9. Program the controller for a ramp up of 5 ^{o}C/min to set temperature.
10. Allow pyrolysis to take place at temperature for 1.5 hours.
11. Depressurize the reactor by opening the needle valve in the last half hour of the final residence time to boost mesophase coalescence.
12. Stop the heating and start the cooling process.

Please note that based on the solvent and/or additive involved in the experiment, minor changes were made to the temperature control program. In batch A, we vented the system during heating to keep the reaction pressure close to its initial value. In batch B, we aimed to keep the volatiles in throughout the course of heating and final reaction temperature and as a result, the pressure increased during the course of each test. The temperature profile for each test is included in Appendix I.

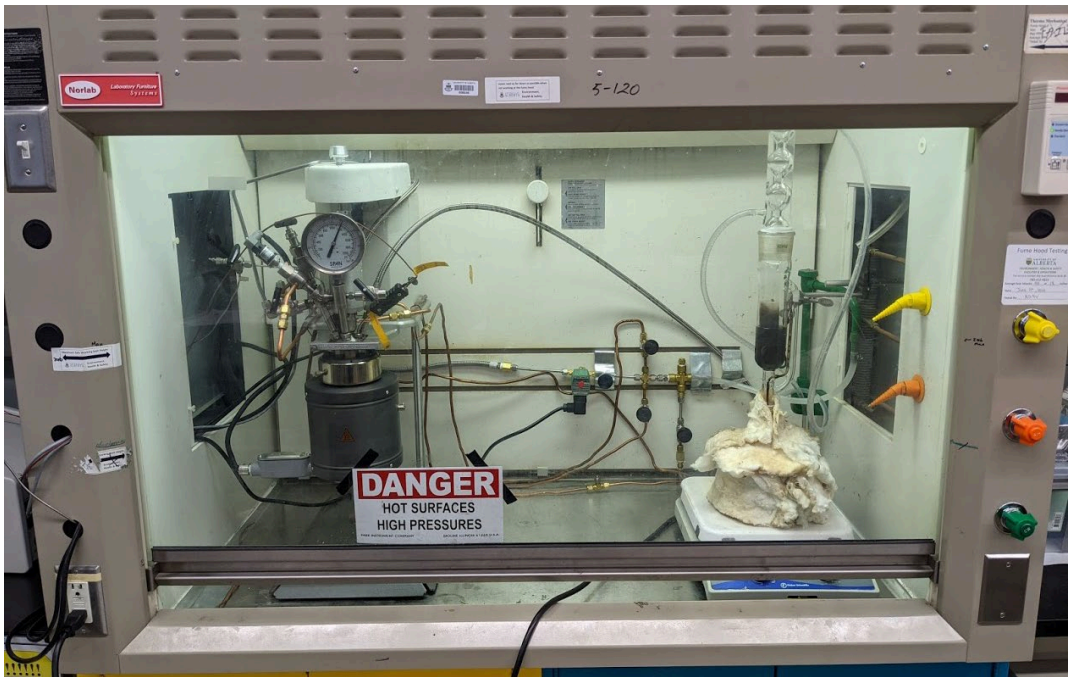


Figure 5: (Right) Parr Instruments reactor installed under fume hood where the pyrolysis took place and. (Left) Recovery of toluene from dissolved asphaltenes.

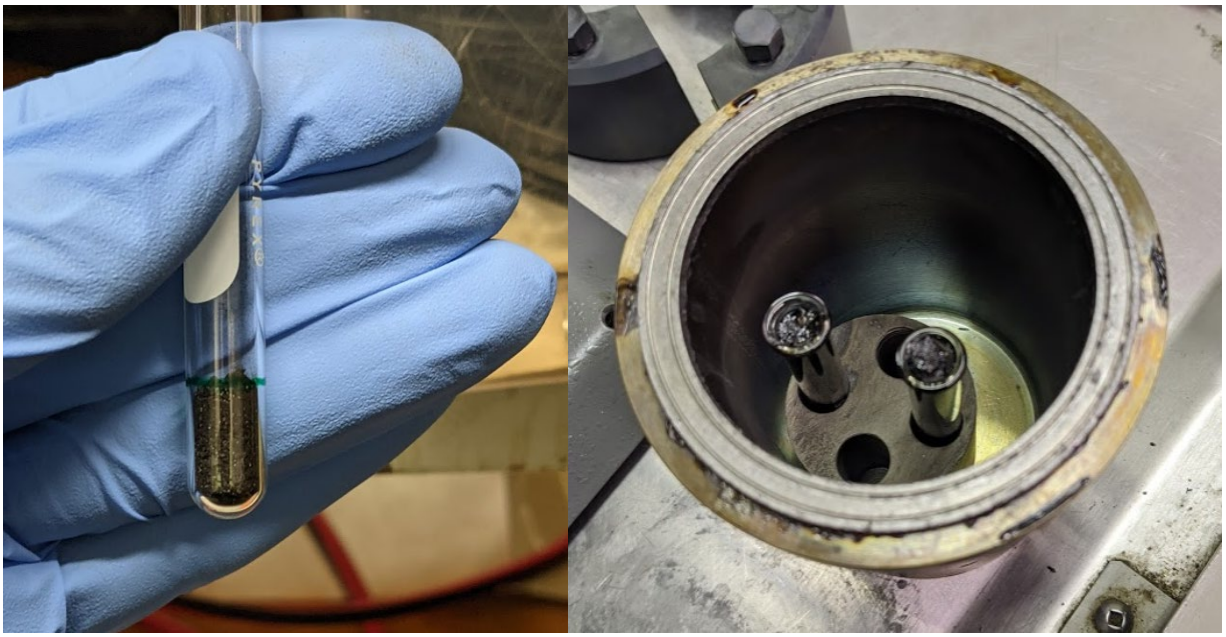


Figure 6. (Left) Sample in the Pyrex tube pre-test, which is then placed in the sample holder inside the reactor vessel. (Right) A view of the inside of the reactor showing the sample holder along with two asphaltenes samples that underwent pyrolysis.

MICROSCOPY

In order to capture micrographs of each asphaltene sample, the glass vial holding the sample was broken via a glass cutter to retrieve the sample. The asphaltene specimen was then mounted in epoxy, followed by grinding and polishing with 1 micron diamond slurry. The samples could then be imaged using an optical microscope.

The mesophase yield was determined using an adaption of ASTM D4616, where a grid of 1000 points was superimposed across the sample. A positive point would be one that is in contact with mesophase while a negative point would be in contact with epoxy, porosity or asphaltene that did not convert. Three measurements were taken on each sample by moving the grid to a different location. No overlapping of the grids occurred.

E. PROJECT RESULTS

Please provide a narrative describing the key results using the project's milestones as sub-headings.

- Describe the importance of the key results.
- Include a discussion of the project specific metrics and variances between expected and actual performance.

RESPOND BELOW

MESOPHASE YIELD

Table 3 contains the complete run of experiments, while Fig. 7 reports the yield of the mesophase content for each experiment. Note that the experiments are run out of order and labelled blindly, so when microscopy calculations were taking place the parameters of the test were unknown at the time. These two methods ensure a decreased chance of any bias. The highest mesophase content can be seen in sample 11B (Fig. 8). When compared to the experimental parameters of Table 3, several observations can be made. First, the addition of diesel had a serious negative consequence on the mesophase yield. In both cases only a maximum 16% mesophase was observed (Figure 9). It is suggested that the high porosity in both cases is from the fact that no stirring took place during the reaction. It is possible to add a mechanical stirring action, but the reactor was not equipped with a stirrer at this time. It is hoped that by introducing stirring a more homogenous mesophase pre-cursor could be produced.

Table 3: The complete design of experiments showing the various parameter changes achieved during this project.

	% MP	450C	425C	Raw	C5	2 MPa	4 MPa	Solvent	Add	
1A	0									
1B	10.7							Diesel		
2A	0									
2B	15.6							Diesel		
3A	17.4									
3B	36.1							Tetra		1:1 Tetra
4A	24.2									
4B	40.1							Tetra		1:1 Tetra
5A	39.6								Anth	
5B	40.8							Tetra		25% Tetra
6A	0								Anth	
6B	38.4							Tetra		25% Tetra
7A	44.0							Tetra		
7B	30.7							Iso		25% Iso
8A	0							Tetra		
8B	28.4							Iso		25% Iso
9A	0							Tetra	Anth	
9B	36.1								Pyrene	10% Pyrene
10A	29.1							Tetra	Anth	
10B	34.5								Pyrene	10% Pyrene
11A	27.5									
11B	48.0							Tetra		25% Tetra
12A	9.3									
12B	42.3							Tetra		25% Tetra
13A	28.8							Iso		
13B	0								Anth	20% Anth
14A	30.5							Iso		
14B	0								Anth	10% Anth
15A	45.8									CO2 Purge
16A	34.2									CO2 Purge

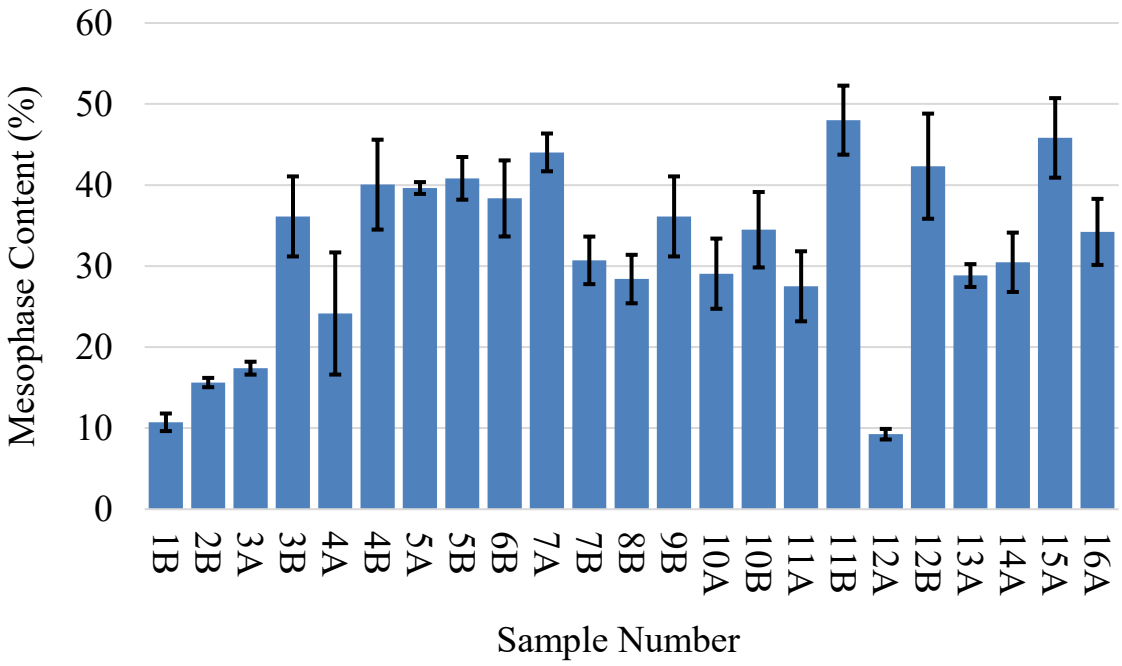


Figure 7: The mesophase content of each sample reported as a percentage of the total area.

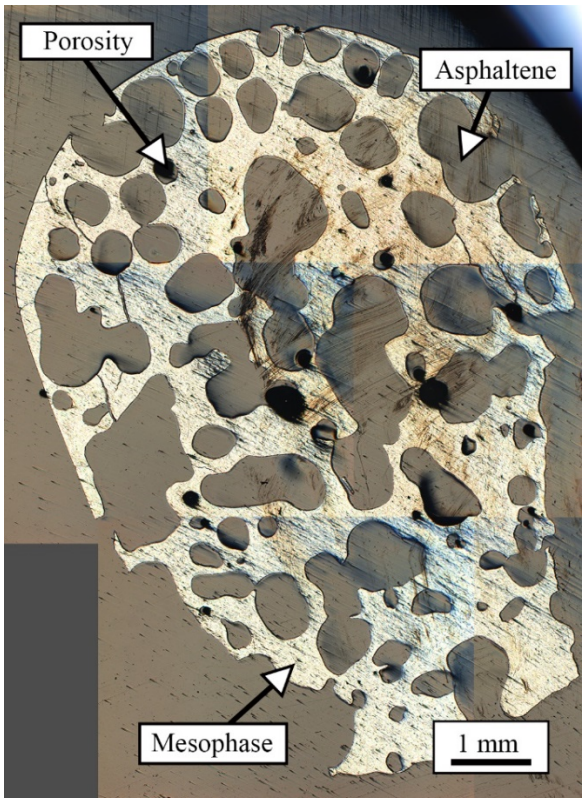


Figure 8: A compilation of micrographs of sample 11B showing 48% mesophase.

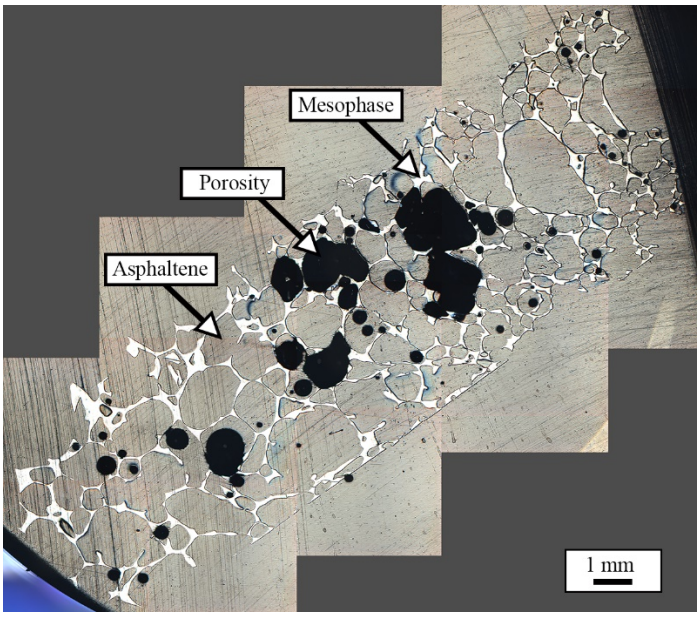


Figure 9: A compilation of micrographs of sample 2B showing 16% mesophase.

Secondly, in order to understand better the relationships between the parameters a mean effects plot was created (Fig. 10). The plots show the mean effects of the different parameters in relation to mesophase content. This allows for quick decision-making regarding screening of parameters. The largest effect is from the addition of a solvent, which is highlighted in Fig. 11 where a Pareto of means is presented. Additionally, the second parameter with the largest effect is the initial feedstock, emphasizing the importance of the filtration step mentioned earlier in methodology to capture the C5 asphaltenes. Mesophase yield of raw asphaltene pyrolysis only reached 28%. There is an indication of pressure having an effect on mesophase content, however, it must be noted that the recorded pressure is that of the *initial* pressure within the reactor. Throughout the experiment the pressure inside the reactor increases due to the nitrogen, volatiles and solvents in gaseous form expanding in the closed system. In some cases, the pressure increased to 8 MPa before depressurization occurs.

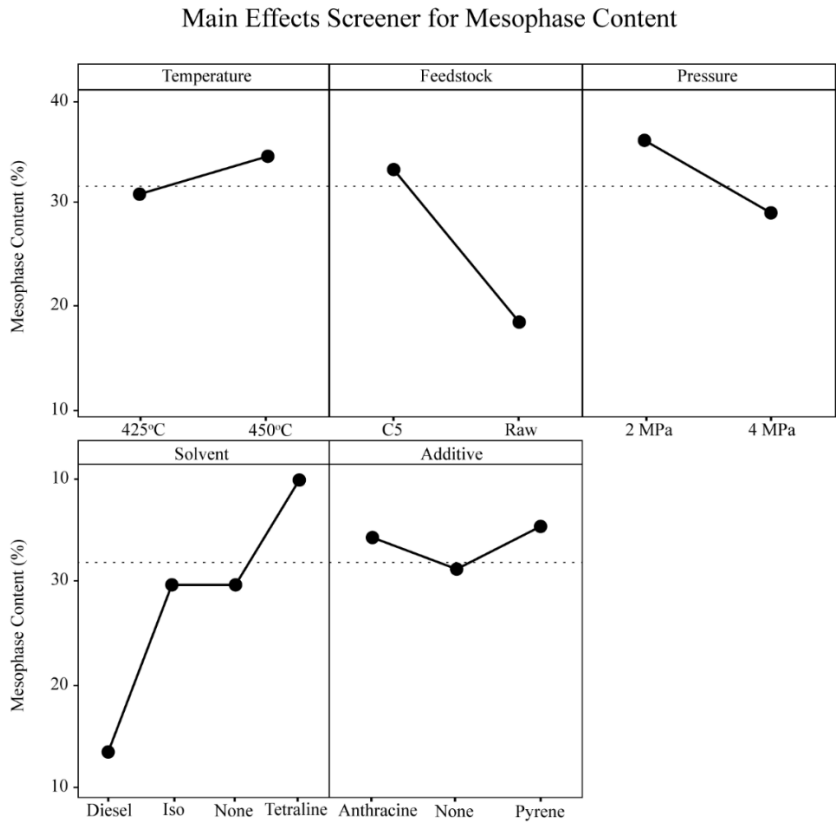


Figure 10: Mean effects screener for mesophase content. The type of solvent used has the largest effect on mesophase content followed by the type of feedstock, indicating the importance of filtration before pyrolysis.

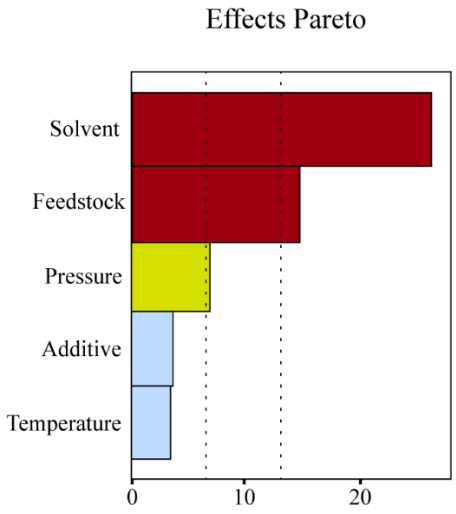


Figure 11: A Pareto chart showing the most significant effects on mesophase content. Effect is calculated as the largest mean minus the smallest mean. Red indicates two deviations, yellow is one deviation and grey indicate the effect is within one standard deviation of the mean.

GLASS TRANSITION TEMPERATURES

DSC profiles were captured for the asphaltene samples that underwent pyrolysis and are presented in Fig. 12. The raw data for DSC can be found in Appendix II. The DSC runs outline the major differences between batch A and batch B (i.e. 1A vs 1B), which was whether the volatiles were vented our contained within the reactor during pyrolysis, respectively. Leaving the volatiles within the reactor allowed the pressure to build to 8 MPa in some cases. For samples 8A and 9A the initial decline in heat output is most likely due to remaining volatiles within the air-dried sample. Regardless, the glass transition point remains around 350 °C for batch A. For batch B, when the pressure is allowed to build the DSC data becomes less noisy, but there is a major shift to a higher transition point. When combined with the TGA analysis (Fig. 13 and Appendix III) that shows decomposition around 350 °C for batch A, it can be determined that batch B contains a much more stable mesophase product. Therefore, there is an inverse relationship between glass transition temperature and stability that will require further optimization.

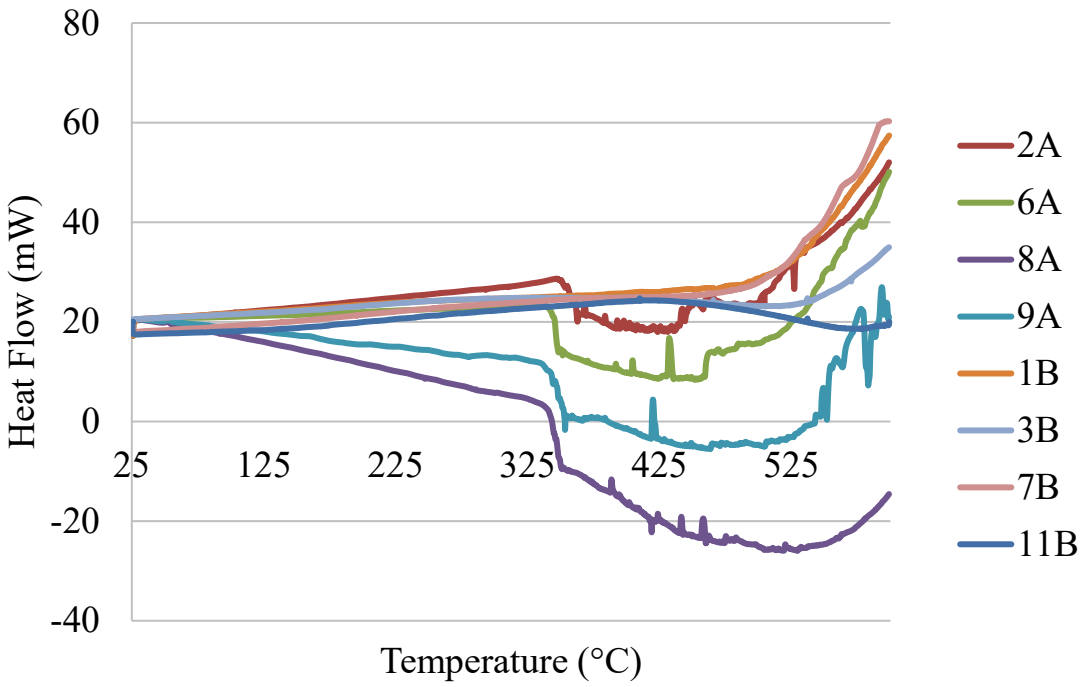


Figure 12: DSC results for the products of the pyrolysis tests

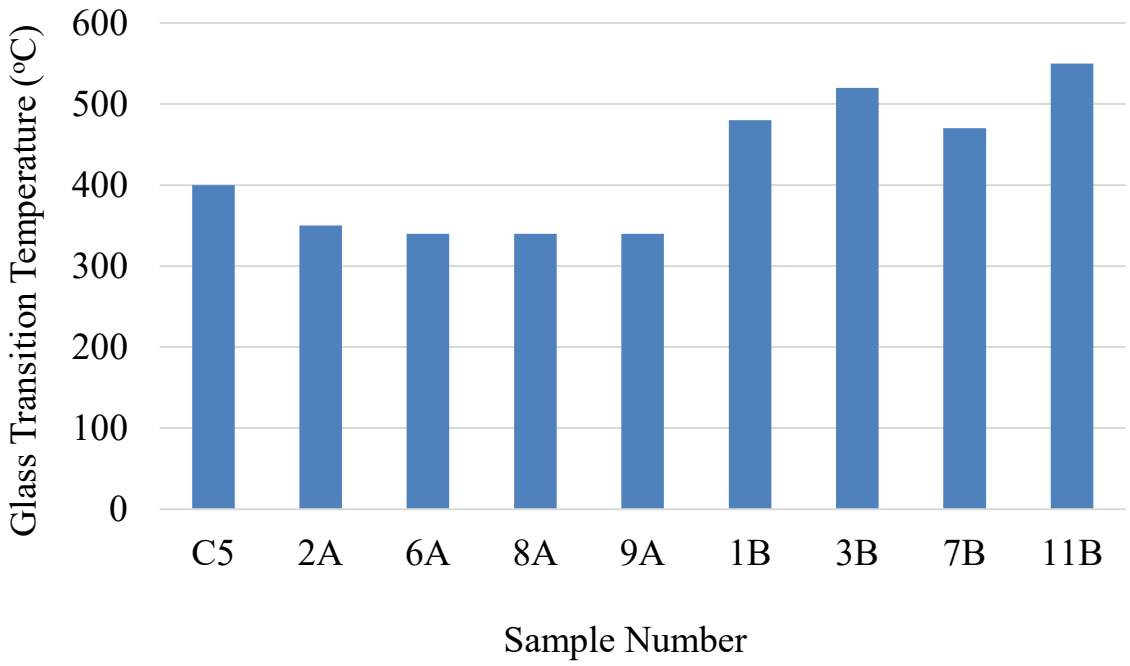


Figure 13: The glass transition temperature (T_g) of select samples acquired from DSC.

The mass of each sample was measured before and after pyrolysis, allowing observations to be made about the yield of sample expected after the reaction (Fig. 14). It is important to note that the sample with the highest mesophase content also proved to retain one of the lowest masses on completion of the reaction. It is suggested that the mass from tetralin is evaporated during the experiment, resulting in a much lower mass after pyrolysis. Fig. 14 can be combined with the TGA results of Appendix III, which shows the approximate mass after heating in an inert atmosphere to 900 °C. Heating to 900 °C would be synonymous with the carbonization step required for CF production. For example, sample 11B retained 94% of its mass after pyrolysis and TGA, suggesting a potentially higher yield of CF production.

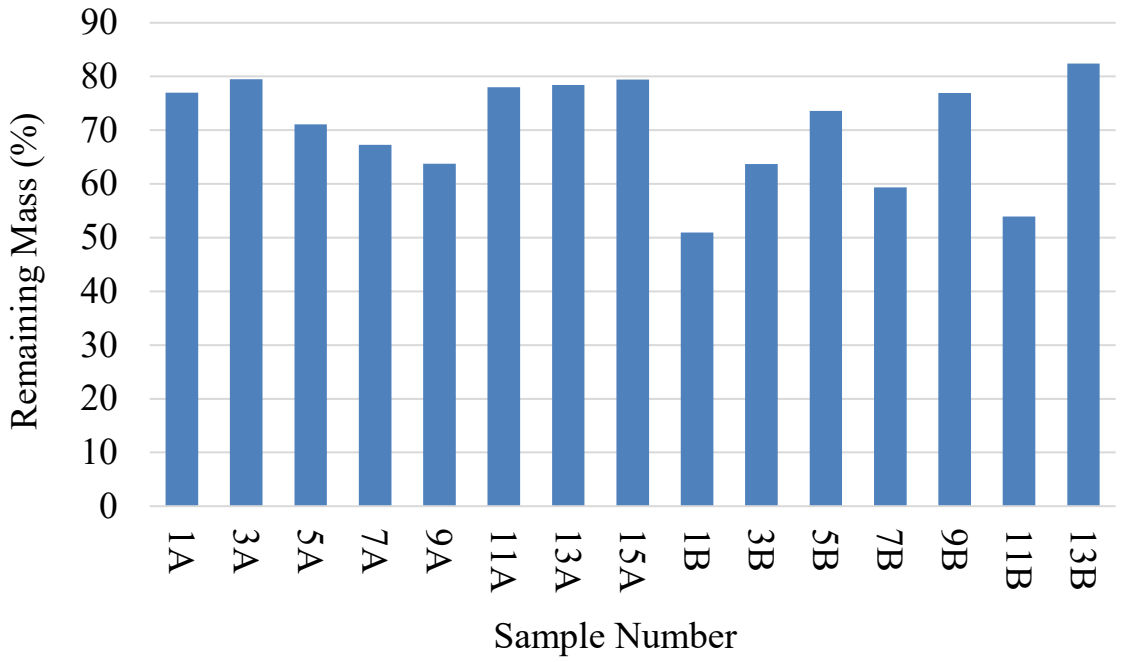


Figure 14: The remaining mass of the samples after pyrolysis

CARBON FIBRE SPINNING

A secondary objective of this project was to melt spin the mesophase pre-cursor into a carbon fibre for mechanical testing. Unforuntately, the University imposed heavy restrictions, which cu tour project timein half. Since our main objective was to focus on the mesophase precursor, we spent our resources on optimizing our procedure. The project continues to date and melt spinning data will be available for Phase II of the Carbon Fibre Grand Challenge.

F. KEY LEARNINGS

Please provide a narrative that discusses the key learnings from the project.

- Describe the project learnings and importance of those learnings within the project scope. Use milestones as headings, if appropriate.
- Discuss the broader impacts of the learnings to the industry and beyond; this may include changes to regulations, policies, and approval and permitting processes

RESPOND BELOW

- Appearance of mesophase in asphaltene sample S2 upon processing under heat and pressure and with various treatment options is confirmed.
- The softening point of the feedstock is higher than desirable range of spinning. It is confirmed that further heat treatment especially at pyrolysis temperatures increases the softening point.
- Hydrogenation is a well-established method to increase the hydrogen to carbon ratio of pitch material in CF manufacturing applications. Various solvents with such hydrogen donating properties were explored to achieve this objective, which has been shown to increase mesophase yield as well as lower the softening point. Aromatic diluents such as anthracene and pyrene serve to lower the average molecular weight of the asphaltenes to reduce the softening point. Aromaticity of the feedstock is also boosted by their presence, which is conducive to mesophase formation.
- There exists a relationship with mesophase content and softening point. By increasing the mesophase content, the softening point also increases, which makes the spinnability of the pre-cursor decrease.

G. OUTCOMES AND IMPACTS

Please provide a narrative outlining the project's outcomes. Please use sub-headings as appropriate.

- **Project Outcomes and Impacts:** Describe how the outcomes of the project have impacted the technology or knowledge gap identified.
- **Clean Energy Metrics:** Describe how the project outcomes impact the Clean Energy Metrics as described in the *Work Plan, Budget and Metrics* workbook. Discuss any changes or updates to these metrics and the driving forces behind the change. Include any mitigation strategies that might be needed if the changes result in negative impacts.
- **Program Specific Metrics:** Describe how the project outcomes impact the Program Metrics as described in the *Work Plan, Budget and Metrics* workbook. Discuss any changes or updates to these metrics and the driving forces behind the change. Include any mitigation strategies that might be needed if the changes result in negative impacts.
- **Project Outputs:** List of all obtained patents, published books, journal articles, conference presentations, student theses, etc., based on work conducted during the project. As appropriate, include attachments.

RESPOND BELOW

PROJECT OUTCOMES AND IMPACTS:

The major outcomes and impacts of this project were the bridging of the following knowledge gaps:

A standardized method that produces a consistent feedstock for pyrolysis.

Solution: Starting with ASTM standard D4124 and SARA (Saturates, Aromatics, Resins and Asphaltenes) classifications and building on the recommended guidelines in the academic literature. We combined our expertise with the Department of Chemistry at the University of Alberta. Drs. Jeff Stryker, Rik Tykwinski, David Scott and Robin Hamilton who have several years of research investigating asphaltene upgrading. We then created a method for lab scale extraction of asphaltenes, using the material provided by the supplier, which is outlined in the methodology. This procedure allows for an efficient way to collect the C5 asphaltenes needed for optimized mesophase formation as per the results of the DOE.

The physical and chemical parameter relationships that affect the formation of a mesophase precursor are unknown.

Solution: We investigated the effect of temperature and pressure and other parameters in a systematic way using a DOE method. The experimental matrix determined that the addition of certain solvents and the quality of the feedstock prior to pyrolysis are critical for mesophase formation. Secondly, we discovered that fluctuations in temperature and additives have a very minor effect on mesophase formation during pyrolysis. In addition, we identified an alternative and indirect way to calculate the softening point through DSC and TGA. Indirect calculation of the softening point is critical since the

softening point measurement device with the required temperature range is rare and expensive in Alberta partner labs.

Lack of existing research infrastructure in Alberta.

Solution: The conversion of pitch into CF requires a strong mesophase content. There should be no difference with an asphaltene feedstock, so it is critical that mesophase formation be maximized. By focusing on the pre-cursor to CF, we have essentially built a foundation for further research in this sector. Without a proper pre-cursor, sub-par mechanical properties are to be expected from resulting CF. It is critical that the research within this report be disseminated in a way that is impactful to Alberta, which means careful deliberation of receiving parties with regards to the intellectual property within.

We propose an innovation center like facilities such as those in the United States (Carbon Fiber Technology Facility – Oak Ridge National Lab) and Australia that has the potential to boost the research speed, quality and technology development. This will be achieved through continued research of the mesophase precursor, which we believe is mandatory for any CF production in Alberta. A facility to melt spin CF is one requirement, but a facility to produce a high mesophase content pre-cursor trumps any production of CF. Once the problem of lowering the softening point of the precursor is achieved, CF production can begin immediately.

CLEAN ENERGY METRICS

The number of publications expected from this project remains at two, which are currently being drafted from the results obtained recently. There are no patents as of yet until a concrete recipe can be determined, as well as consultation with TEC Edmonton. The significant time loss due to University restrictions has limited our ability to complete the experimental work within the first half of the project. Originally, the second half of the project was melt spinning along with attraction of key industrial partners, which we are continuing to pursue at the time of this report.

For mitigation purposes, the work will be continued past the deadline for Phase I. This will allow for the completion of most of the clean energy metrics. By melt spinning, a connection was made with Dr. Weixing Chen, who was also a member of Phase I. It is hoped that our expertise in precursor formation along with his expertise in melt spinning will allow for a significant boost in research output.

PROGRAM SPECIFIC METRICS

As above, the restrictions implemented due to the Covid 19 pandemic reduced our time frame to less than half, resulting in our inability to realistically produce a CF product suitable for end users. Again, the work is continuing in order to achieve all the goals we initially set before Covid 19 restrictions were implemented. The publications metric is still on schedule, which will allow for tangible information for potential end users. We believe that the mesophase pre-cursor acts as a new technology, but the TRL remains low at this stage due to the delay in producing CF.

PROJECT OUTPUTS

As of the writing of this report, no publications have been filed.

H. BENEFITS

Please provide a narrative outline the project's benefits. Please use the subheadings of Economic, Environmental, Social and Building Innovation Capacity.

- **Economic:** Describe the project's economic benefits such as job creation, sales, improved efficiencies, development of new commercial opportunities or economic sectors, attraction of new investment, and increased exports.
- **Environmental:** Describe the project's contribution to reducing GHG emissions (direct or indirect) and improving environmental systems (atmospheric, terrestrial, aquatic, biotic, etc.) compared to the industry benchmark. Discuss benefits, impacts and/or trade-offs.
- **Social:** Describe the project's social benefits such as augmentation of recreational value, safeguarded investments, strengthened stakeholder involvement, and entrepreneurship opportunities of value for the province.
- **Building Innovation Capacity:** Describe the project's contribution to the training of highly qualified and skilled personnel (HQSP) in Alberta, their retention, and the attraction of HQSP from outside the province. Discuss the research infrastructure used or developed to complete the project.

RESPOND BELOW

ECONOMIC

It is expected that once a suitable thermal recipe is agreed upon, production of CF will begin on a laboratory scale. The literature is clear that a high mesophase content will result in CF with a high modulus and strength, so we are optimistic about the mechanical properties of the proposed CF. This will require a facility to scale up to the requirements set out by Alberta Innovates. If a facility is not created, then a partnership with a reputable CF producer would be necessary. However, the production of a facility capable of fabricating CF from the mesophase cursor is realized, dozens of jobs will be created into a new employment sector for Alberta.

The CF production will continually increase to the point that it will attract new investments to Alberta through purchasing of CF. Due to our natural resources, the supply of CF will only be limited to the speed of production, which can be increased with more equipment and personnel as needed.

ENVIRONMENTAL

Through producing a value-added product such as CF, the asphaltene waste produced from Alberta's energy sector will provide a continuous stream of material supply for CF production in Alberta. The reduction of waste will be an indirect way of diverting GHG emissions, while avoiding destruction of the natural environment.

SOCIAL

This project holds immense promise in the area of value for Alberta. If the mesophase pre-cursor can be replicated easily and then scaled, there will be an endless supply of a waste material than can be transformed into a value-added product. Due to Alberta's standing within the oil and gas sector, it is paramount that the move to adopt asphaltenes into the CF production stream begins. However, this will require an entirely new sector of pre-cursor formation that would require investment into Alberta.

BUILDING INNOVATION CAPACITY

A process stream involving a mesophase pre-cursor would open a brand-new field of expertise in Alberta that would attract HQSP. Due to the sophisticated chemical engineering requirements with respect to scaling, mesophase pre-cursor production would be in constant improvement. Dozens of HQSP will be required to continually optimize the process so that more efficient methods can be obtained in producing mesophase pre-cursors. Most notably, a pre-cursor with a yield of 100% mesophase.

I. RECOMMENDATIONS AND NEXT STEPS

Please provide a narrative outlining the next steps and recommendations for further development of the technology developed or knowledge generated from this project. If appropriate, include a description of potential follow-up projects. Please consider the following in the narrative:

- Describe the long-term plan for commercialization of the technology developed or implementation of the knowledge generated.
- Based on the project learnings, describe the related actions to be undertaken over the next two years to continue advancing the innovation.
- Describe the potential partnerships being developed to advance the development and learnings from this project.

RESPOND BELOW

Implementation of the technology has already begun, as we collaborate further with the Department of Chemistry at the University of Alberta. As mesophase content increases, we are actively researching a solution to lower the softening point. We are confident it can be achieved and the next logical step is to begin CF production, where a melt spinning run has already been scheduled. The long-term plan is to scale our intellectual findings. They can be easily replicated due to our cataloguing of all experimental steps.

The next two years will follow a rigorous experimental plan of mesophase production followed by CF production. Our aim is to gradually increase scale over time so that long term investors will see viability of investing in Alberta's future.

There is now a strong collaboration between our group and the Department of Chemistry, which has allowed us to provide a much more thorough chemical and thermal analysis of the asphaltenes and their behaviour. This collaboration will continue and a joint proposal is expected for Phase II.

J. KNOWLEDGE DISSEMINATION

Please provide a narrative outlining how the knowledge gained from the project was or will be disseminated and the impact it may have on the industry.

RESPOND BELOW

The knowledge gained within his project will be disseminated in two planned publications. There will be little impact to industry at this stage, but will be used as a source of information as we continue talks with potential research partners. However, the knowledge gained during Phase I has already laid the foundation for Phase II where CF production and scaling can begin. We are confident that the mesophase pre-cursor that is being optimized within this report will allow for CF mechanical properties that surpass current benchmarks.

K. CONCLUSIONS

Please provide a narrative outlining the project conclusions.

- Ensure this summarizes the project objective, key components, results, learnings, outcomes, benefits and next steps.

RESPOND BELOW

The main objective of this project was to develop a methodology to produce a mesophase pre-cursor from Alberta oilsands asphaltenes. From the sample supplied by Alberta Innovates, we were able to achieve a mesophase content of 48%, with the number being much higher if porosity is not taken into account. The key components of the project were establishment of a suitable DOE along with a collaboration with the Department of Chemistry. The collaboration with Chemistry allowed us to pursue more advanced techniques at a quicker pace, such as vacuum extraction, DSC, TGA and elemental analysis.

L. ACKNOWLEDGEMENTS

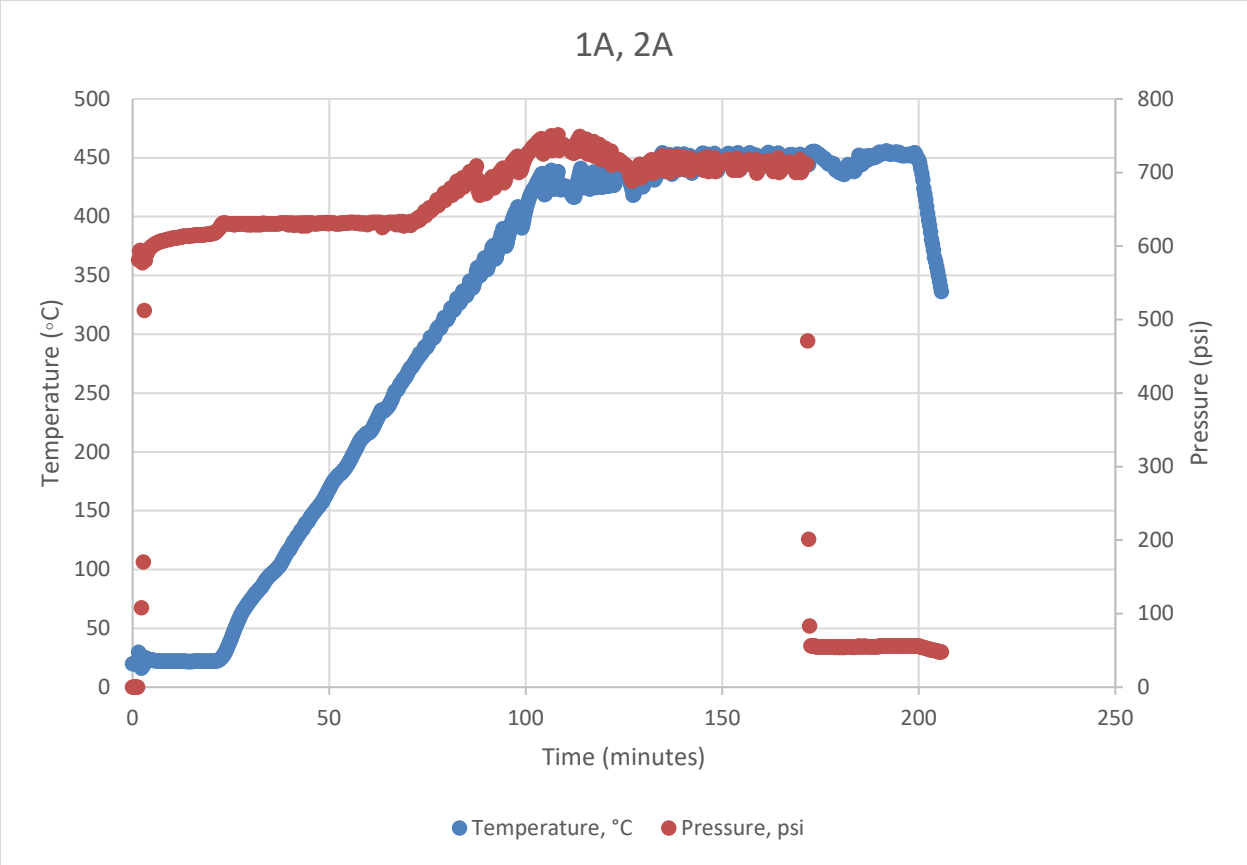
We thank Dmytro Pantov for his work on setting up and helping with the execution of pyrolysis experiments. We also thank Behnaz Bazoubandi and Dr. Raquel Pereira Reolon for their help to coordinate with Dr. Joao Soares' labs. A special thank you to Linda Kaert for financial expertise during this project.

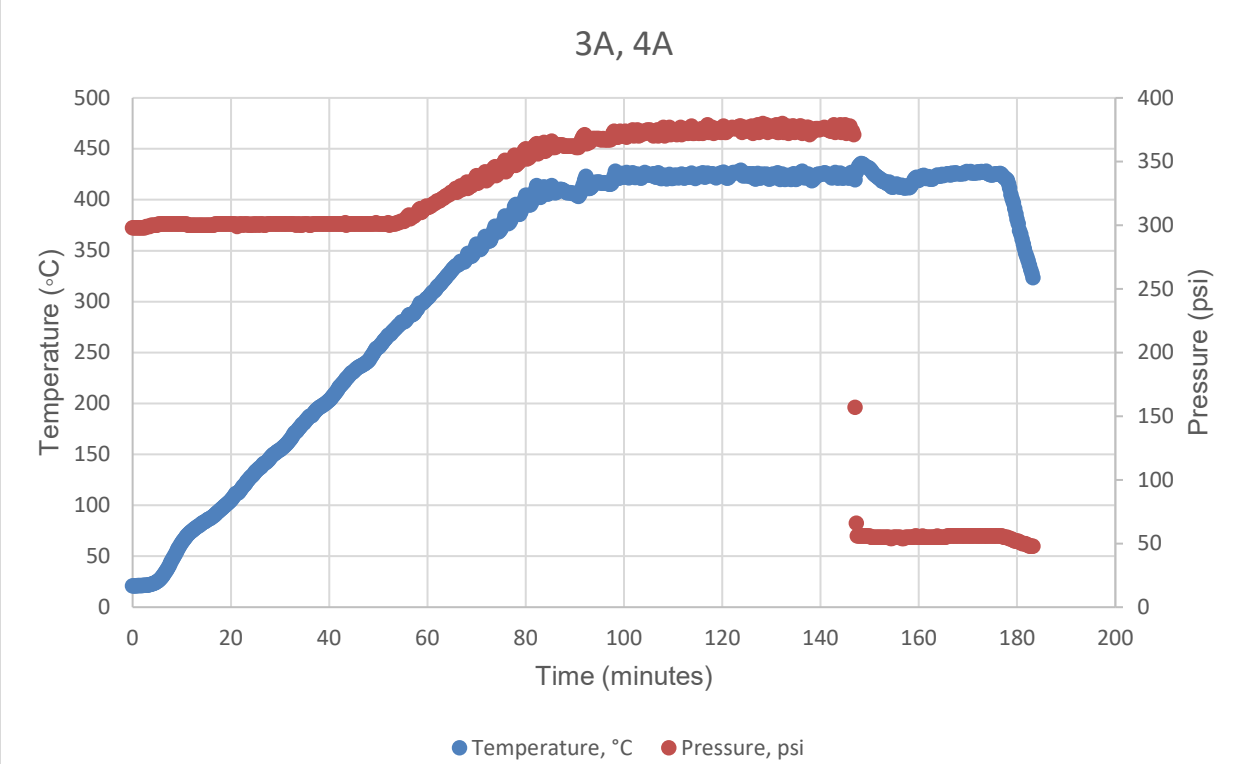
M. REFERENCES

Park, S. (2018). *Carbon Fibers.* Springer.

APPENDIX I: PRESSURE AND TEMPERATURE VS TIME PLOTS FOR THE PYROLYSIS TESTS

The pressure and temperature profiles for the pyrolysis tests conducted in this study are presented in full. As laid out in the report, a rate of 5 °C/min was chosen as a heating rate to the final temperature for pyrolysis. The sample was maintained at the final temperature for 1.5 hrs to react under inert gas (N_2). 0.5 hrs before the conclusion of the test, the system was depressurized to boost mesophase formation and coalescence.




3A, 4A
Temperature (°C)
Pressure (psi)
Time (minutes)
0
50
100
150
200
250
300
350
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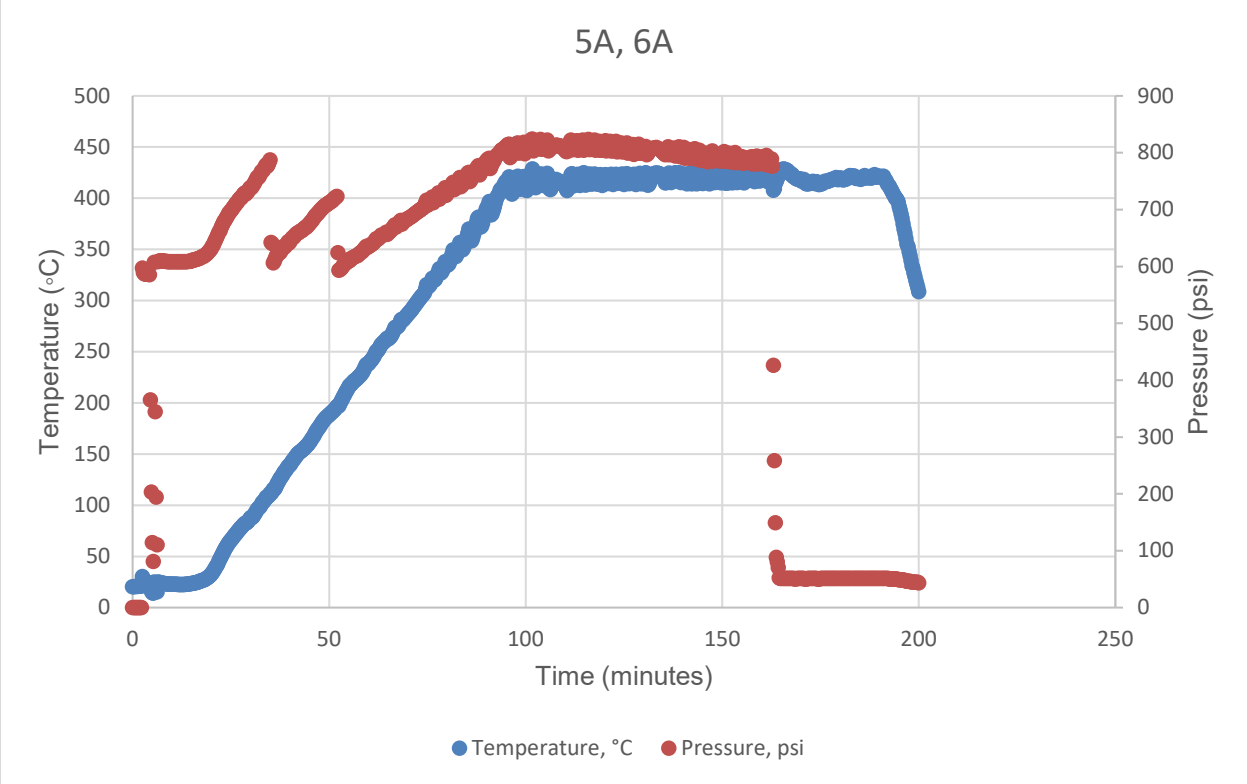
450
500
0
20
40
60
80
100
120
140
160
180
200
Temperature, °C
Pressure, psi


5A, 6A
Temperature (°C)
Pressure (psi)
Time (minutes)
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100
150
200
250
300
350
400
450
500
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200
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500
600
700
800
900
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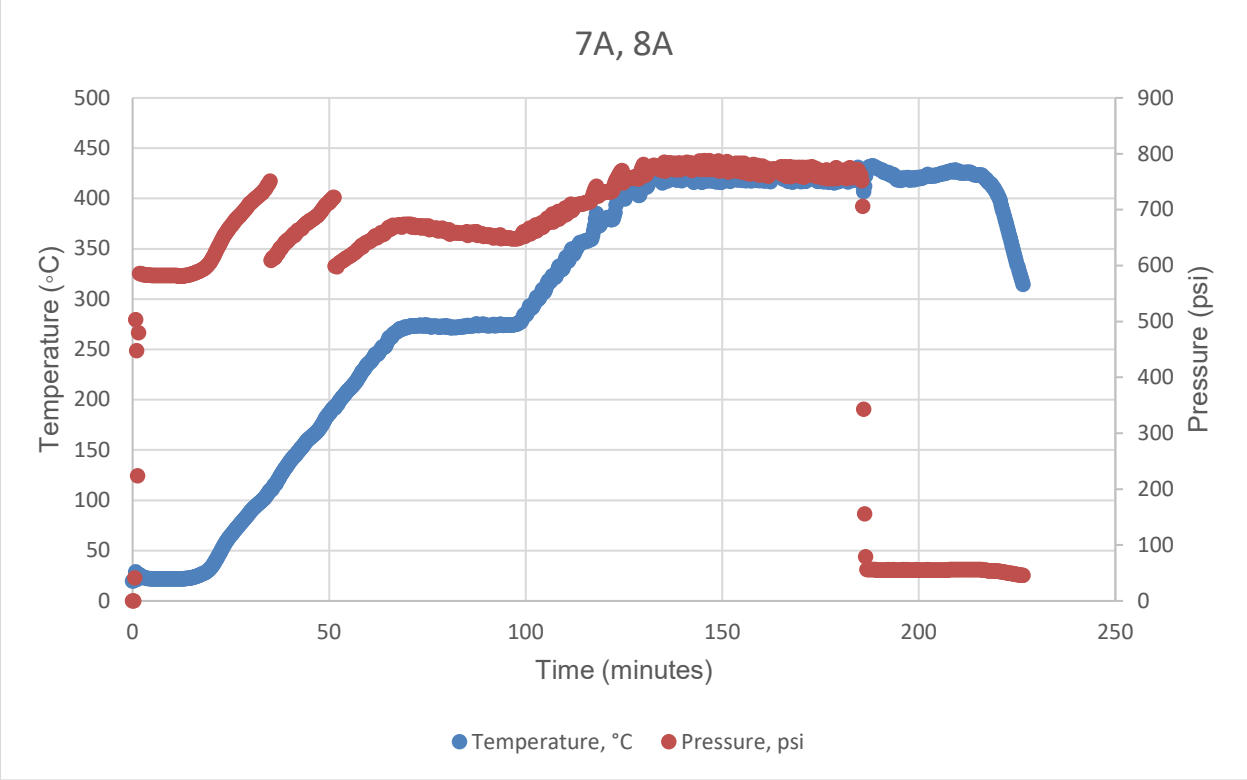
100
150
200
250
Temperature, °C
Pressure, psi


7A, 8A
Temperature (°C)
Pressure (psi)
Time (minutes)
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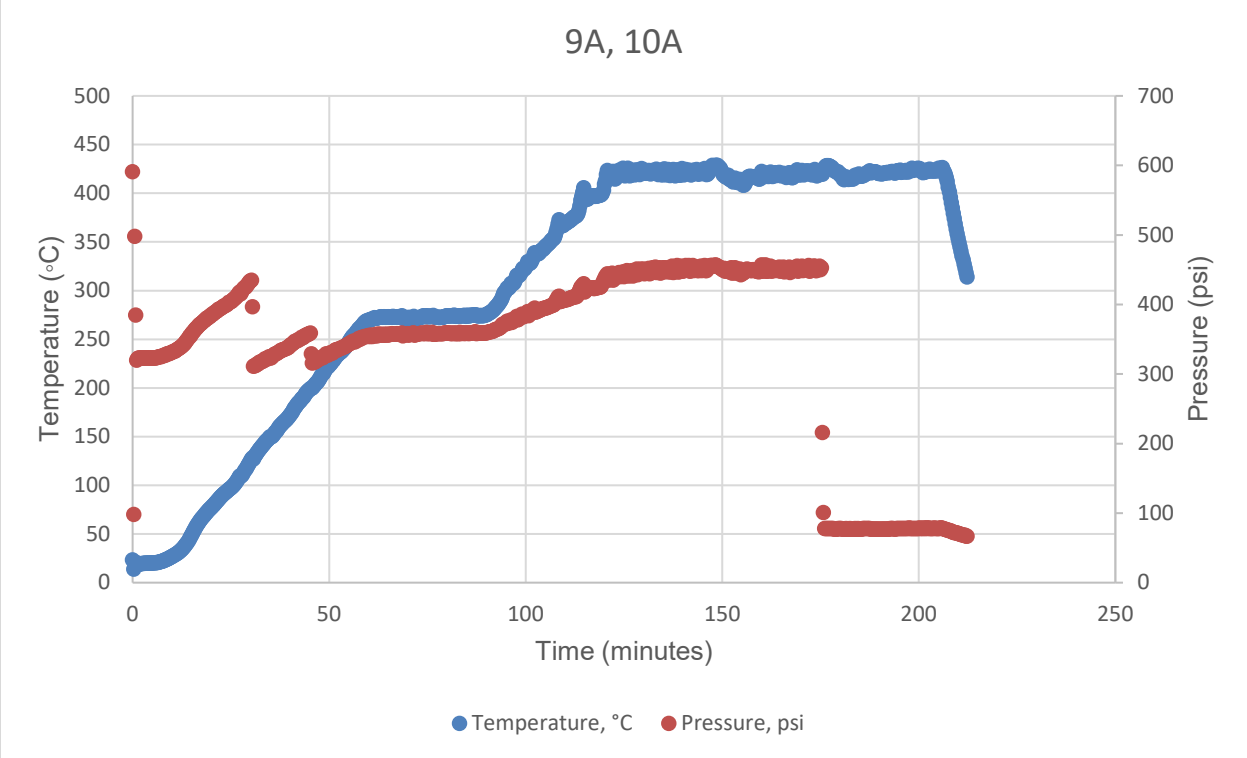
50
100
150
200
250
0
50
100
150
200
250
300
350
400
450
500
0
100
200
300
400
500
600
700
800
900
Temperature, °C
Pressure, psi


9A, 10A
Temperature (°C)
Pressure (psi)
Time (minutes)
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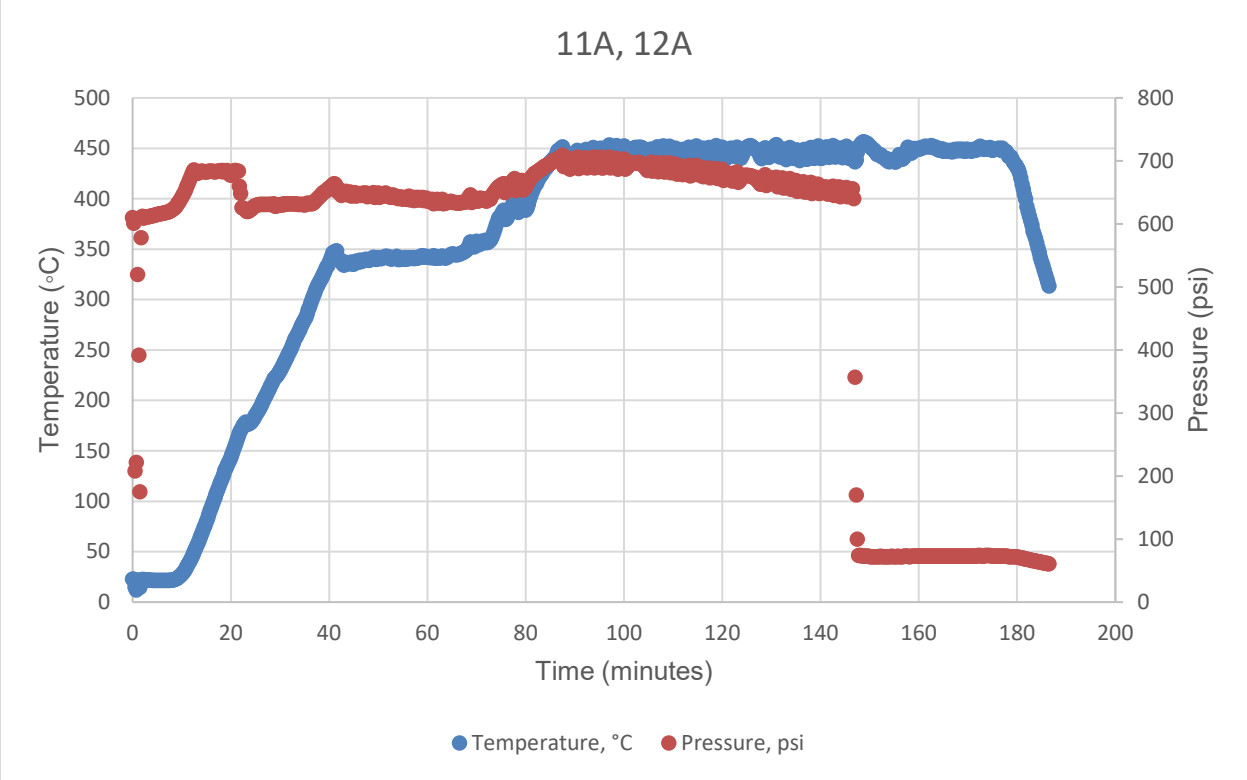
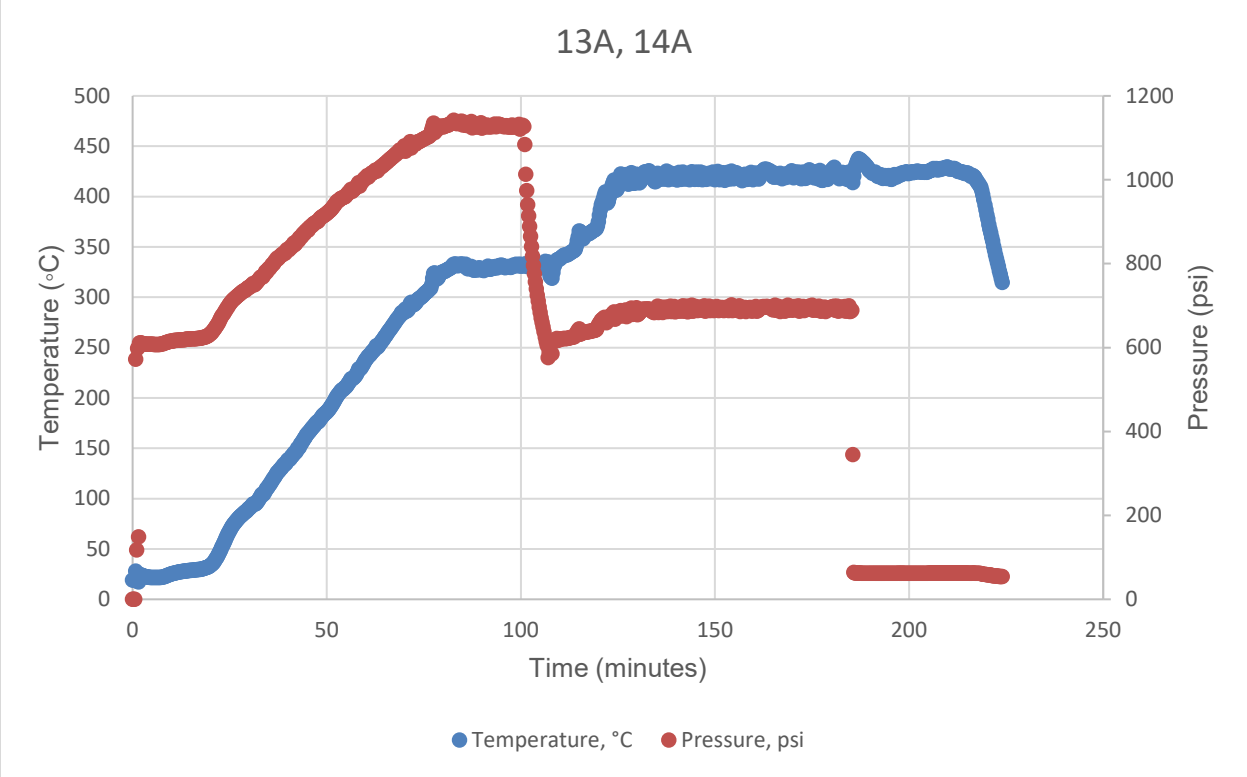
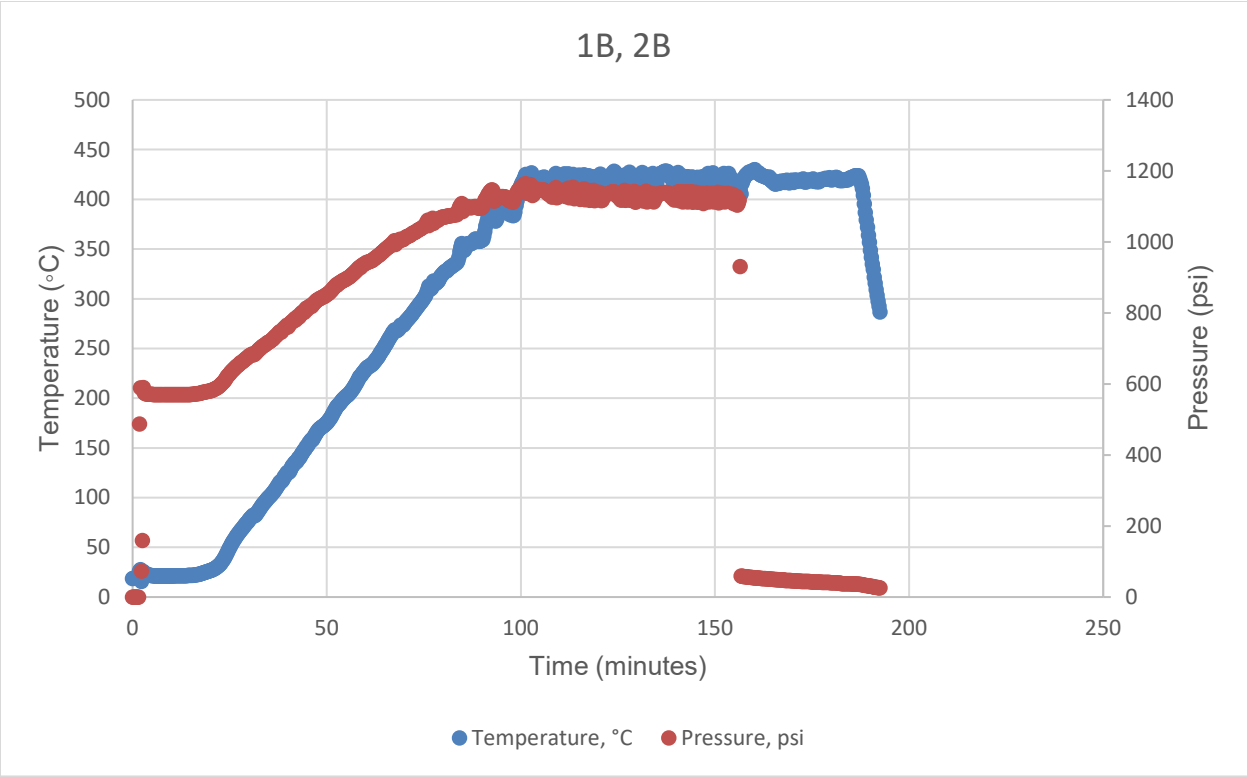
100
150
200
250
0
50
100
150
200
250
300
350
400
450
500
0
100
200
300
400
500
600
700
Temperature, °C
Pressure, psi





*The P-T vs time data for samples 15A and 16A was not saved due to an error. Our notes indicate Initial pressure was 2.25 MPa and by the end of heating ramp, the pressure increased to 5.8 MPa.


1B, 2B
Temperature (°C)
Pressure (psi)
Time (minutes)
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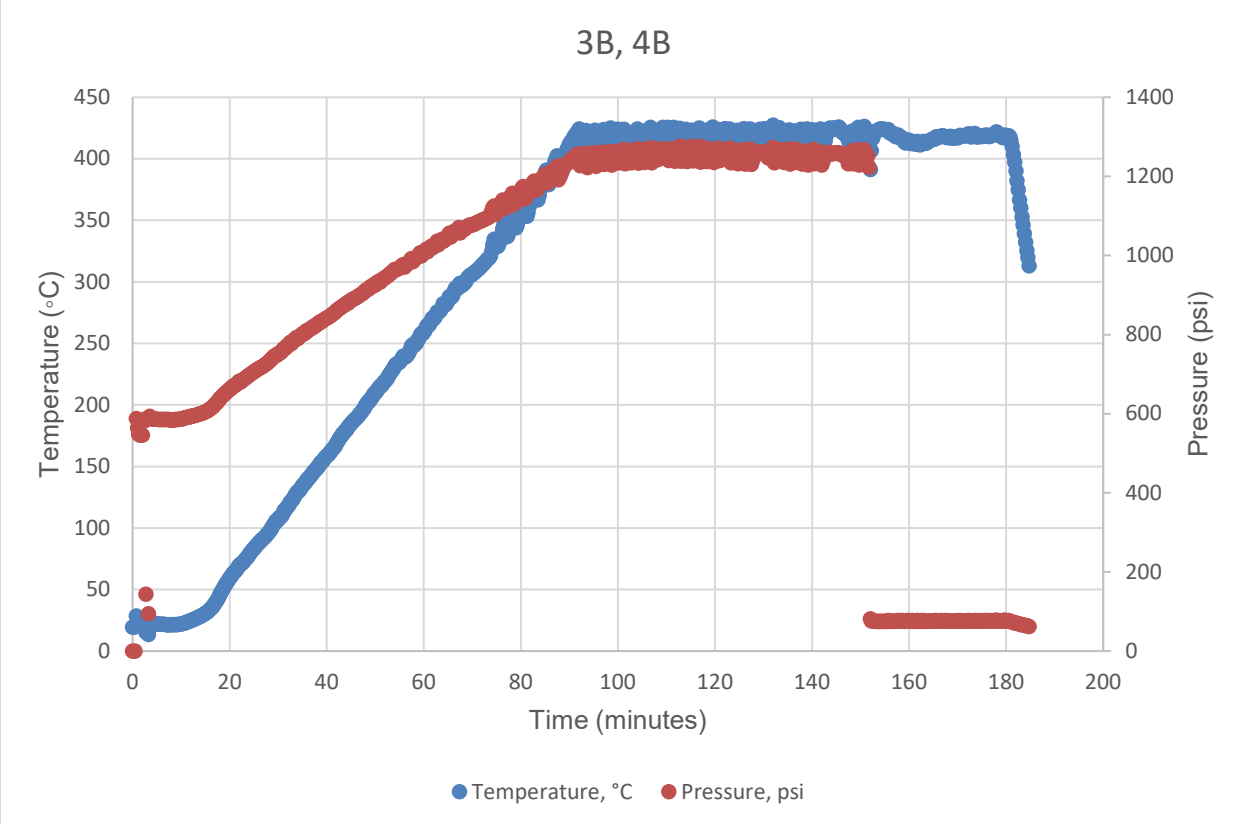
50
100
150
200
250
0
50
100
150
200
250
300
350
400
450
500
0
200
400
600
800
1000
1200
1400
Temperature, °C
Pressure, psi


3B, 4B
Temperature (°C)
Pressure (psi)
Time (minutes)
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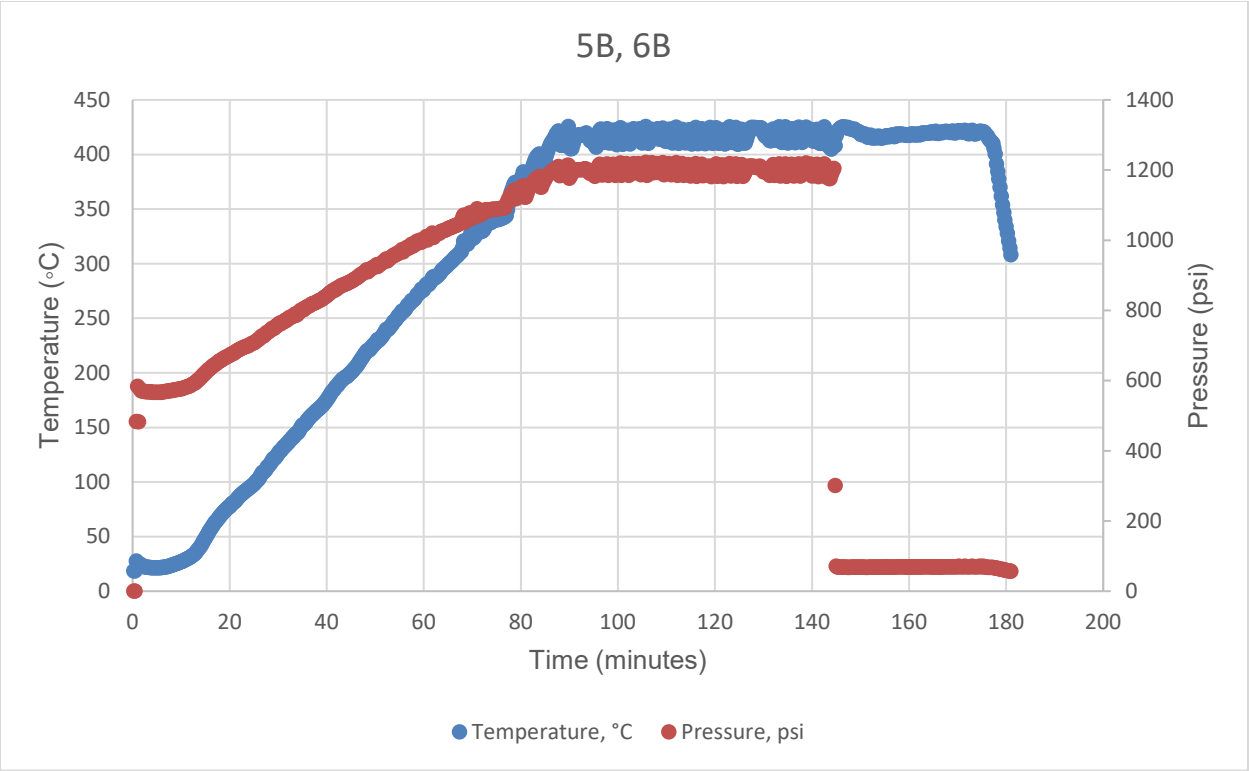
20
40
60
80
100
120
140
160
180
200
0
50
100
150
200
250
300
350
400
450
0
200
400
600
800
1000
1200
1400
Temperature, °C
Pressure, psi


5B, 6B
Temperature (°C)
Pressure (psi)
Time (minutes)
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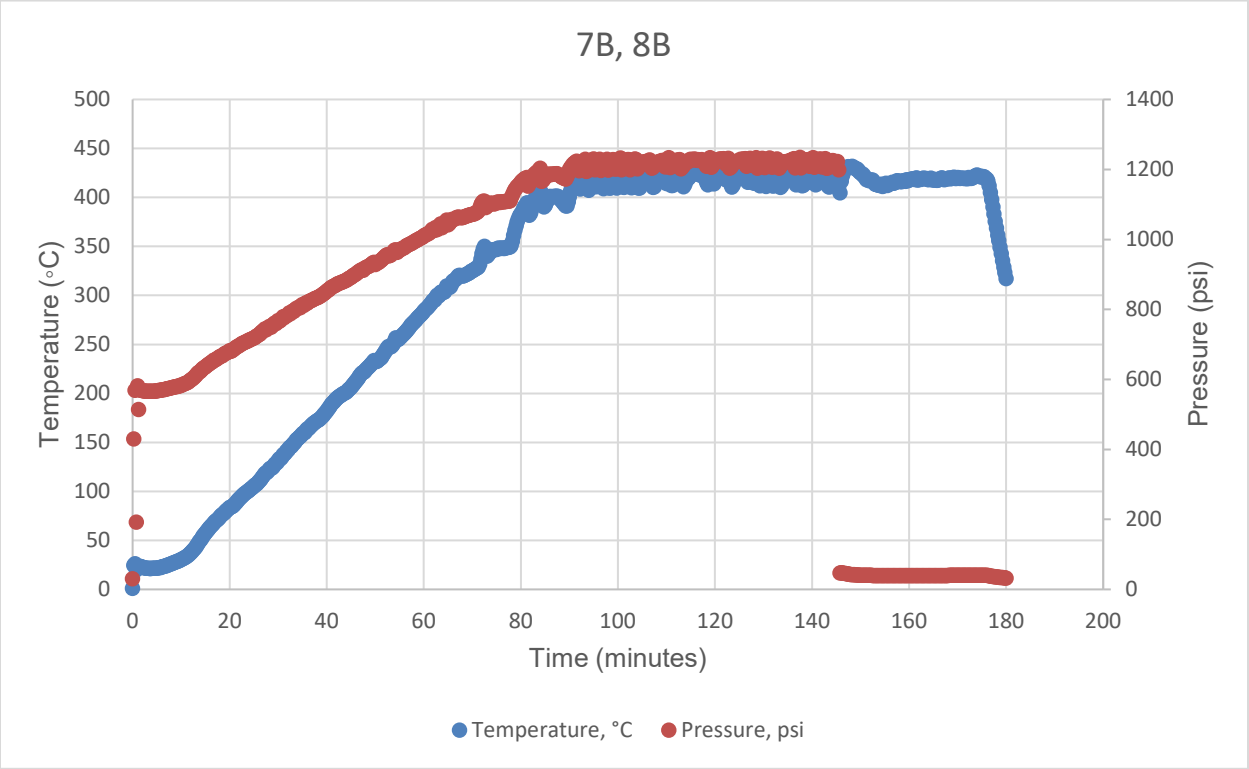
50
100
150
200
250
300
350
400
450
0
200
400
600
800
1000
1200
1400
0
20
40
60
80
100
120
140
160
180
200
Temperature, °C
Pressure, psi


7B, 8B
Temperature (°C)
Pressure (psi)
Time (minutes)
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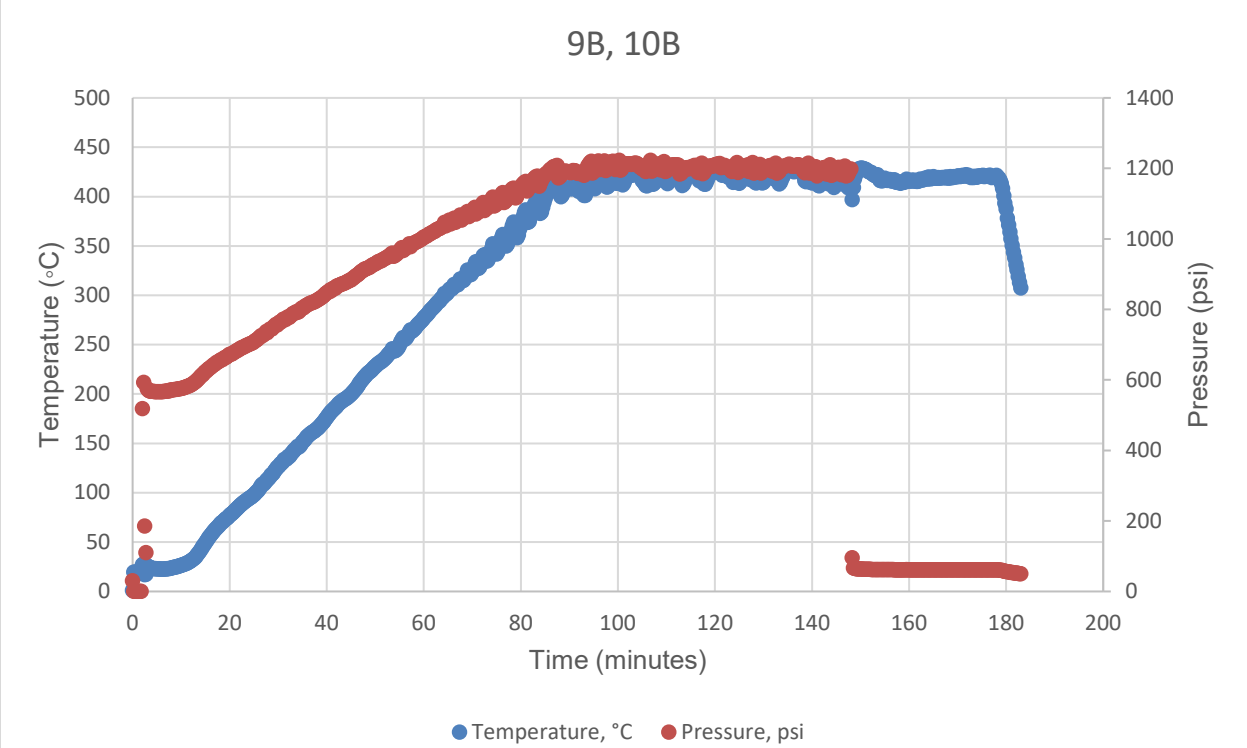
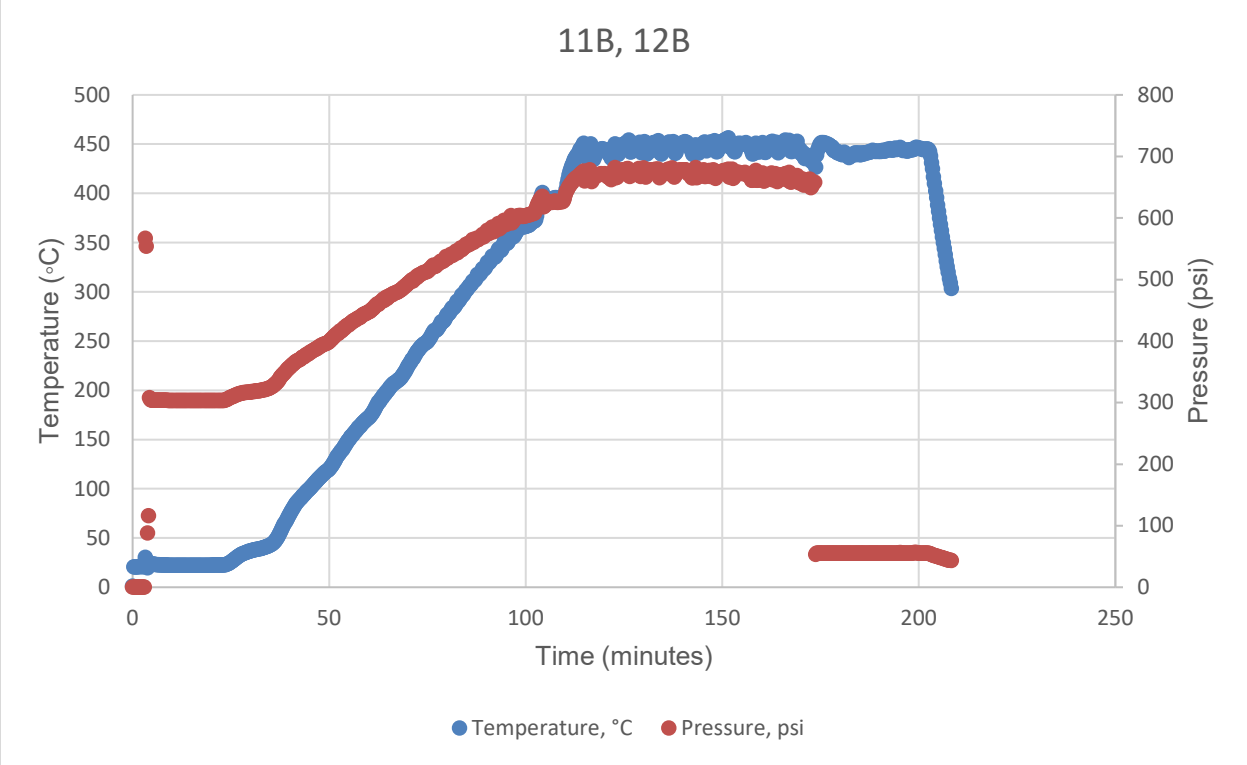
50
100
150
200
250
300
350
400
450
500
0
200
400
600
800
1000
1200
1400
0
20
40
60
80
100
120
140
160
180
200
Temperature, °C
Pressure, psi


9B, 10B
Temperature (°C)
Pressure (psi)
Time (minutes)
0
50
100
150
200
250
300
350
400
450
500
0
200
400
600
800
1000
1200
1400
0
20
40
60
80
100
120
140
160
180
200
Temperature, °C
Pressure, psi


11B, 12B
Temperature (°C)
Pressure (psi)
Time (minutes)
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100
150
200
250
300
350
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600
700
800
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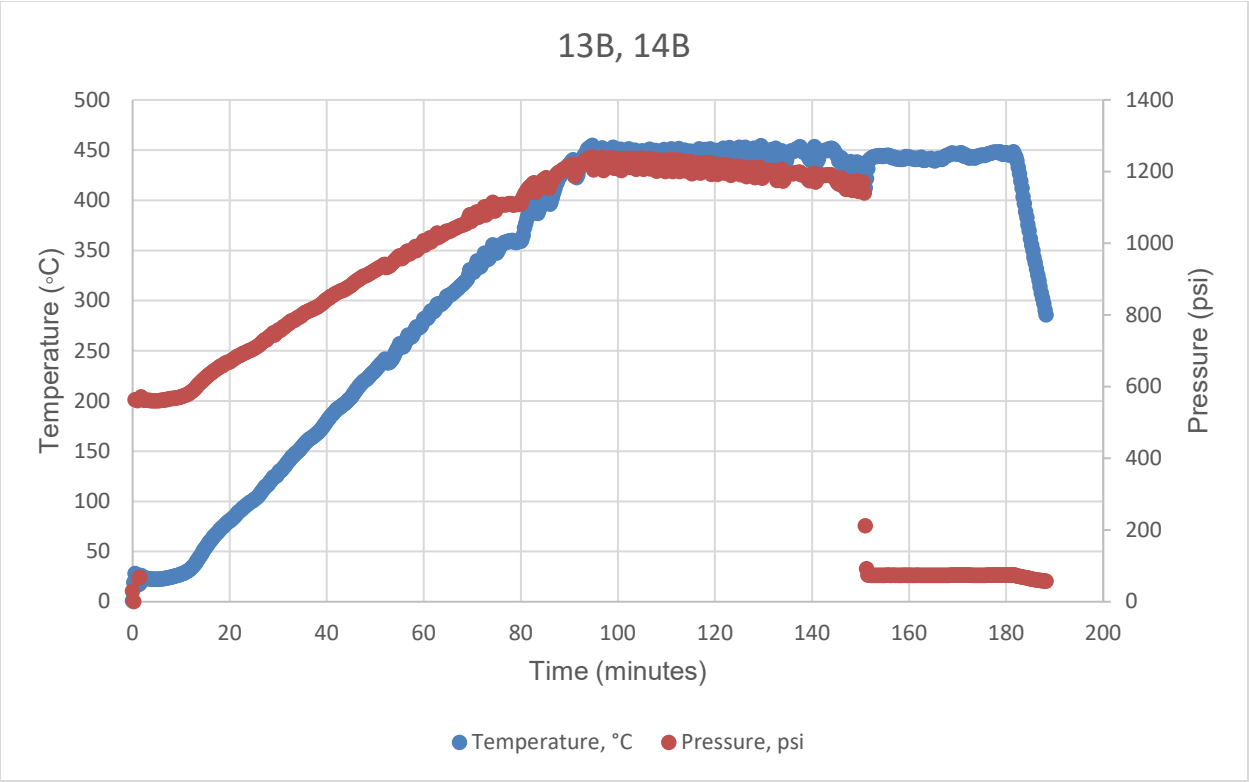
100
150
200
250
Temperature, °C
Pressure, psi


13B, 14B
Temperature (°C)
Pressure (psi)
Time (minutes)
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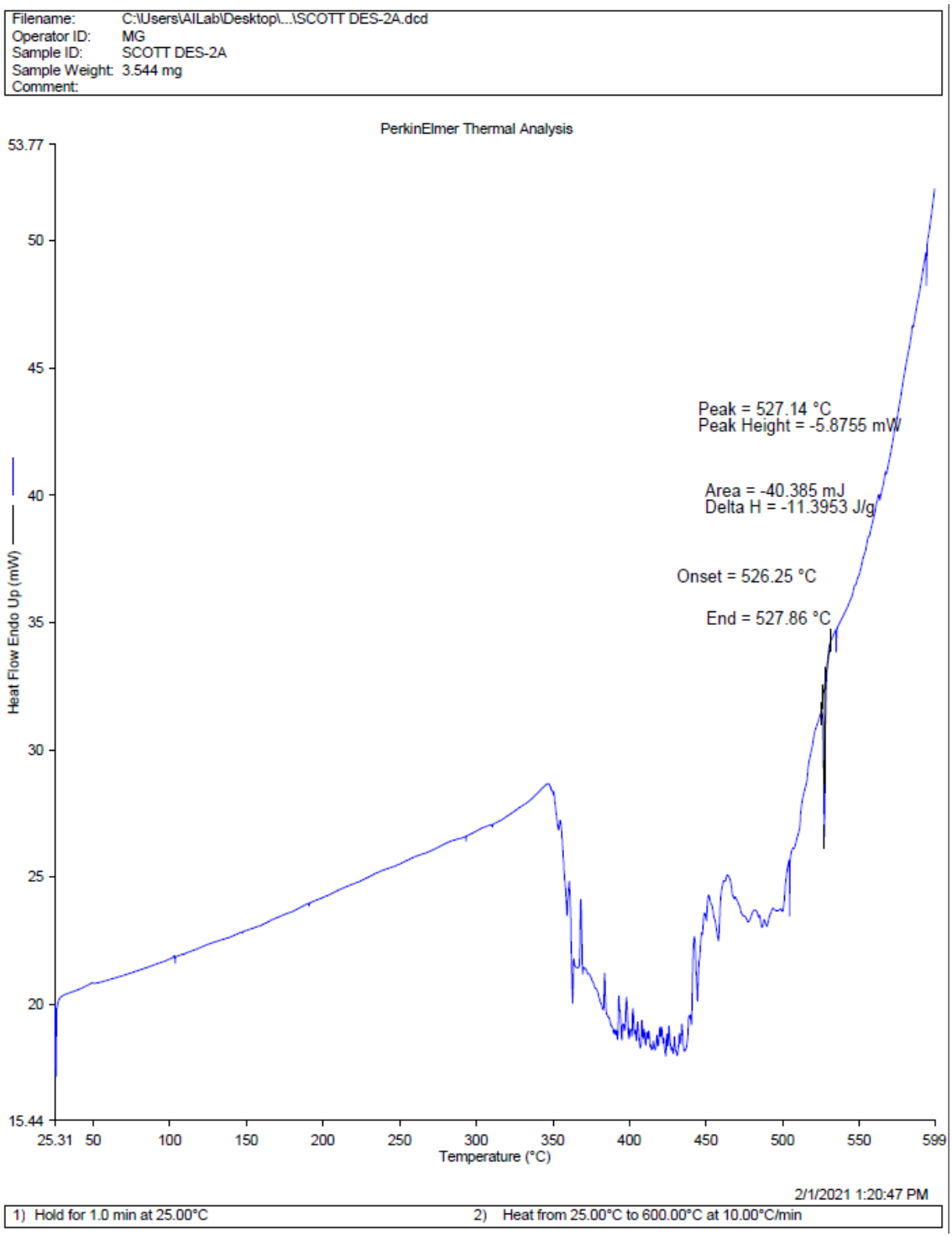
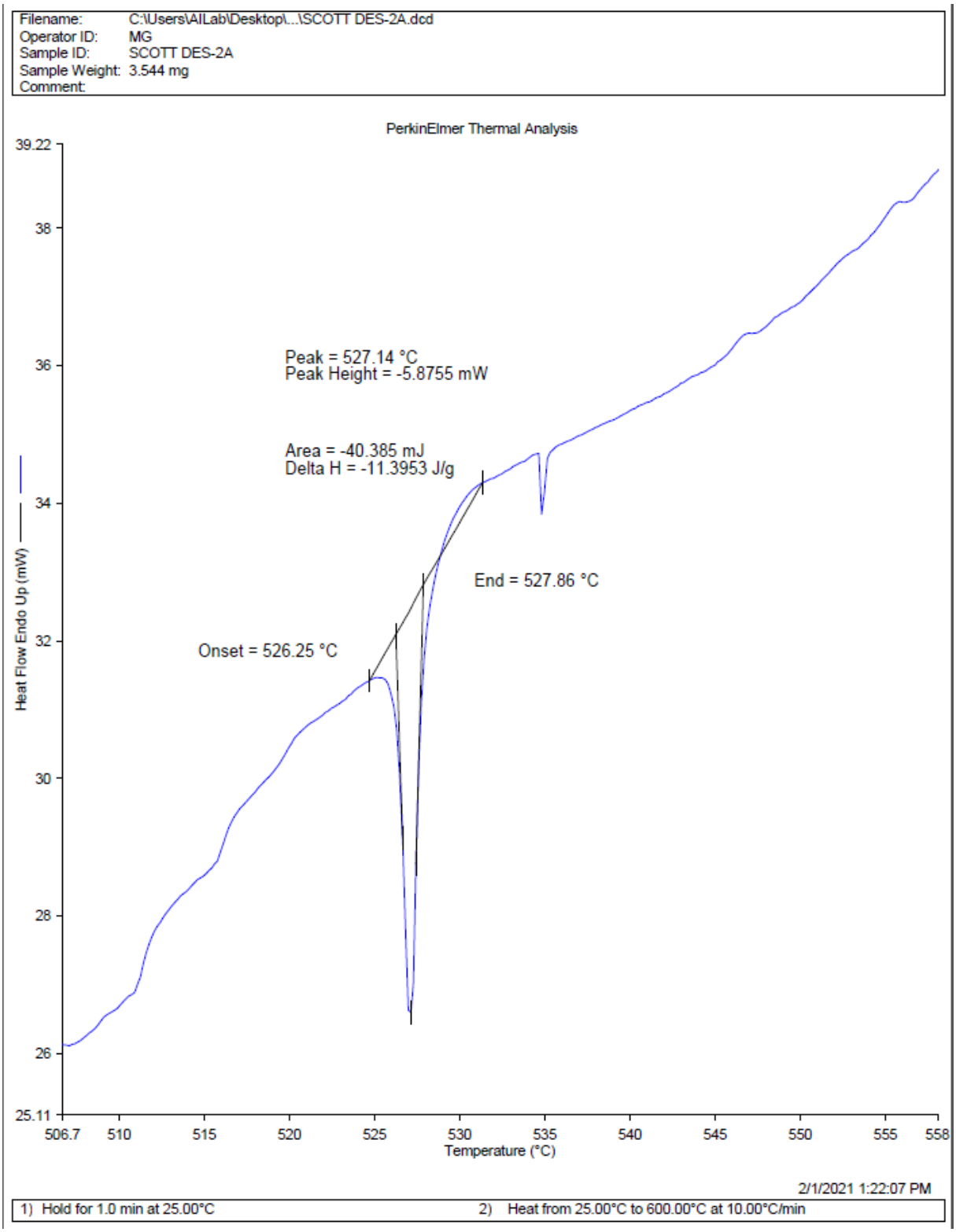
100
150
200
250
300
350
400
450
500
0
200
400
600
800
1000
1200
1400
0
20
40
60
80
100
120
140
160
180
200
Temperature, °C
Pressure, psi

APPENDIX II: DIFFERENTIAL SCANNING CALORIMETRY ANALYSIS RESULTS FOR PYROLYSIS TESTS



2A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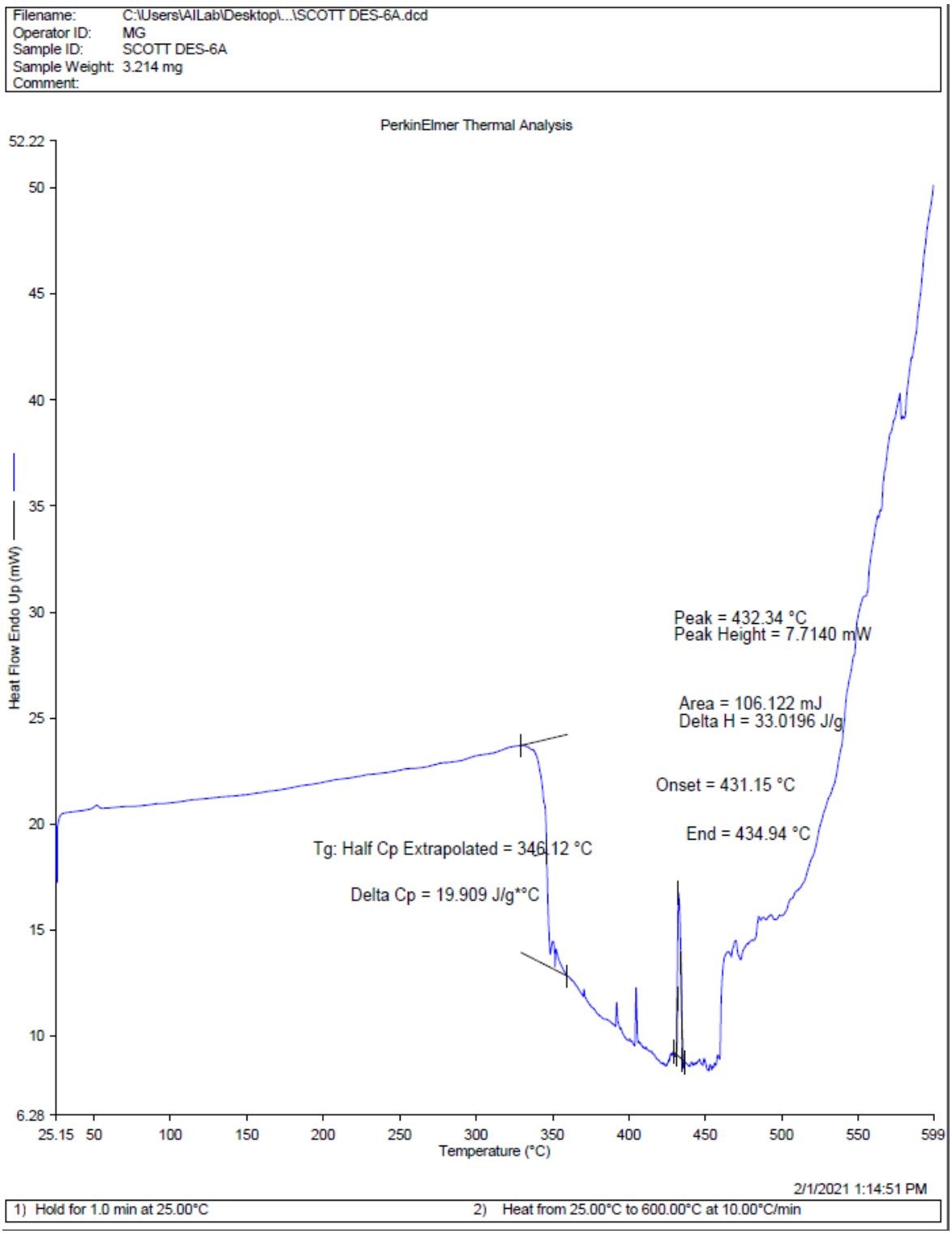
Operator ID: MG
Sample ID: SCOTT DES-2A
Sample Weight: 3.544 mg
Comment:
PerkinElmer Thermal Analysis
Peak = 527.14 °C
Peak Height = -5.8755 mW
Area = -40.385 mJ
Delta H = -11.3953 J/g
End = 527.86 °C
Onset = 526.25 °C
Heat Flow Endo Up (mW)
Temperature (°C)
39.22
38
36
34
32
30
28
26
25.11
506.7
510
515
520
525
530
535
540
545
550
555
558
2/1/2021 1:22:07 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

2A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Sample ID: SCOTT DES-6A
Sample Weight: 3.214 mg
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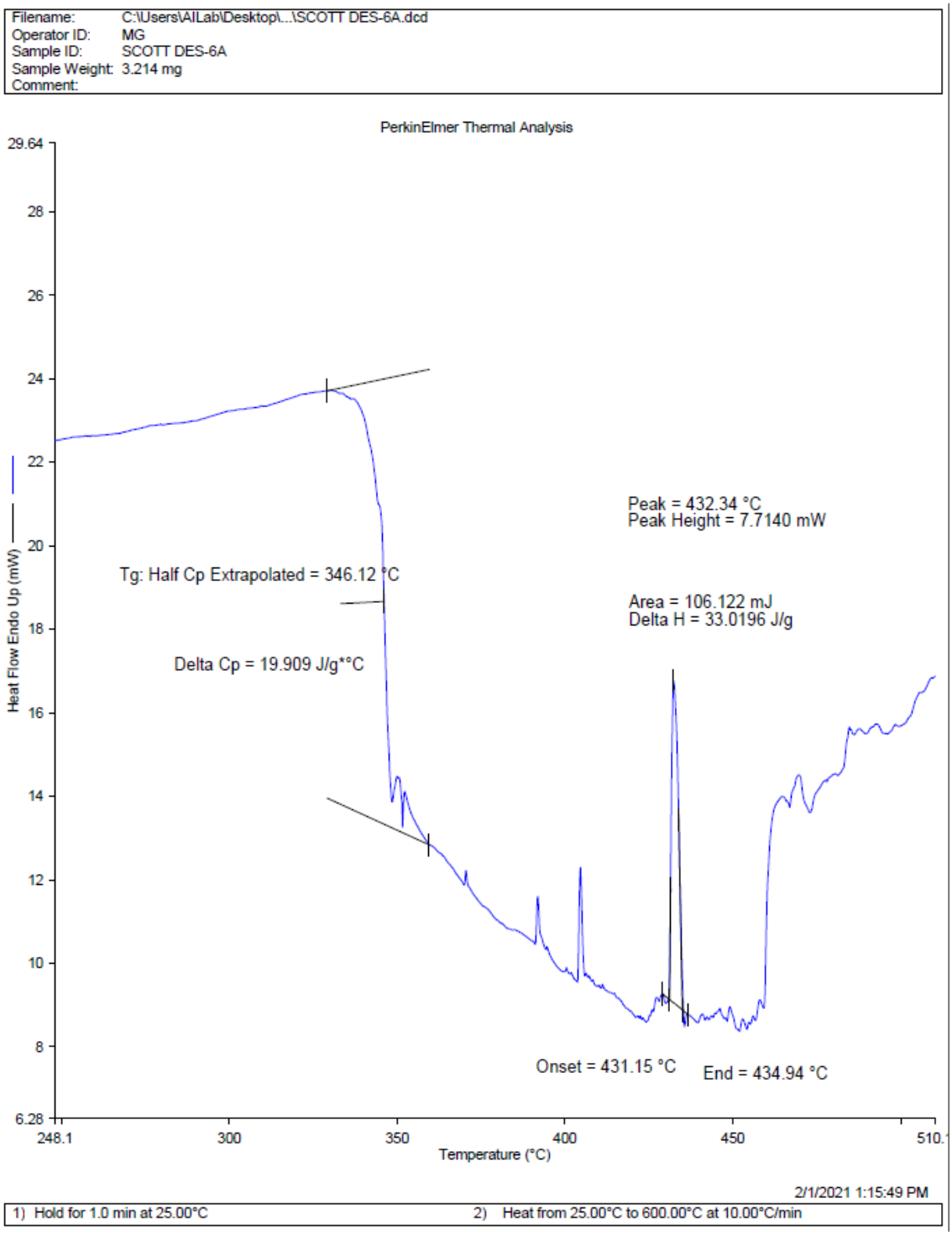
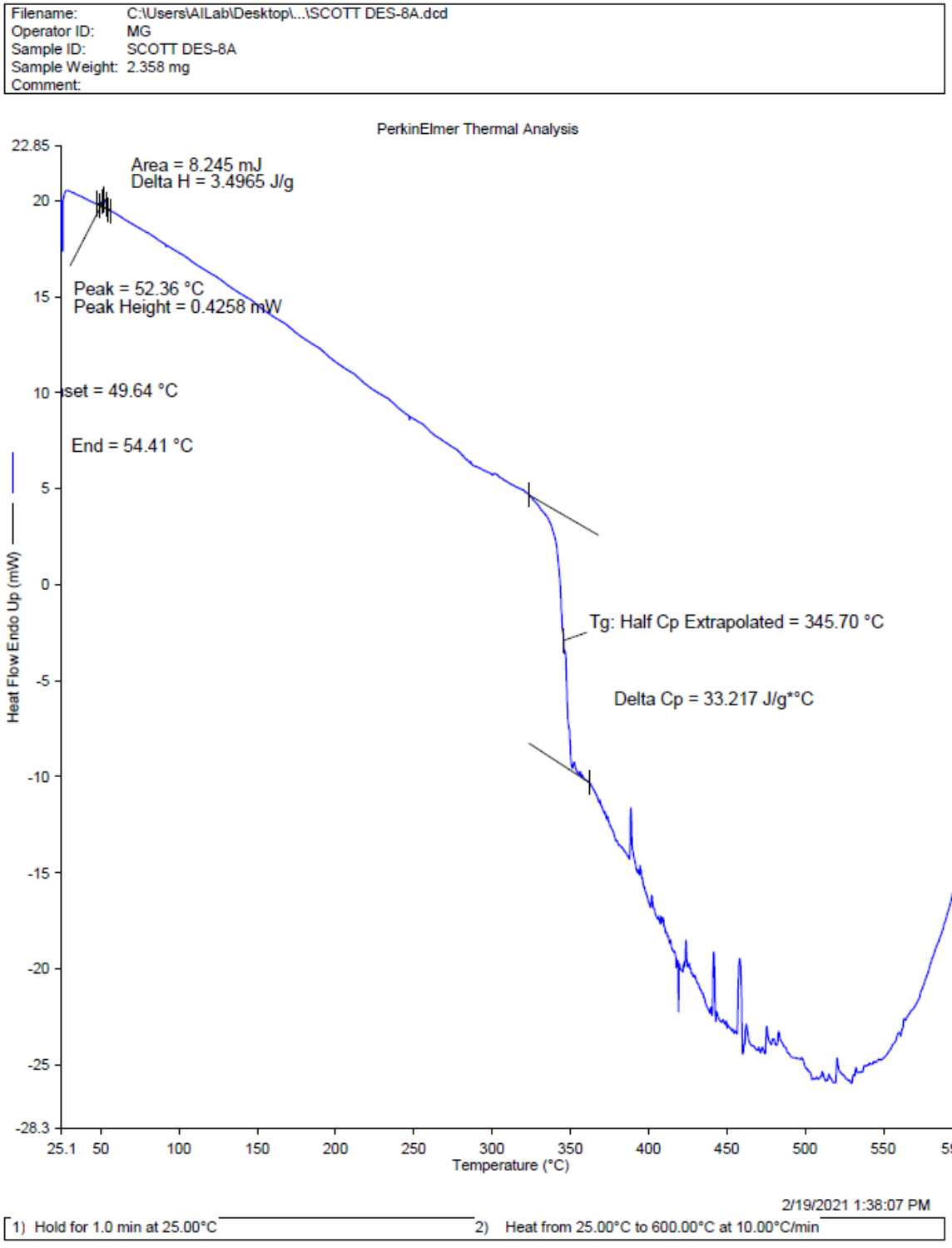
PerkinElmer Thermal Analysis
Heat Flow Endo Up (mW)
52.22
50
45
40
35
30
25
20
15
10
6.28
25.15
50
100
150
200
250
300
350
400
450
500
550
599
Temperature (°C)
Peak = 432.34 °C
Peak Height = 7.7140 mW
Area = 106.122 mJ
Delta H = 33.0196 J/g
Onset = 431.15 °C
End = 434.94 °C
Tg: Half Cp Extrapolated = 346.12 °C
Delta Cp = 19.909 J/g*°C
2/1/2021 1:14:51 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

6A


Filename: C:\Users\AILab\Desktop\...\SCOTT DES-6A.dcd
Operator ID: MG
Sample ID: SCOTT DES-6A
Sample Weight: 3.214 mg
Comment:
PerkinElmer Thermal Analysis
Heat Flow Endo Up (mW)
29.64
28
26
24
22
20
18
16
14
12
10
8
6.28
248.1
300
350
400
450
510.
Temperature (°C)
Peak = 432.34 °C
Peak Height = 7.7140 mW
Tg: Half Cp Extrapolated = 346.12 °C
Area = 106.122 mJ
Delta H = 33.0196 J/g
Delta Cp = 19.909 J/g*°C
Onset = 431.15 °C
End = 434.94 °C
2/1/2021 1:15:49 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

6A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Operator ID: MG
Sample ID: SCOTT DES-8A
Sample Weight: 2.358 mg
Comment:
PerkinElmer Thermal Analysis
Area = 8.245 mJ
Delta H = 3.4965 J/g
Peak = 52.36 °C
Peak Height = 0.4258 mW
set = 49.64 °C
End = 54.41 °C
Tg: Half Cp Extrapolated = 345.70 °C
Delta Cp = 33.217 J/g*°C
Heat Flow Endo Up (mW)
Temperature (°C)
2/19/2021 1:38:07 PM
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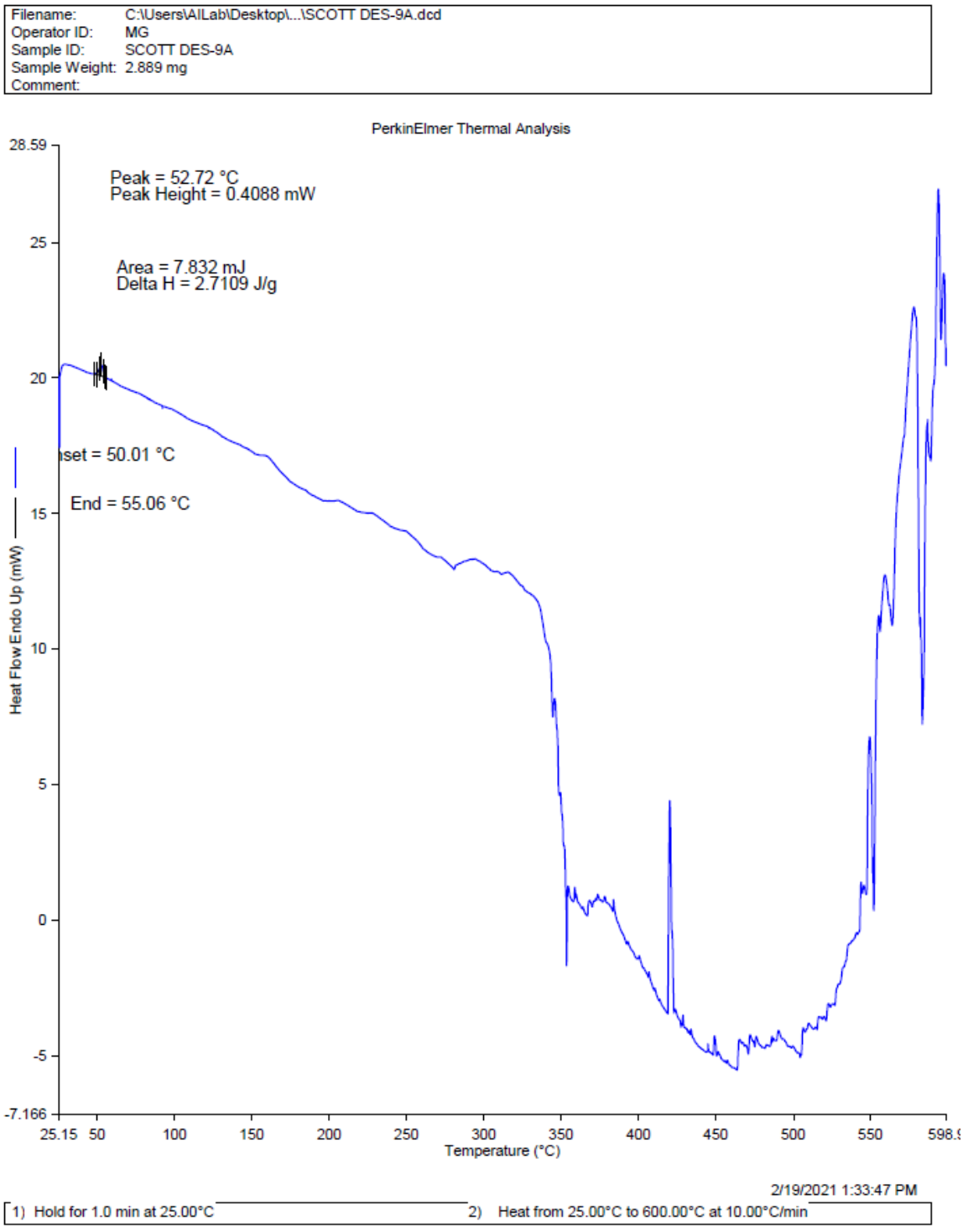
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

8A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PerkinElmer Thermal Analysis
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Peak Height = 0.4088 mW
Area = 7.832 mJ
Delta H = 2.7109 J/g
set = 50.01 °C
End = 55.06 °C
Heat Flow Endo Up (mW)
Temperature (°C)
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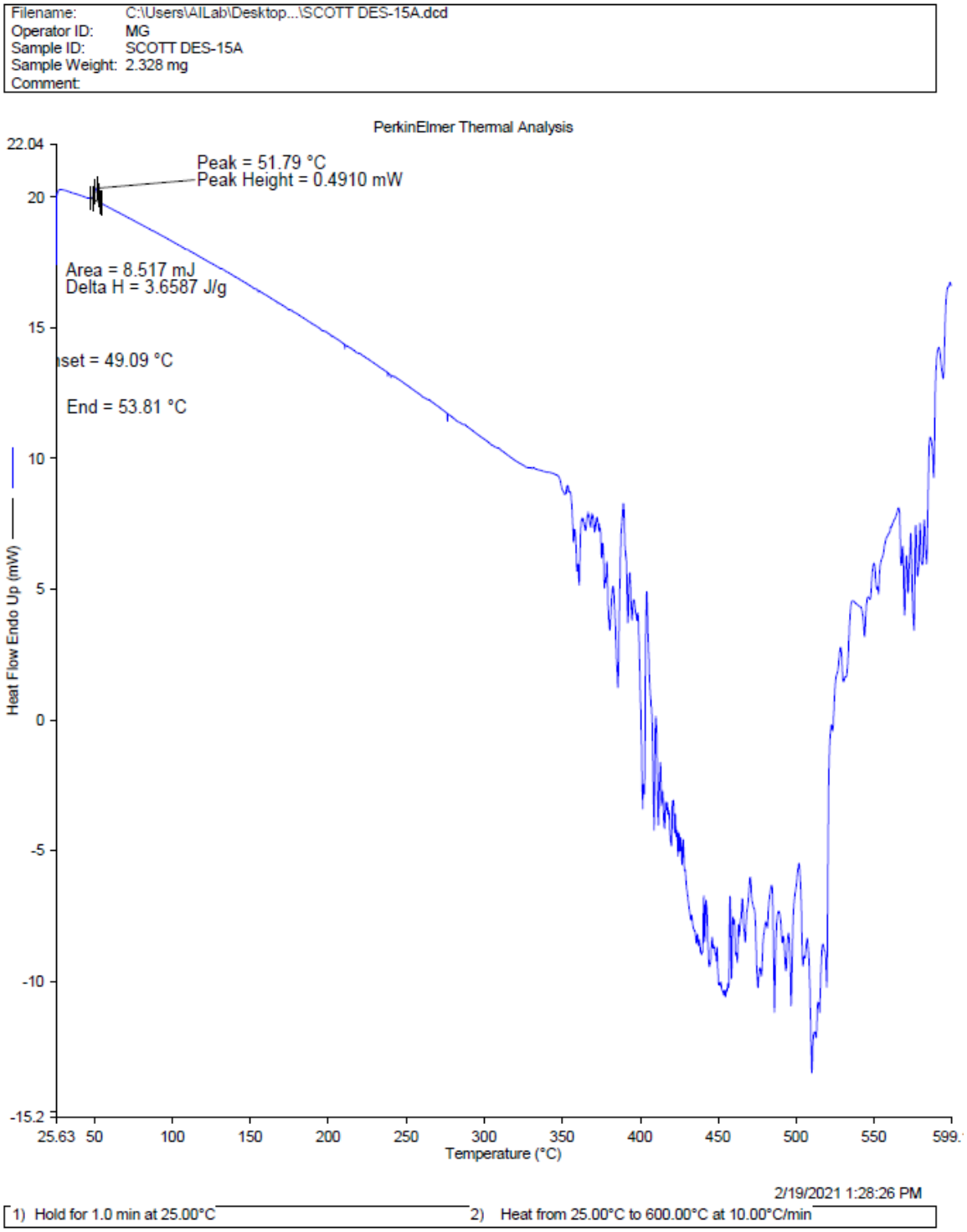
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

9A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Sample Weight: 2.328 mg
Comment:
PerkinElmer Thermal Analysis
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Peak Height = 0.4910 mW
Area = 8.517 mJ
Delta H = 3.6587 J/g
set = 49.09 °C
End = 53.81 °C
Heat Flow Endo Up (mW)
Temperature (°C)
2/19/2021 1:28:26 PM
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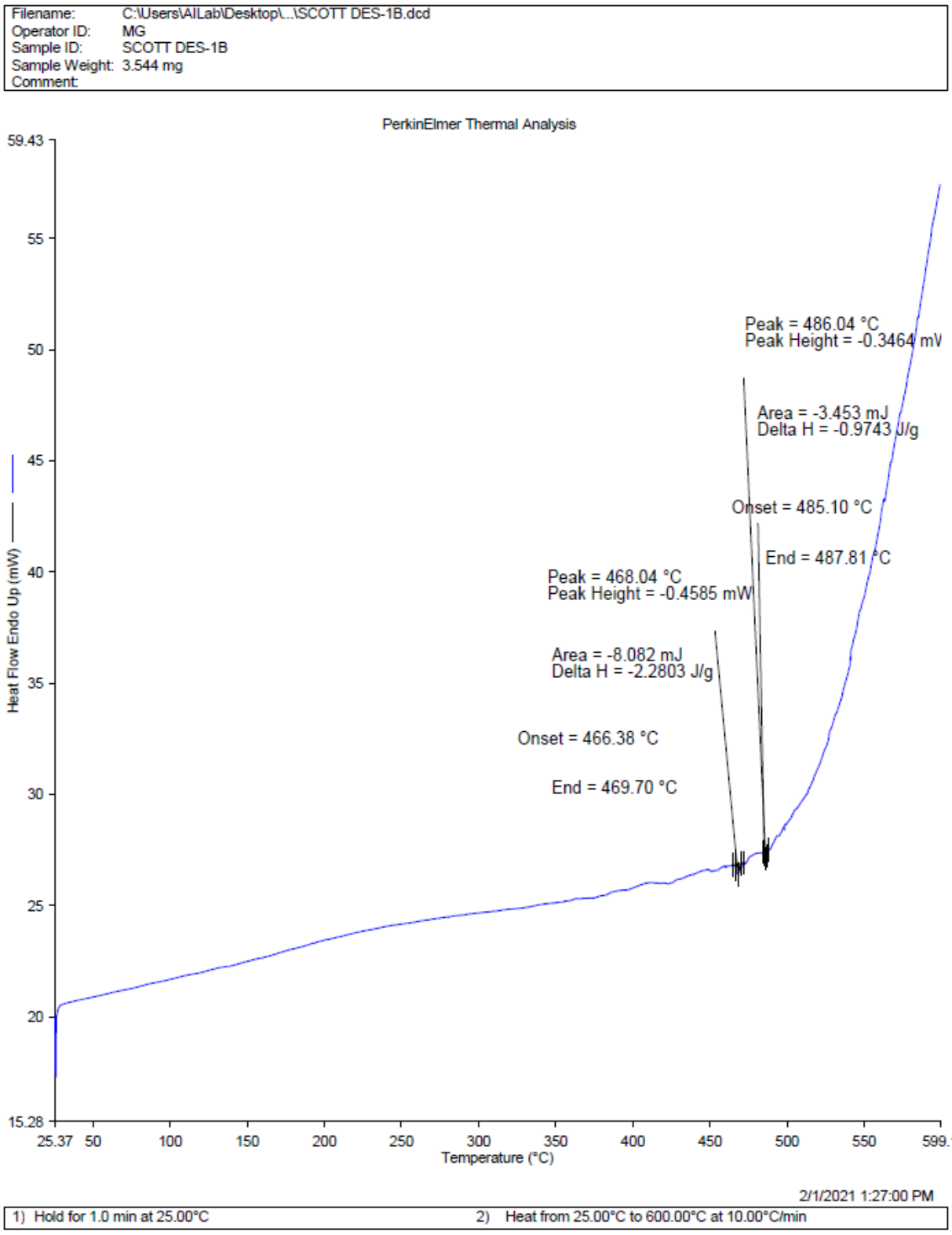
15A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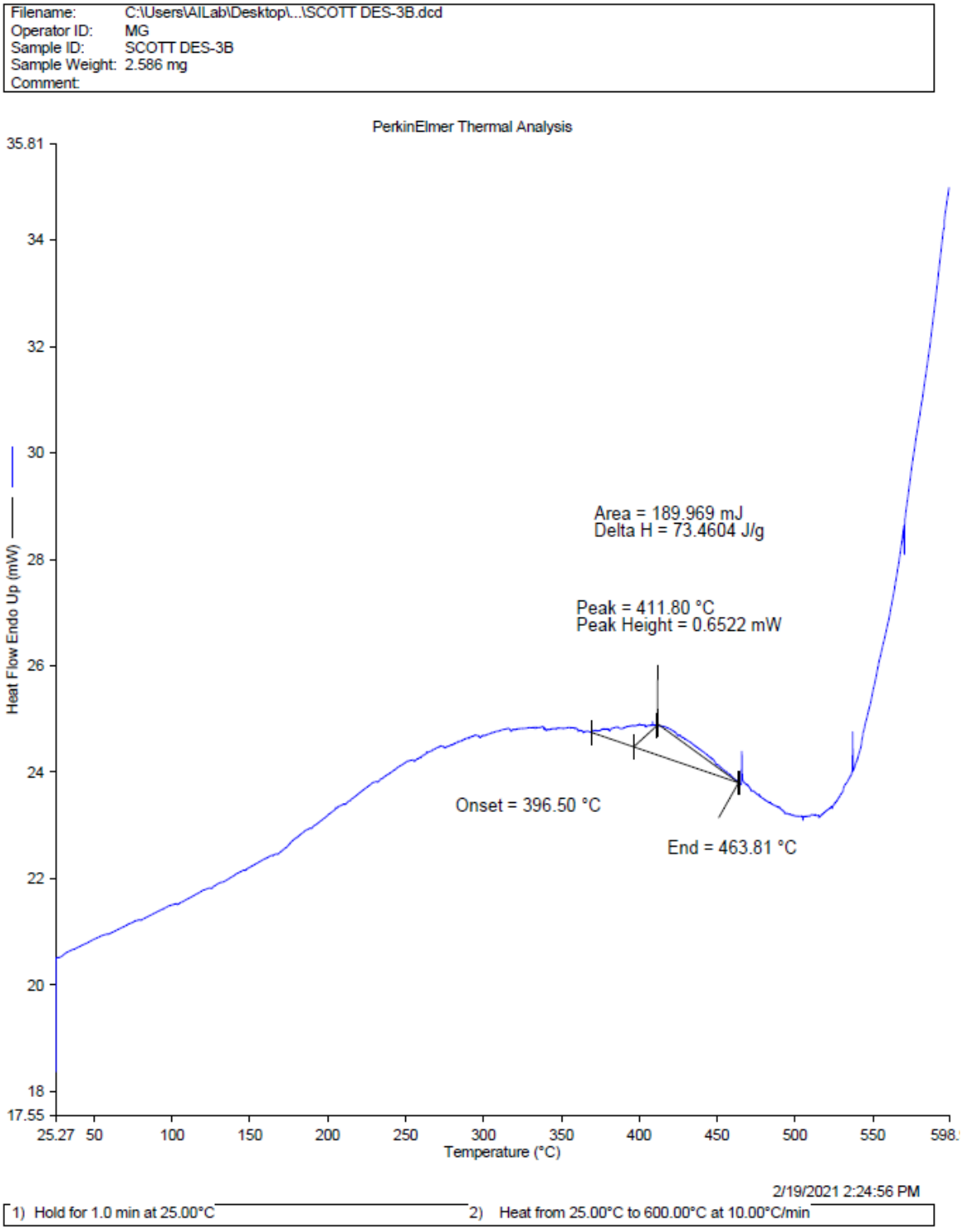
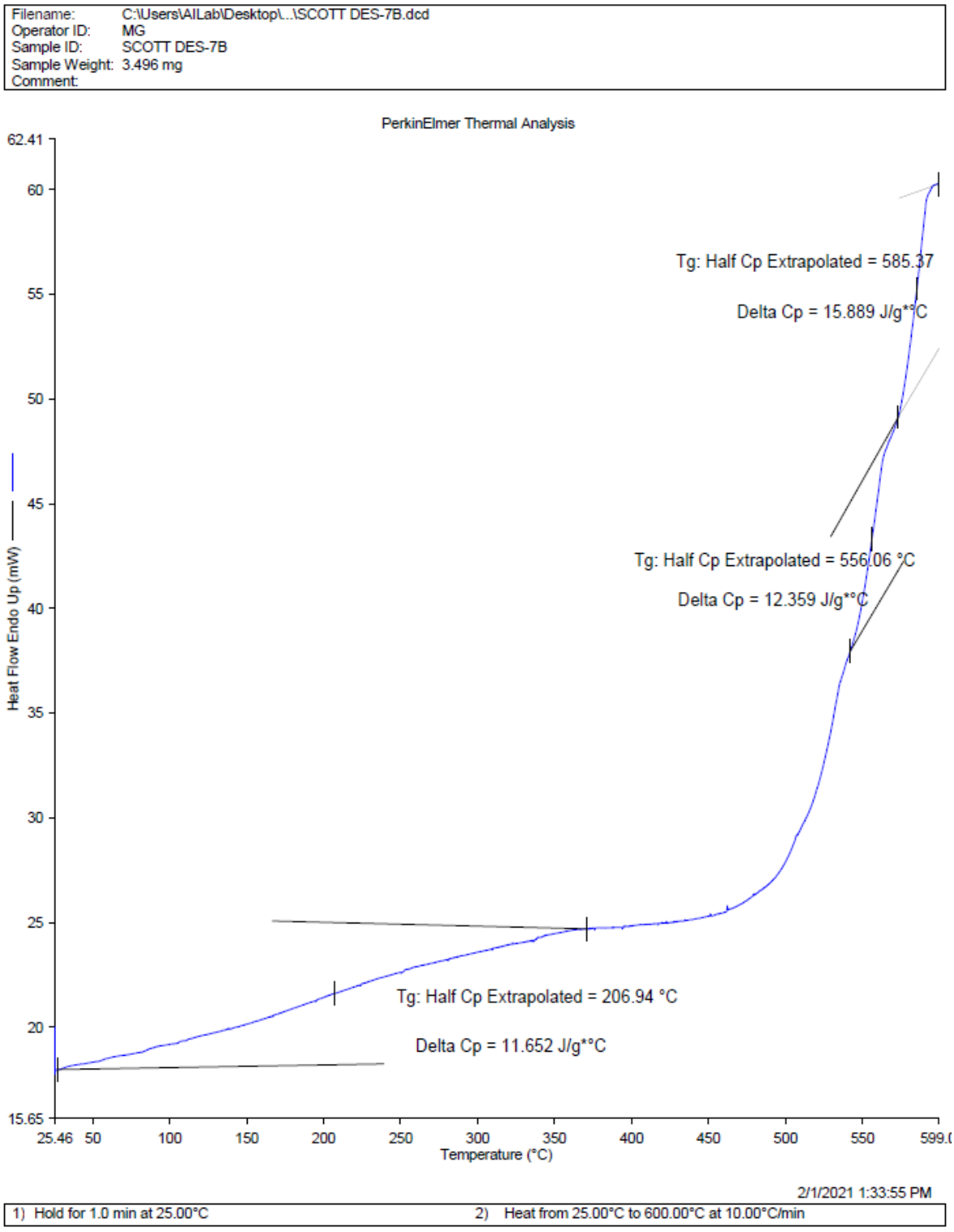
Sample ID: SCOTT DES-1B
Sample Weight: 3.544 mg
Comment:
PerkinElmer Thermal Analysis
Heat Flow Endo Up (mW)
Temperature (°C)
Peak = 486.04 °C
Peak Height = -0.3464 mW
Area = -3.453 mJ
Delta H = -0.9743 J/g
Onset = 485.10 °C
End = 487.81 °C
Peak = 468.04 °C
Peak Height = -0.4585 mW
Area = -8.082 mJ
Delta H = -2.2803 J/g
Onset = 466.38 °C
End = 469.70 °C
2/1/2021 1:27:00 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

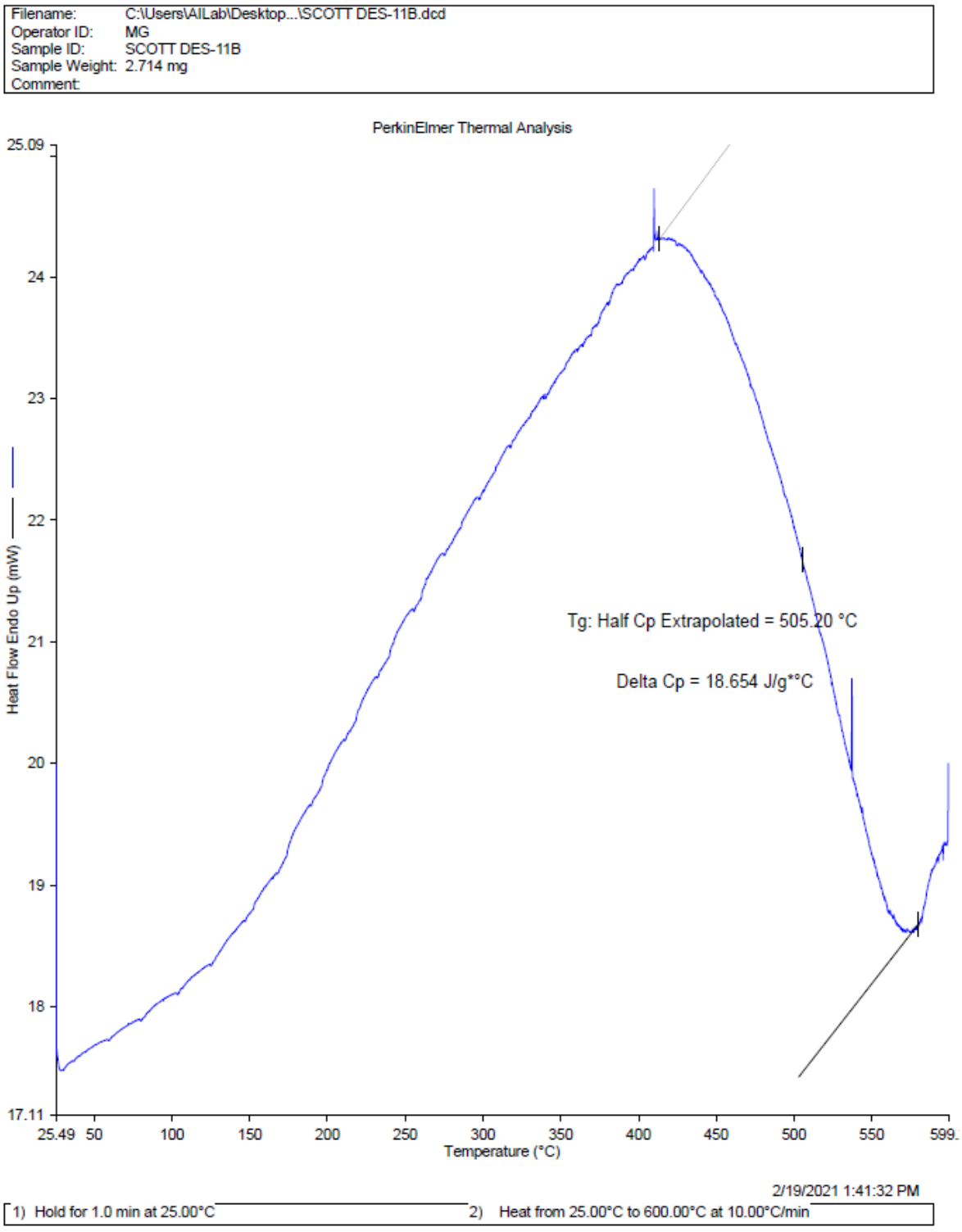
1B


Filename: C:\Users\AILab\Desktop\...\SCOTT DES-3B.dcd
Operator ID: MG
Sample ID: SCOTT DES-3B
Sample Weight: 2.586 mg
Comment:
PerkinElmer Thermal Analysis
Heat Flow Endo Up (mW)
Temperature (°C)
Area = 189.969 mJ
Delta H = 73.4604 J/g
Peak = 411.80 °C
Peak Height = 0.6522 mW
Onset = 396.50 °C
End = 463.81 °C
2/19/2021 2:24:56 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

3B

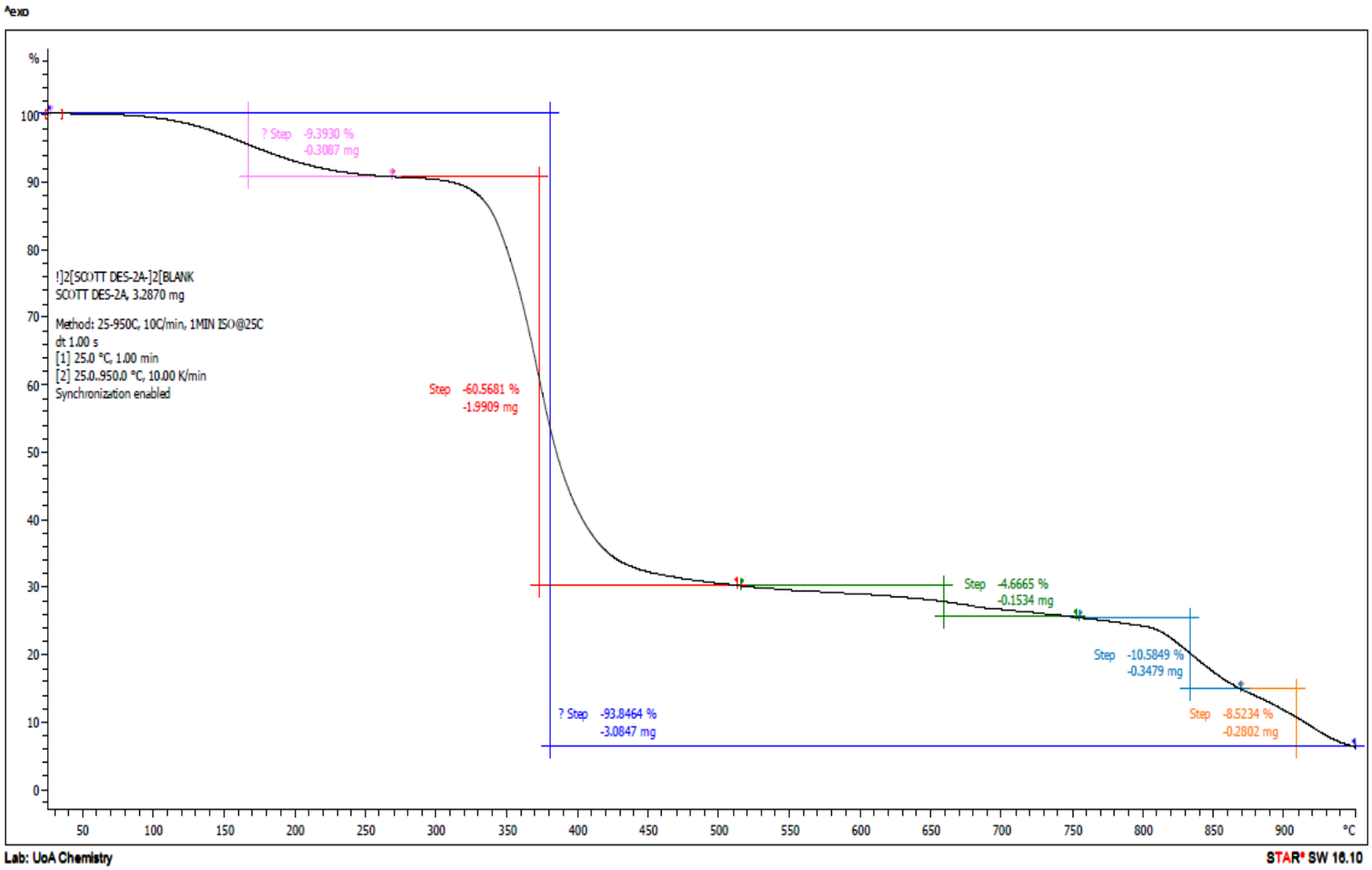

Filename: C:\Users\AILab\Desktop\...\SCOTT DES-7B.dcd
Operator ID: MG
Sample ID: SCOTT DES-7B
Sample Weight: 3.496 mg
Comment:
PerkinElmer Thermal Analysis
Tg: Half Cp Extrapolated = 585.37
Delta Cp = 15.889 J/g*°C
Tg: Half Cp Extrapolated = 556.06 °C
Delta Cp = 12.359 J/g*°C
Tg: Half Cp Extrapolated = 206.94 °C
Delta Cp = 11.652 J/g*°C
Heat Flow Endo Up (mW)
Temperature (°C)
2/1/2021 1:33:55 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

7B

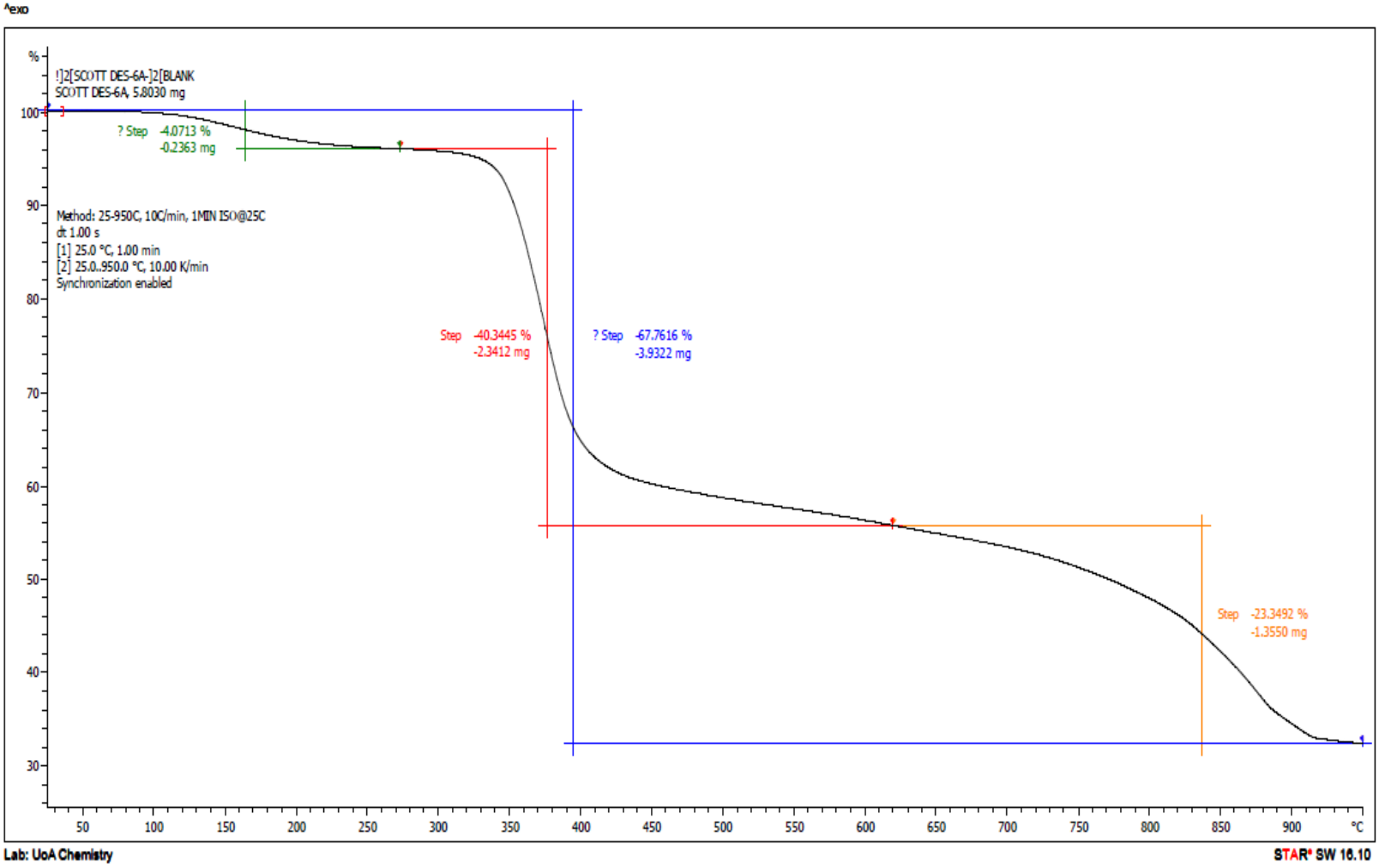

Filename: C:\Users\AILab\Desktop...\SCOTT DES-11B.dcd
Operator ID: MG
Sample ID: SCOTT DES-11B
Sample Weight: 2.714 mg
Comment:
PerkinElmer Thermal Analysis
Heat Flow Endo Up (mW)
25.09
24
23
22
21
20
19
18
17.11
25.49 50 100 150 200 250 300 350 400 450 500 550 599.
Temperature (°C)
Tg: Half Cp Extrapolated = 505.20 °C
Delta Cp = 18.654 J/g*°C
2/19/2021 1:41:32 PM
1) Hold for 1.0 min at 25.00°C
2) Heat from 25.00°C to 600.00°C at 10.00°C/min

11B

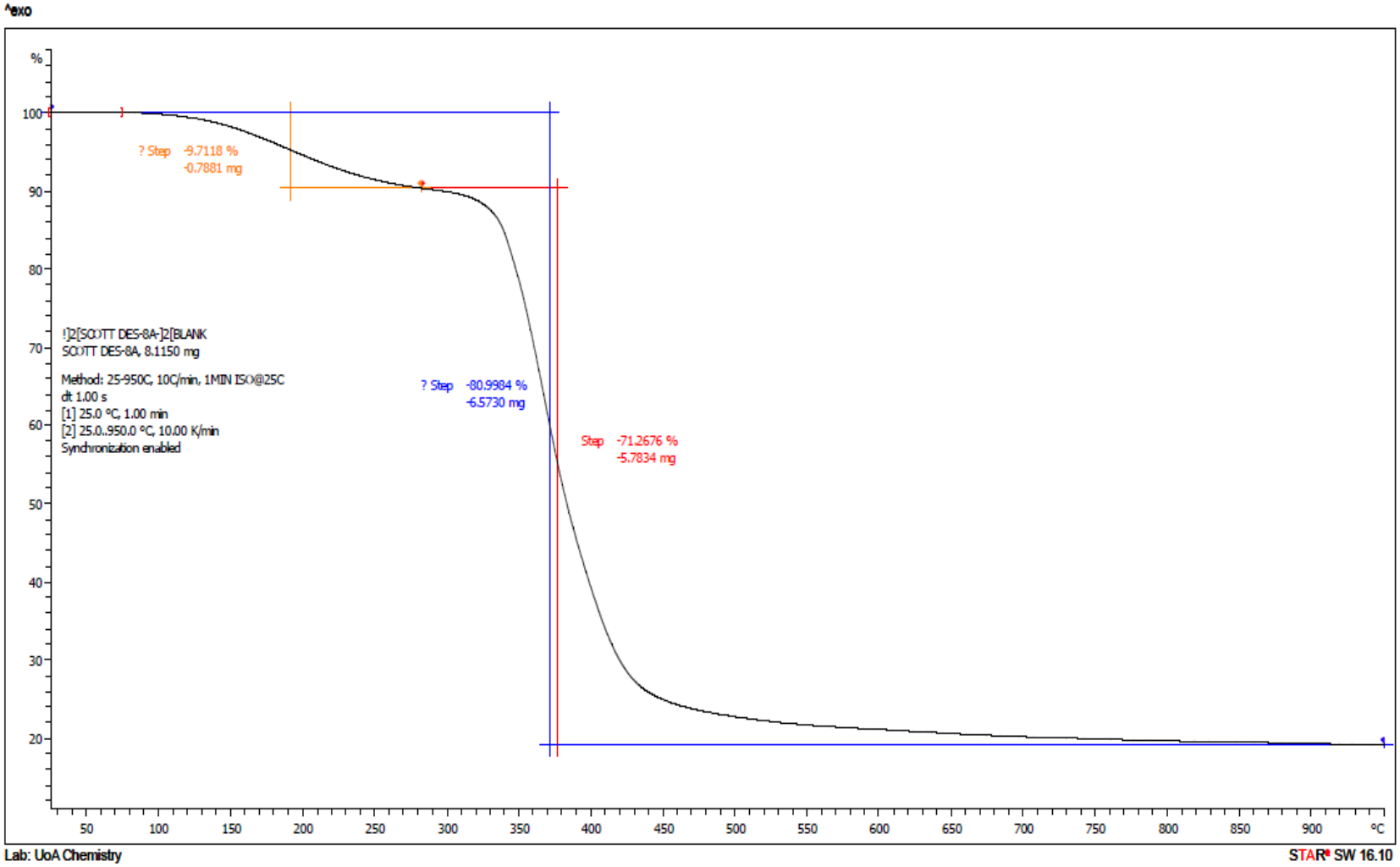
APPENDIX III: THERMOGRAVIMETRIC ANALYSIS RESULTS



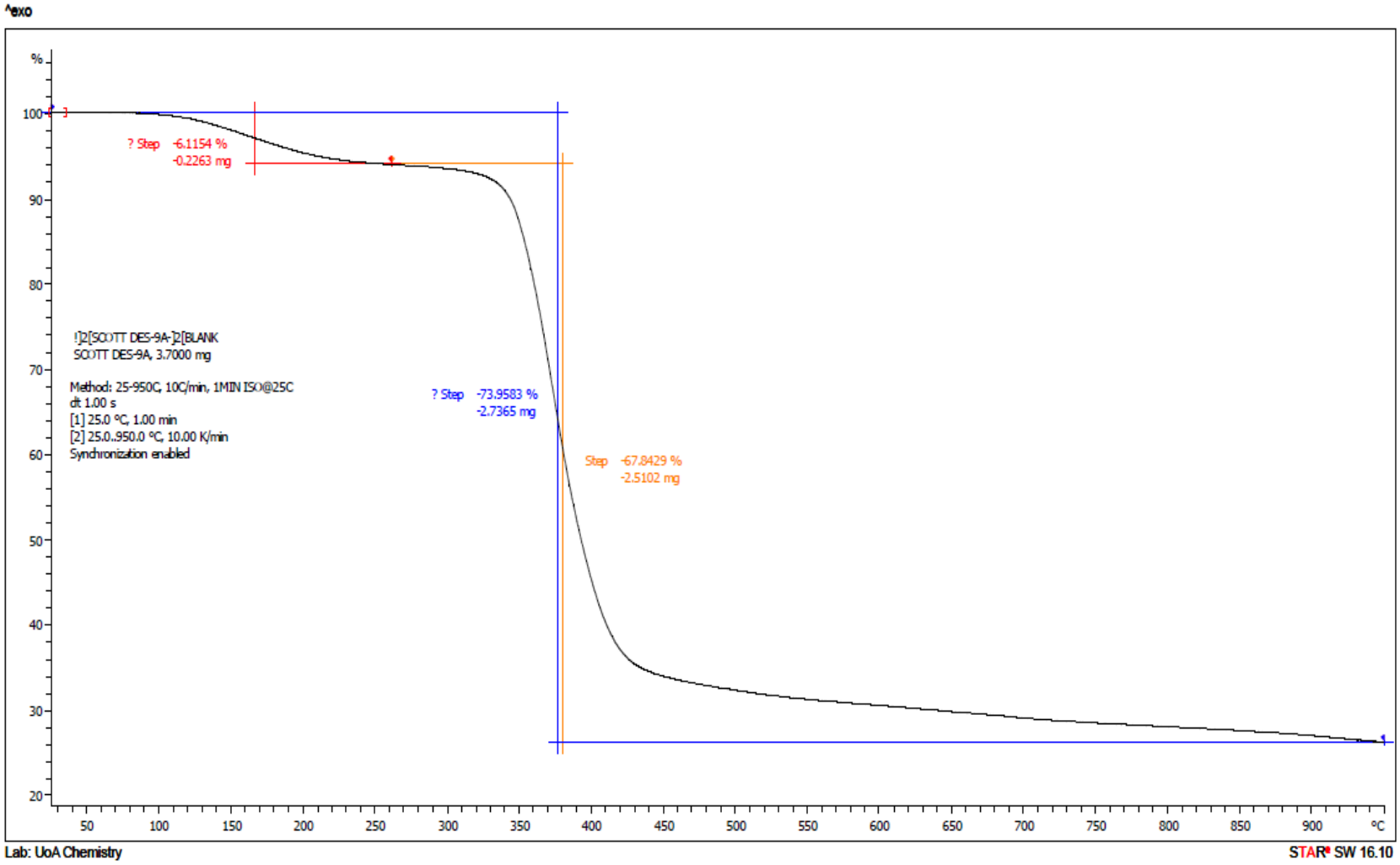
2A


^exo
%
!]2[SCOTT DES-6A-]2[BLANK
SCOTT DES-6A, 5.8030 mg
? Step -4.0713 %
-0.2363 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
Step -40.3445 %
-2.3412 mg
? Step -67.7616 %
-3.9322 mg
Step -23.3492 %
-1.3550 mg
100
90
80
70
60
50
40
30
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STARe SW 16.10

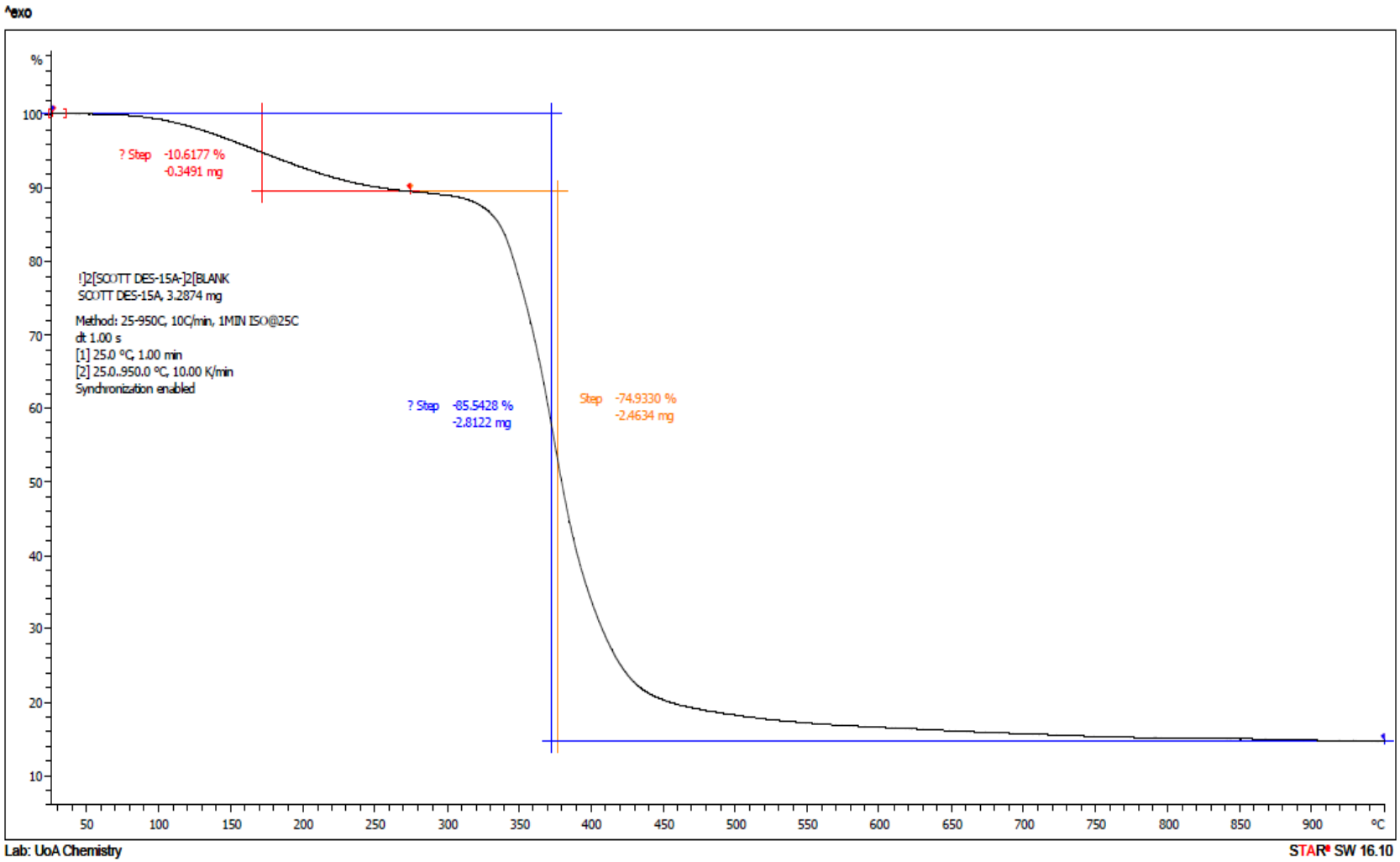
6A


^exo
%
100
90
80
70
60
50
40
30
20
? Step -9.7118 %
-0.7881 mg
!]2[SCOTT DES-8A-]2[BLANK
SCOTT DES-8A, 8.1150 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
? Step -80.9984 %
-6.5730 mg
Step -71.2676 %
-5.7834 mg
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STARe SW 16.10

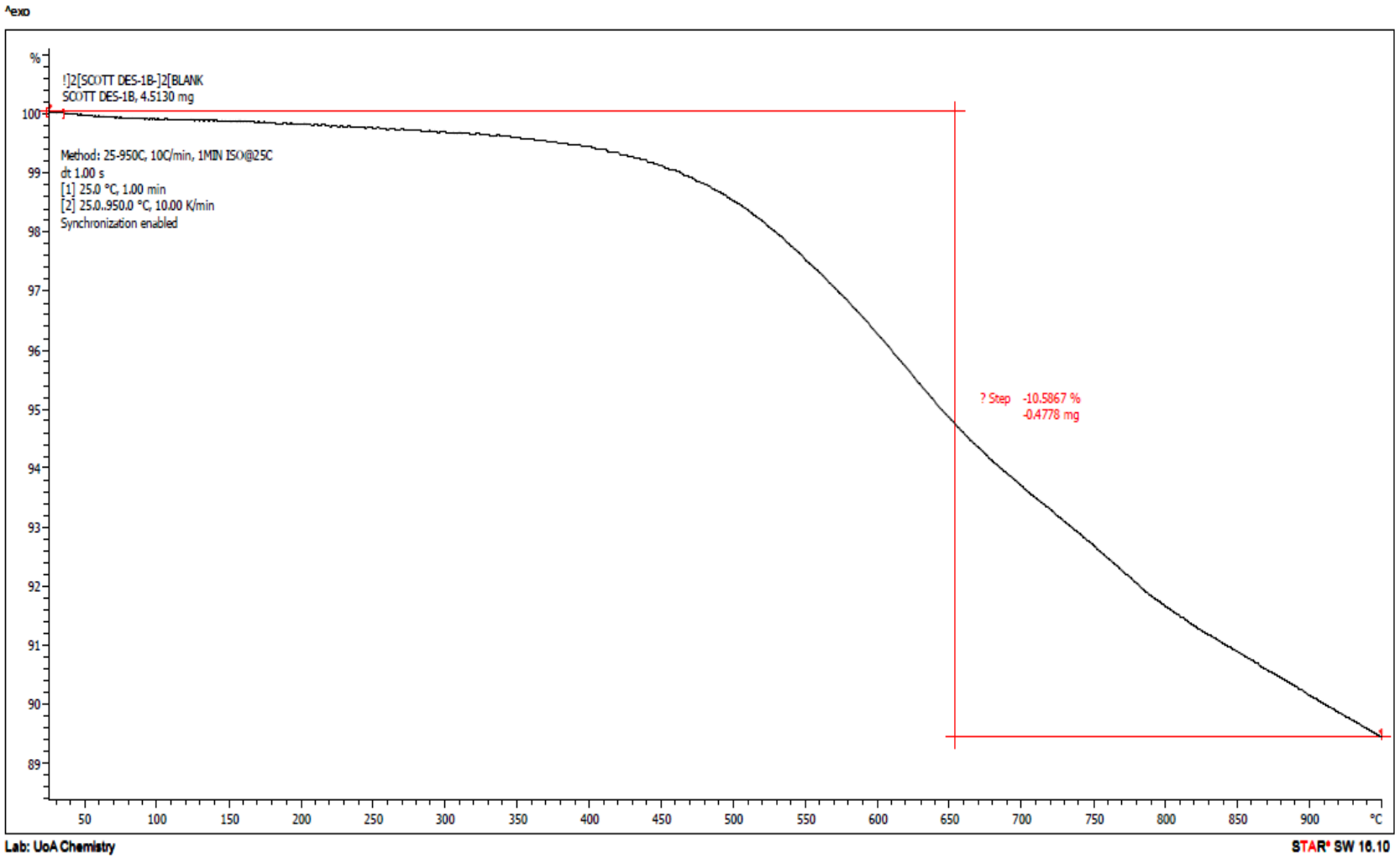
8A


^exo
%
100
90
80
70
60
50
40
30
20
? Step -6.1154 %
-0.2263 mg
!]2[SCOTT DES-9A-]2[BLANK
SCOTT DES-9A, 3.7000 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
? Step -73.9583 %
-2.7365 mg
Step -67.8429 %
-2.5102 mg
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STARe SW 16.10

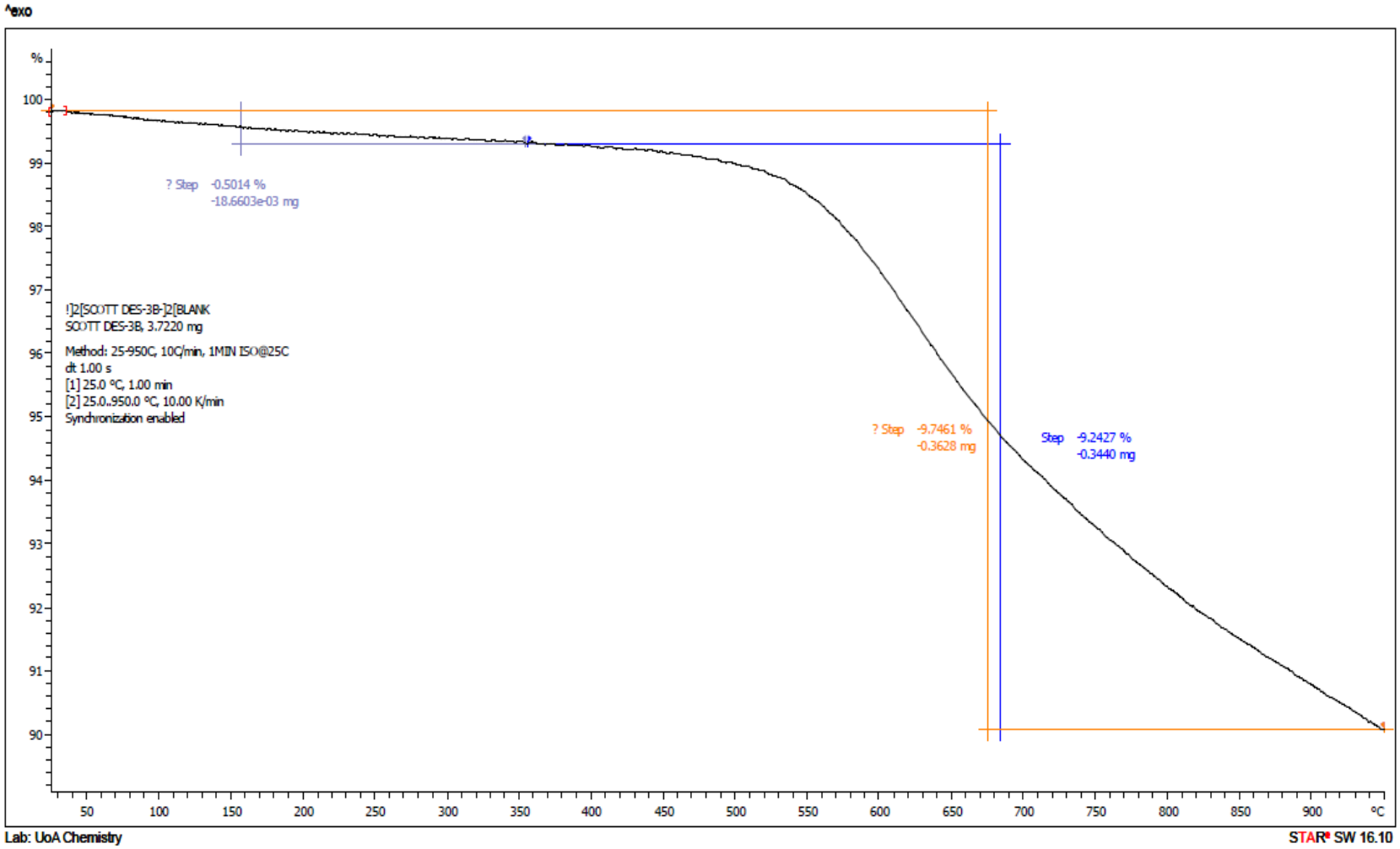
9A


^exo
%
100
90
80
70
60
50
40
30
20
10
? Step -10.6177 %
-0.3491 mg
!]2[SCOTT DES-15A-]2[BLANK
SCOTT DES-15A, 3.2874 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
? Step -65.5428 %
-2.8122 mg
Step -74.9330 %
-2.4634 mg
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STARe SW 16.10

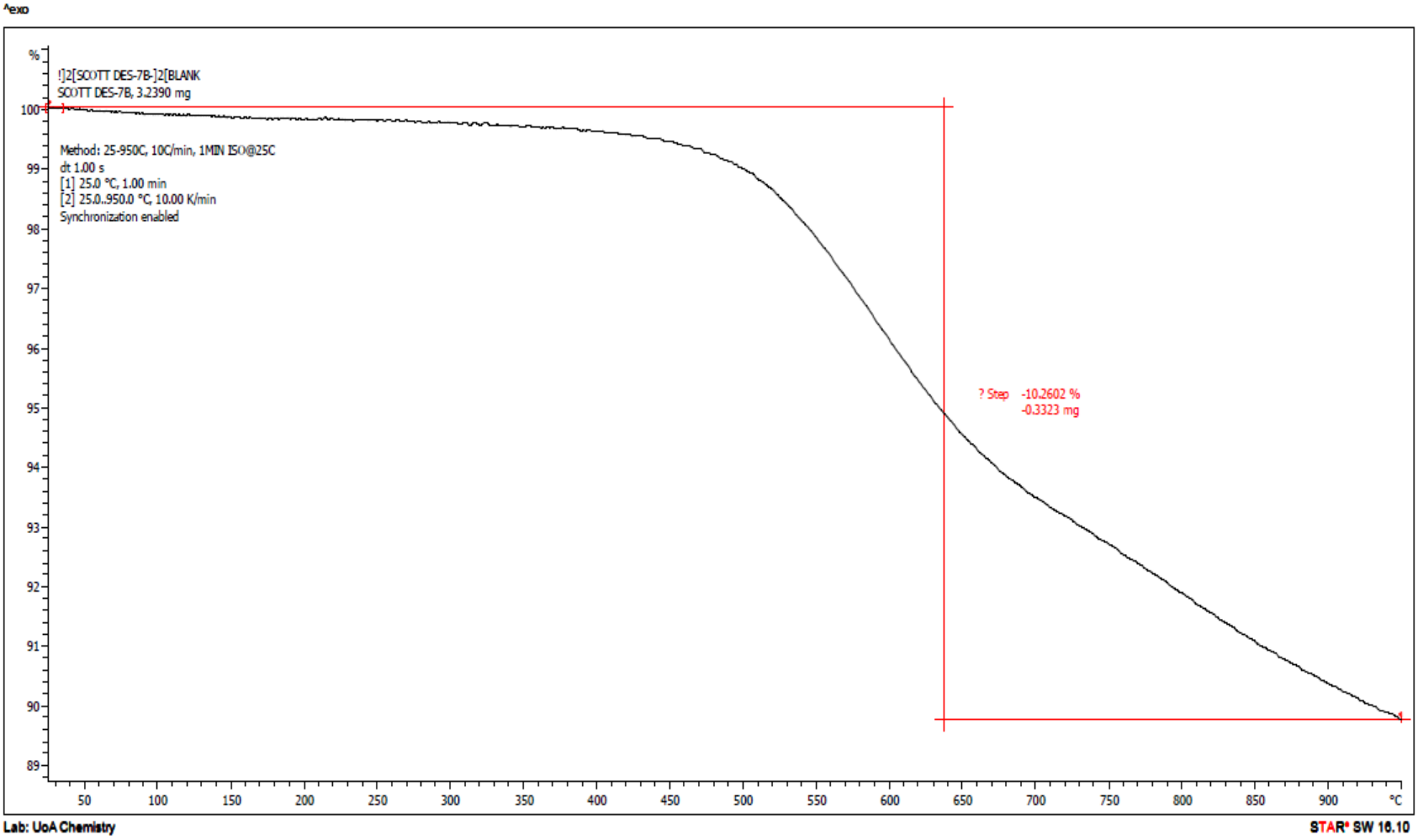
15A


^exo
%
!]2[SCOTT DES-1B-]2[BLANK
SCOTT DES-1B, 4.5130 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
? Step -10.5867 %
-0.4778 mg
100
99
98
97
96
95
94
93
92
91
90
89
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STAR° SW 16.10

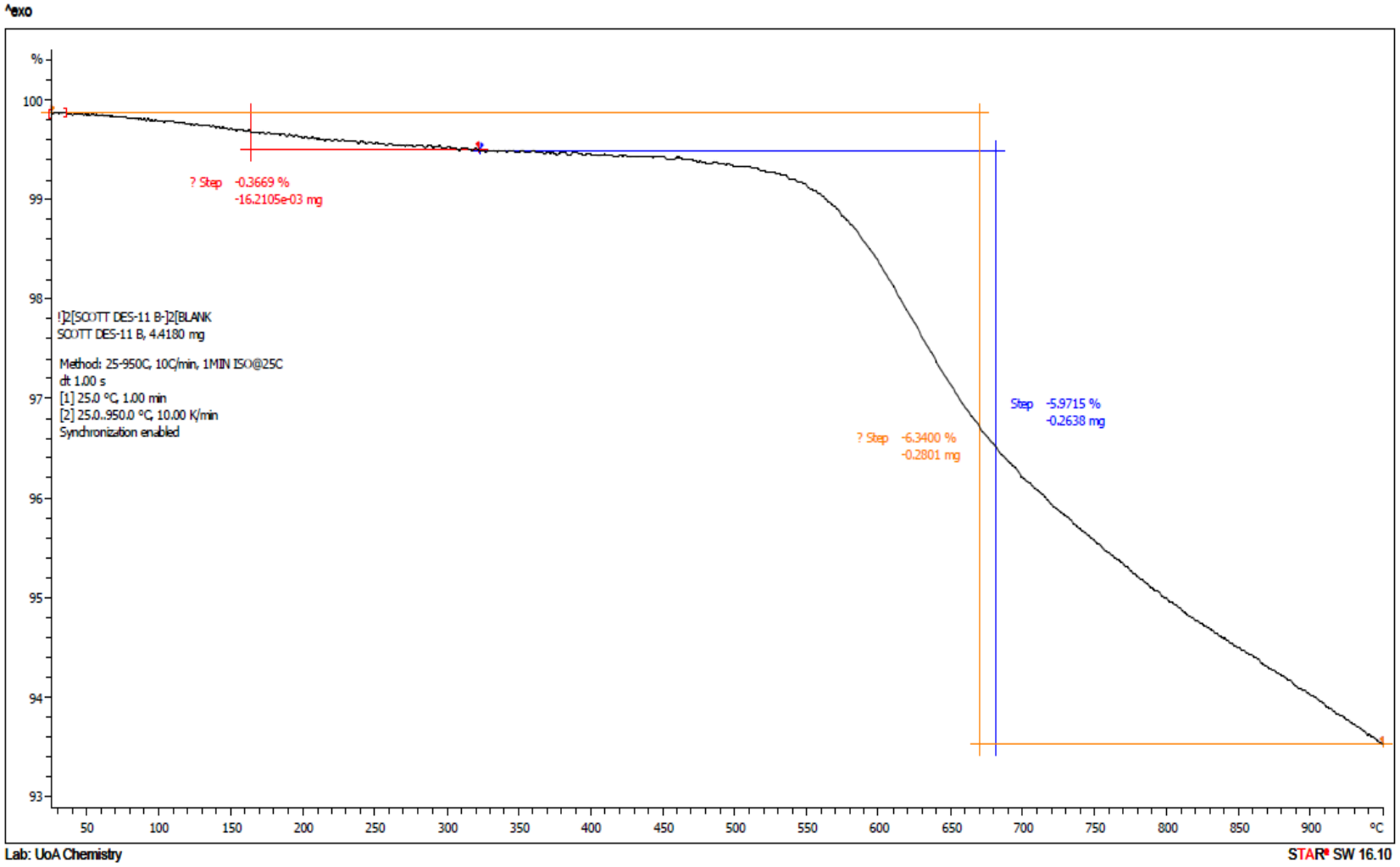
1B


^exo
%
100
99
98
97
96
95
94
93
92
91
90
? Step -0.5014 %
-18.6603e-03 mg
!]2[SCOTT DES-3B-]2[BLANK
SCOTT DES-3B, 3.7220 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
? Step -9.7461 %
-0.3628 mg
Step -9.2427 %
-0.3440 mg
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STARe SW 16.10

3B


^exo
%
!]2[SCOTT DES-7B-]2[BLANK
SCOTT DES-7B, 3.2390 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
? Step -10.2602 %
-0.3323 mg
100
99
98
97
96
95
94
93
92
91
90
89
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STARe SW 16.10

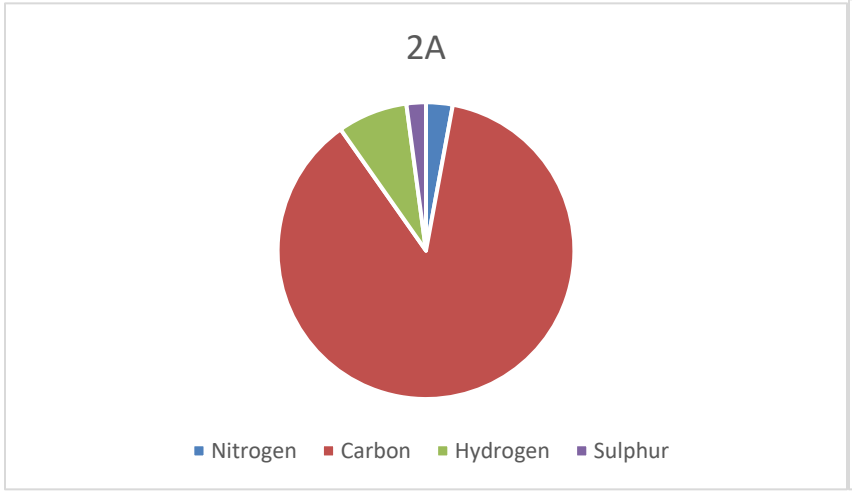
7B

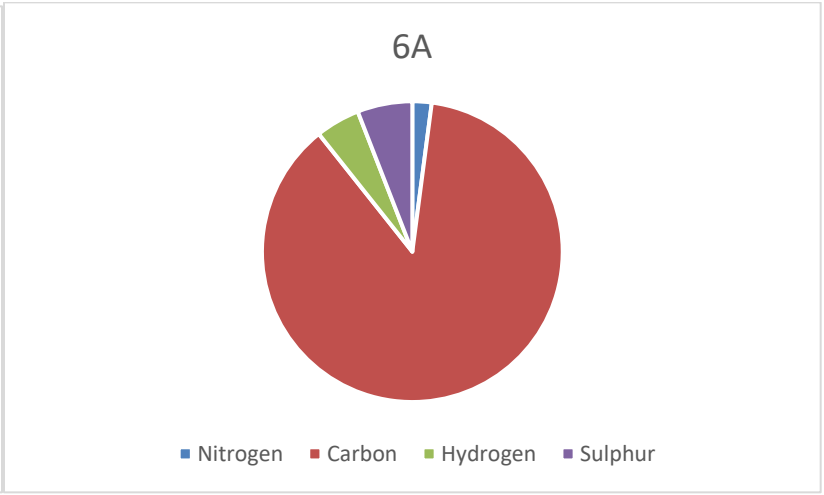

^exo
%
100
99
98
97
96
95
94
93
? Step -0.3669 %
-16.2105e-03 mg
!2[SCOTT DES-11 B-]2[BLANK
SCOTT DES-11 B, 4.4180 mg
Method: 25-950C, 10C/min, 1MIN ISO@25C
dt 1.00 s
[1] 25.0 °C, 1.00 min
[2] 25.0..950.0 °C, 10.00 K/min
Synchronization enabled
Step -5.9715 %
-0.2638 mg
? Step -6.3400 %
-0.2801 mg
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
°C
Lab: UoA Chemistry
STAR° SW 16.10

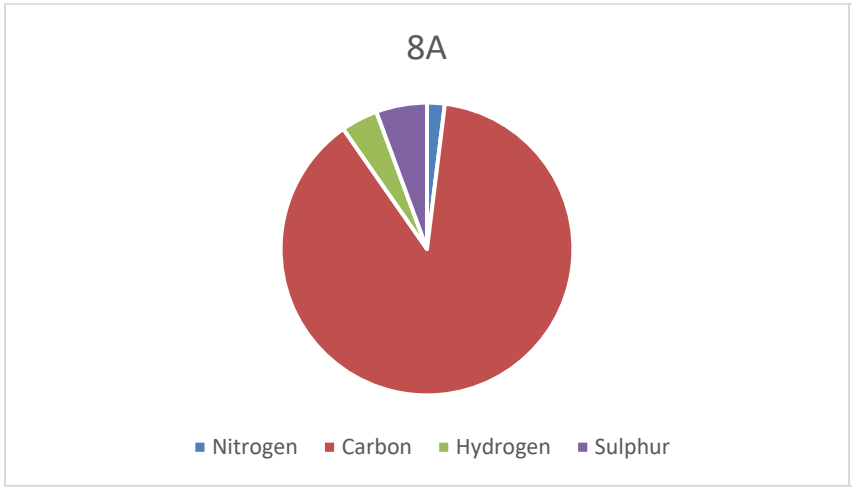
11B

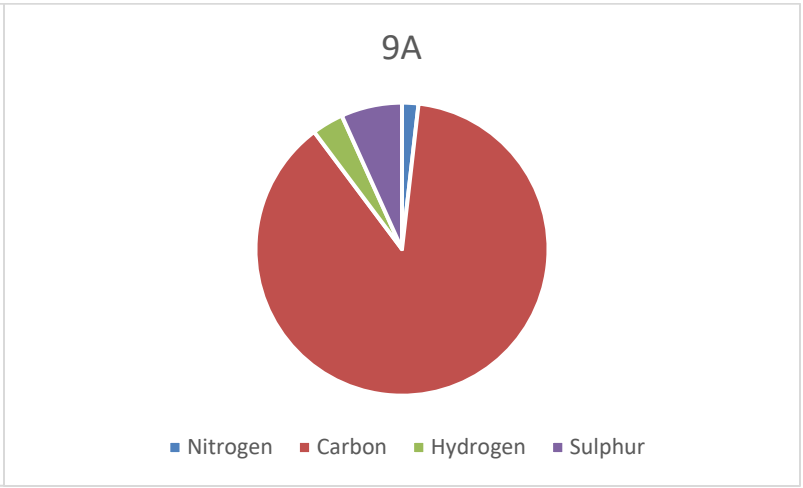
APPENDIX IV: ELEMENTAL ANALYSIS RESULTS

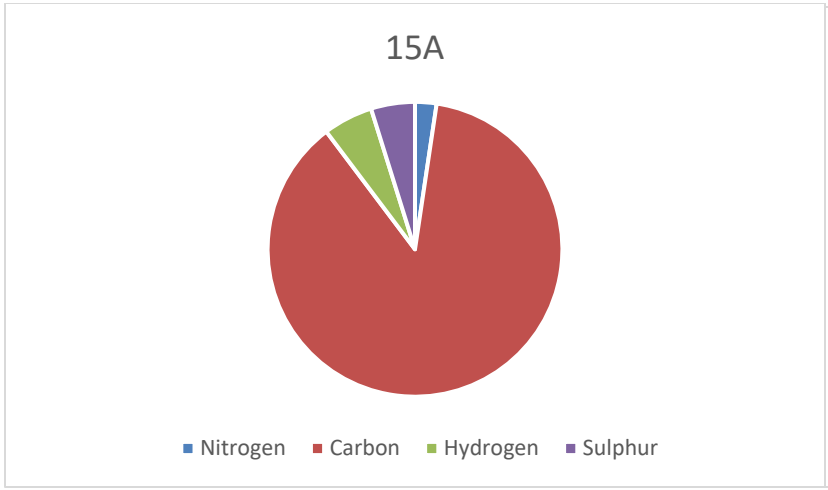
The following elemental analysis tests were performed at the Department of Chemistry at the University of Alberta.

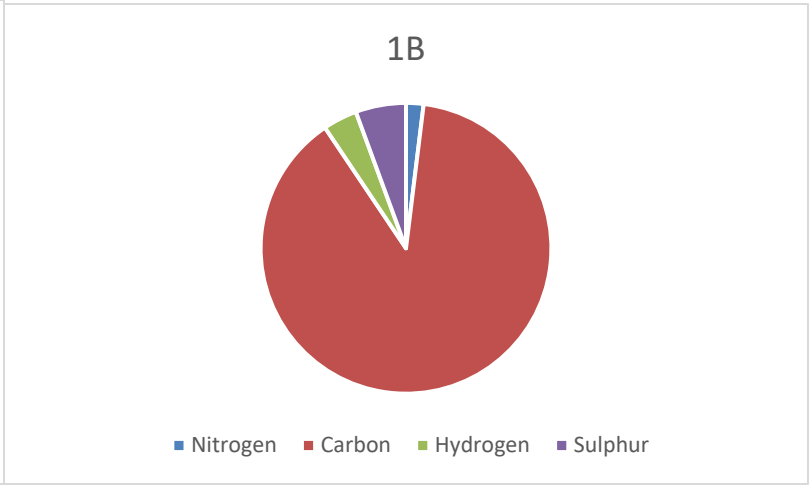


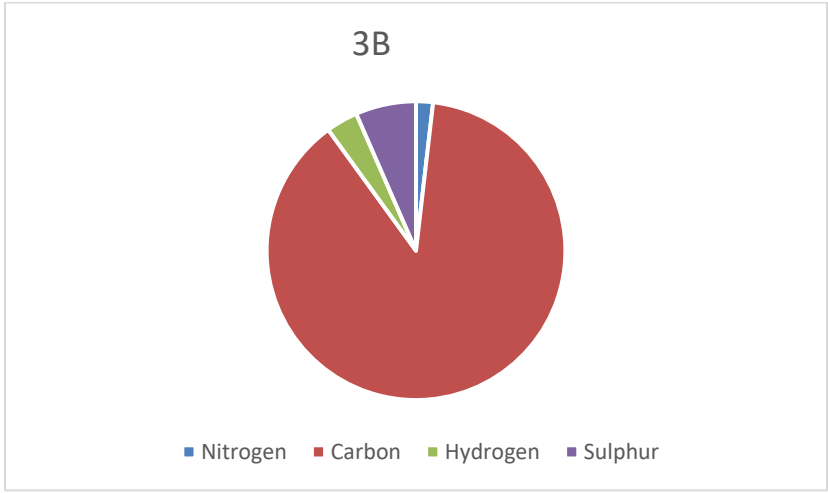


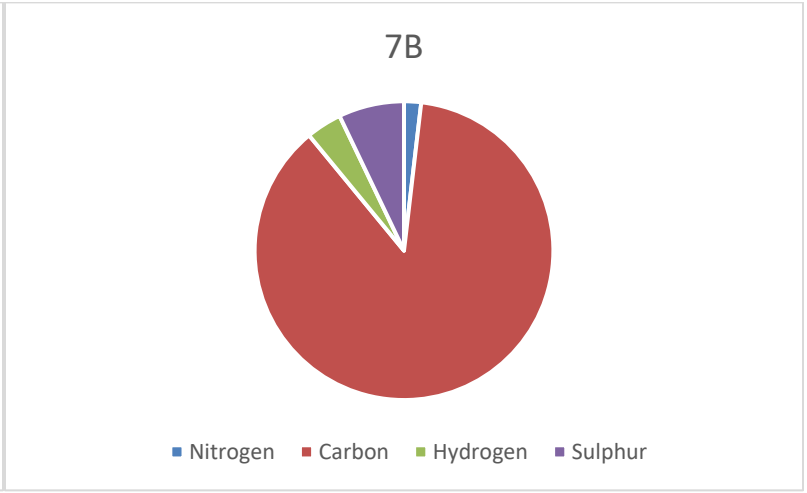


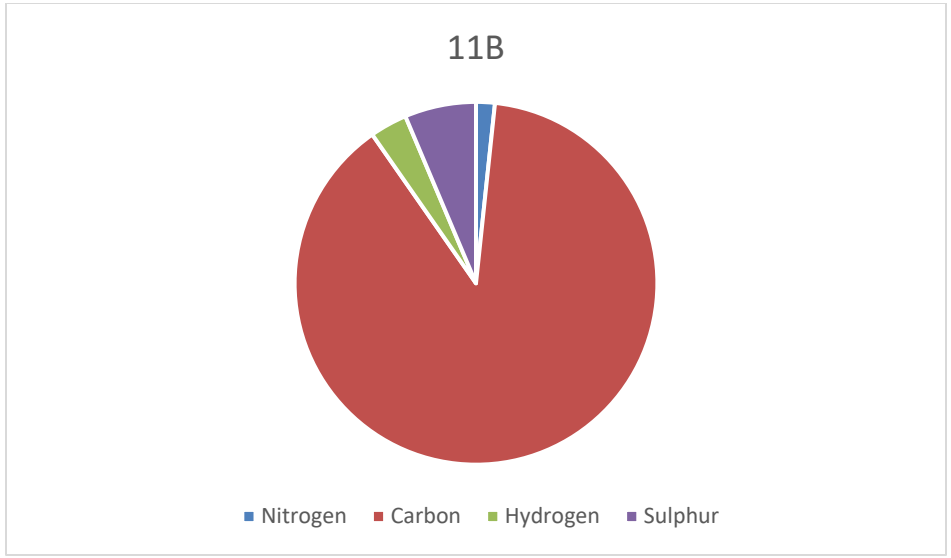



15A
Nitrogen
Carbon
Hydrogen
Sulphur


1B
Nitrogen
Carbon
Hydrogen
Sulphur


3B
Nitrogen
Carbon
Hydrogen
Sulphur


7B
Nitrogen
Carbon
Hydrogen
Sulphur


11B
Nitrogen
Carbon
Hydrogen
Sulphur