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Original Article

Studying Details of Phase Transitions of 7OCB Liquid Crystal using DSC Technique

Robert Nichol[1], Dipti Sharma[2]

[1,2]*Emmanuel College, Boston, MA, USA.*

[2]*Corresponding Author : Dr_dipti_sharma@yahoo.com*

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Abstract - *In this study, the Liquid Crystal (LC) 4-Cyano-4'-Heptyloxybiphenyl (7OCB) was studied using Differential Scanning Calorimetry (DSC). A small amount of the sample was used in the DSC, and the sample was heated from -20ºC to 100ºC and then cooled from that temperature at a 20ºC/min ramp rate. The data for heat flow were collected as a function of temperature and time for heating and cooling from DSC. Further, the detailed analysis was performed using LoggerPro. The 7OCB liquid crystal belongs to the family of nOCB that has an atom of Oxygen. Usually, nCB, a family of LC without Oxygen, has been used in smart devices such as Liquid Crystal Display (LCD), but the goal of this paper is to see if the nOCB family of LC, especially the member 7OCB from this family, can be used potentially in LCDs, comparing nOCB with nCB LCs. The Nematic phase transition and Nematic range are important for LCDs and Smart devices. Based on the detailed study of 7OCB, it is found that it shows a much longer Nematic range with an increase when compared with heat and cool of 41.7ºC. This wide range can be very useful in multiple different climates, the LC remaining in the nematic phase over a wider range of temperatures, withstanding different climates and seasons. This shows that the 7OCB liquid crystal can be very useful in the Liquid Crystal industry to create smart devices such as smart laptops, watches, TVs, etc.*

Keywords - *Liquid Crystals, Crystalline, Nematic, Isotropic, Phases, Nematic Range, Differential Scanning Calorimetry, LCD, Heat Flow, Specific Heat Capacity, Endothermic, Exothermic, Thermodynamics, Thermal speed, Thermal acceleration, Thermal jerk, Temperature, LoggerPro.*

1. Introduction

Liquid Crystals (LCs) are thermotropic materials that undergo multiple states of matter and show multiple phase changes and multiple phase transitions between solid and liquid as temperature changes. LCs exist in states, but more typical states are solid (crystalline), nematic, and liquid (isotropic) phases, each differing from the others in both energy and arrangement of the molecules. Solid phase molecules are clumped in a crystalline, uniform structure, and liquid phase molecules are free, in nonuniform arrangements. Typically, low temperatures dictate the solid phase, while high temperatures dictate the isotropic phase. In liquid crystals, there exists another phase between solid and liquid, a nematic phase. [1-3]

This third, middle phase is the nematic phase, where the molecules of the LC become soft and malleable, without becoming a liquid. The ability for LCs to enter this nematic phase is what makes them unique in this way. The nematic state makes these crystals valuable, where they are receptive to charges, while maintaining a stable state. Additionally, if the nematic range is wide, it can be used for LCDs, smart devices, and smart glasses, and some thermodynamic devices over a wider range of temperatures. If the temperature of the environment is above or below the nematic range, the liquid crystal will either undergo a phase transition into the isotropic or the crystalline range. [4-8] The nematic phase allows for more unique applications, unlike any other material, due to its structural stability and increased sensitivity to charge. In LCDs, the liquid crystal can maintain a vertical position while allowing for polarized light to pass through polarizers to the pixels, displaying an image.

The family of Alkyl Cyano Bipehlyl (nCB) has been studied widely by several authors and researchers because of its various phase transitions. [9-16] The nCB is easily available, costs less, and can be manufactured easily in labs. The members of nCB show the Nematic Phase, which is a required phase for LCDs. The Alkyl Oxy Cyano Biphenyl (nOCB) family of LCs is one of the families of LCs that is drawing more attention from researchers nowadays and has not been studied well yet. The nOCB is a different family from nCB. It is important to explore the hidden properties of nOCB LC and its phase transitions. This family is composed of a cyanobiphenyl group, with an alkoxy group with a number of

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carbons extending out. The difference between the nCB and nOCB molecules is an oxygen at the base of the carbon chain on nOCB molecules, which is absent in nCB molecules. [4-5] Every member of nOCB shows some uniqueness in their phase transitions. Some studies can be seen on a few members of nOCB LCs using the DSC technique. [17-22].

Since the nOCB family is not studied in detail yet, the focus of this research is to explore more members of the nOCB. For this particular paper, an odd member of nOCB, 7OCB, is picked. The goal of studying 7OCB lies in further developing the knowledge base on the nOCB family, exploring other members of the family to see if there are more suitable or efficient molecules that have a wider nematic range. The 7OCB liquid crystal is studied using the Differential Scanning Calorimetry (DSC) technique to explore more details of each phase transition of it. Another goal of this study is to see if 7OCB has any nematic phase or what uniqueness the 7OCB shows with the Nematic Phase. This paper focuses more in depth on the details of Crystalline, Nematic, and Isotropic phases of 7OCB for heating and cooling to determine the details of temperature at each phase transition and the range of each phase, focusing specifically on the range of the nematic phase. Exploring the molecule 7OCB and how chain length and the presence of the alkoxy group impact the nematic phase can lead to better insights into the nOCB family, potentially highlighting overlooked molecules compared to nCB LCs, that can help the world of LCDs and Smart Devices to use more options of LCs outside of nCB.

2. Experimental Details

The member of nOCB, 7OCB, was taken in the form of a fresh sample of 6.3 mg in an aluminum cup and lid system and sealed. Then the sealed cup-lid with the 7OCB was taken into the instrument Differential Scanning Calorimetry (DSC), model 214 instrument, from the NETZSCH company at WPI chemistry and biochemistry department, Worcester, MA. The sample was heated from -20 °C to 100 °C and then cooled back from 100 °C to -20 °C with a ramp rate of 20 °C/min for heating and cooling. The experimental and environmental conditions were kept identical during all experiments and runs. The data produced by DSC was then collected from the DSC to show how heat flows in the sample with time and temperature. These data were then taken to Logger Pro for further detailed analysis to examine details of the phase transitions of 7OCB. Some publications on nOCB and nCB can be seen in these publications, where the DSC technique was used. [8-22].

2.1. Theory

The 7OCB liquid crystal was analyzed through DSC, where the data were then processed in Logger Pro. The equations used all relate to heat flow and heat capacity, all showing different aspects of the properties of the 7OCB liquid crystal through heating and cooling. The heat going in or Leaving (Q), the Mass of the Substance (m), the specific Heat Capacity (Cp), the Temperature (T), Ramp Rate (dT/dt), Heat Flow (HF), the Change in Heat (dQ), and Time (t) are all used in the functions below. The heat flow of 7OCB in DSC can be determined as a function of heating rate and temperature.

1. $HF = dQ/dt$
2. $dQ/dt = m \bullet Cp \bullet dT/dt$

Specific heat capacity of 7OCB is found as

3. $Cp = (1/m) \bullet [(dQ/dt)/(dT/dt)]$

The heat flow of 7OCB can be used to find thermal speed, thermal acceleration, and thermal jerk. Thermal speed can be found through the derivative of the heat flow.

4. $d(HF)/dt = [(d^2)(Q)]/(dt^2)]$

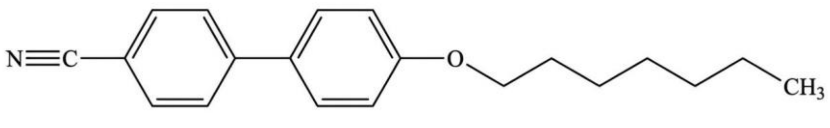
Thermal acceleration can be written as the second derivative of the heat flow.

5. $[(d^2)(HF)]/(dt^2)] = [(d^3)(Q)]/(dt^3)]$

Thermal acceleration can be written as the third derivative of the heat flow.

6. $[(d^3)(HF)]/(dt^3)] = [(d^4)(Q)]/(dt^4)]$

(a) 7OCB



(b) 7CB

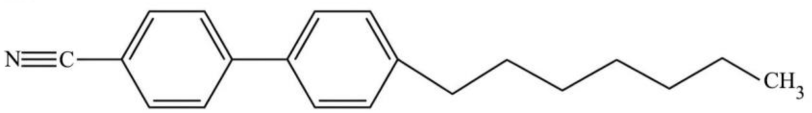


Fig. 1 Molecular structure of 7OCB and 7CB liquid crystal.

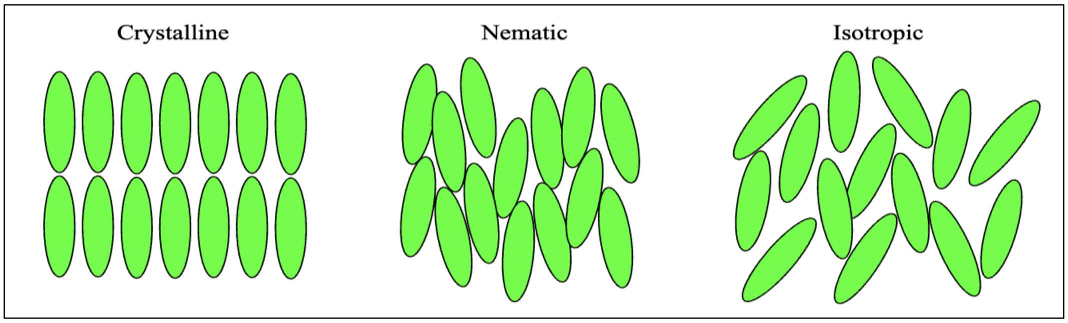


Fig. 2 Molecular Alignment of 7OCB liquid crystal, (a) Crystalline (K), (b) Nematic (N), (c) Isotropic (I).

2.2. Structure of 7OCB Liquid Crystal

In 7OCB liquid crystal, the 7 represents the number of carbon atoms in the alkyl chain, the O represents the presence of the oxygen atom, and the CB stands for Cyanobiphenyl, the two aromatic rings in the molecule. The difference between the nCB and nOCB liquid crystals is Oxygen at the base of the carbon chain on nOCB molecules, which is absent in nCB molecules. The molecular structure of 7OCB can be seen in Figure 1. Because of having a long molecular structure, the 7OCB molecule looks like a rod. The molecules of 7OCB stay in three different states: Solid - Crystalline, Intermediate state - Nematic, Liquid - Isotropic. The 7OCB undergoes two phase transitions, Crystalline to nematic (K-N) and Nematic to Isotropic (N-I) in heating, and then it goes back to the same phase transitions when it is cooled from Isotropic to Crystalline, following I-N and N-K. The molecular alignment of these three states of 7OCB can be seen in Figure 2.

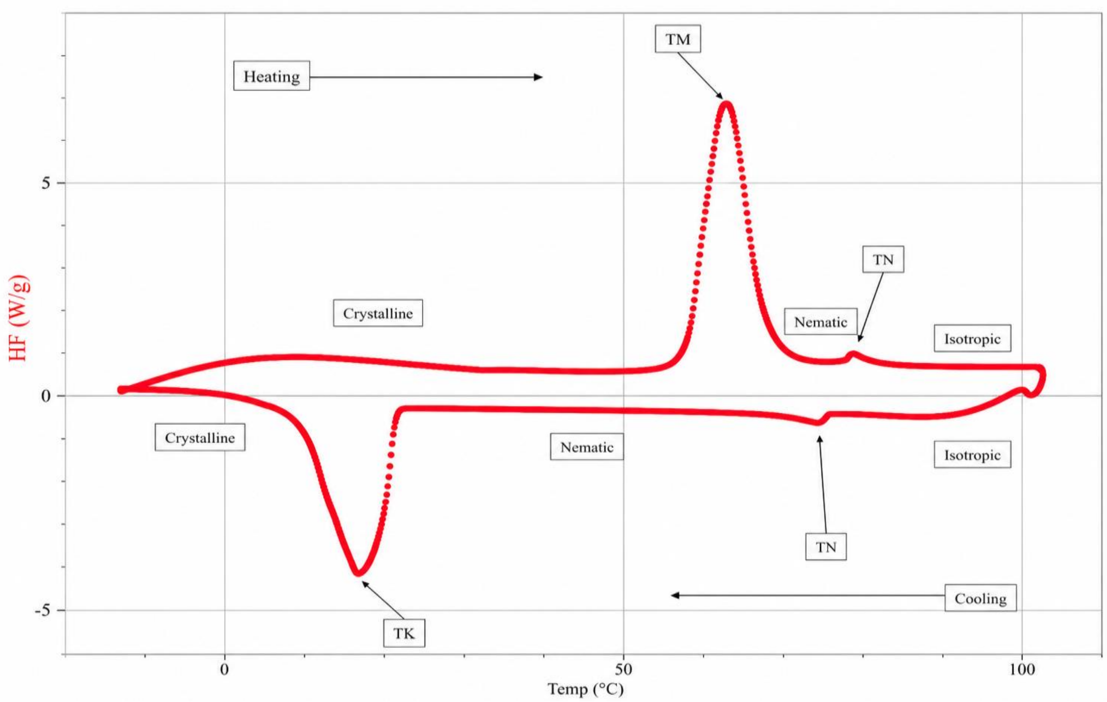


Fig. 3 Heat Flow vs Temperature plot for 7OCB liquid crystal, showing heat and cool.

3. Results

The experimental details of the 7OCB liquid crystal from DSC can be seen in this section. More detailed graphs are plotted after analyzing the DSC data with Logger Pro and are shown here.

Figure 3 shows the heating and cooling cycles of the 7OCB liquid crystal in DSC. Heating started with a ramp rate of 20ºC/ min, the crystalline phase ranging from -20ºC to 62ºC, showing how much heat the 7OCB liquid crystal took to before it had enough energy to change to the nematic phase. The TM spike shows the crystalline phase changing to the nematic phase; the height of the peak shows how much energy the crystal absorbed to change phases. After changing to the nematic phase, ranging from only 62ºC to 70ºC, it quickly changed to the isotropic phase, taking very little energy to change, as shown by the much shorter spike, TN. The 7OCB liquid crystal then stayed in the isotropic phase until the heating cycle was completed at 100ºC, beginning the cooling cycle, where the isotropic phase is seen again from 100 to 75ºC. After a small spike, TN, the crystal entered the nematic phase, staying in that state until it reached 18ºC, where, after a lot of heat exiting the molecule, it returned to a crystalline state, as shown by TK. The nematic range in cooling was much broader, showing how much more stable the 7OCB molecule is in that state. The crystalline peak, TK, is also much broader in cooling than in heating, showing how reluctant the molecule was to return to the crystalline state.

Figure 4 shows the heat and cool of 7OCB separately. Details of each transition peak when it appeared in heating and cooling of 7OCB in DSC, as plotted in Figures 3 and 4, and the range of how long each phase transition occurs during heating and cooling can be seen in Tables 1, 2, and 3. It is clear from these tables that each phase transition is shifting backwards in cooling than in heating. The range for the Nematic phase increases much more in cooling than in heating.

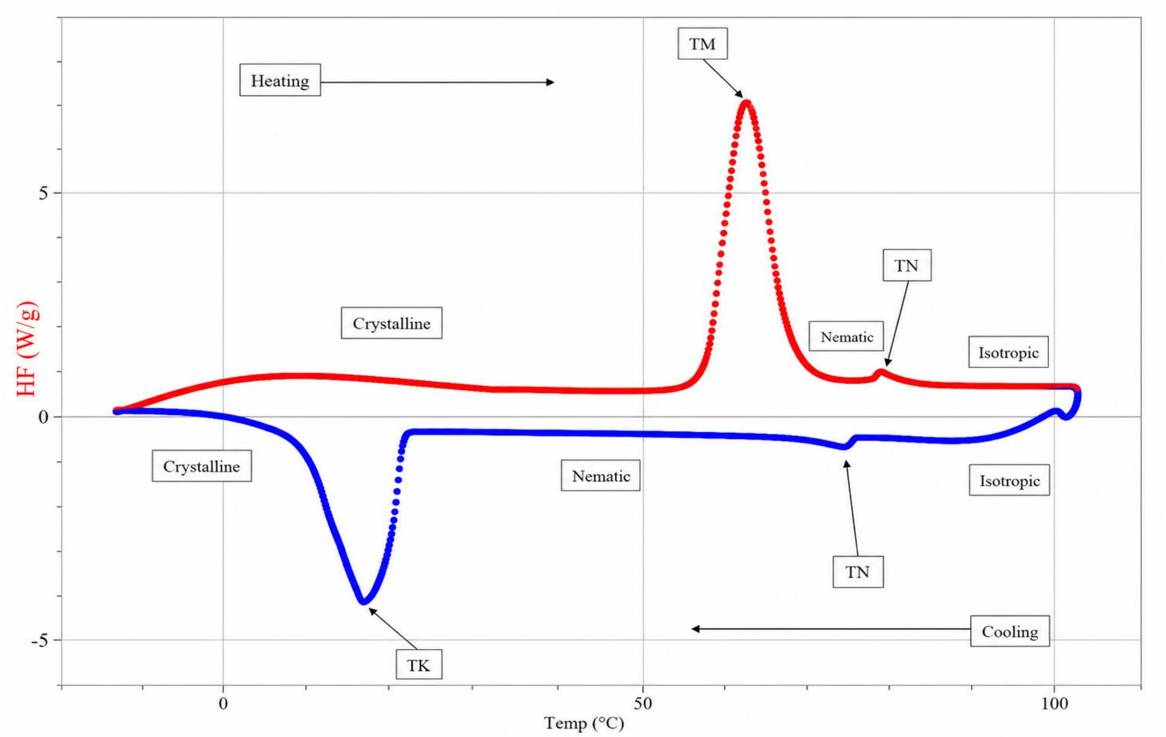


Fig. 4 Heat Flow vs Temperature plot for 7OCB liquid crystal, complete heat in red, and complete cool in blue

Table 1. Temperature Peak Details of each transition of 7OCB.

Heating Peaks	Temp (ºC)	HF (W/g)
Tm	62.7	7.02
Tn	78.7	1.26
Cooling Peaks		
Tn	74.5	-0.66
Tk	16.8	-4.17

Table 2. More details of each transition of 7OCB.

Heating Peaks	Wing Gap (W/g)	Height (W/g)	Width (ºC)
Tm	0.22	6.15	22
Tn	0.094	0.193	11.49
Cooling Peaks			
Tn	0.041	-0.208	14.8
Tk	0.42	-3.873	36.1

Table 3. Details of Transition Temperature Ranges for 7OCB.

HF vs Temp	**Crystalline Range (ºC)**	**Nematic Range (ºC)**	**Isotropic Range (ºC)**
Heating	50.6	16	23.8
Cooling	4.7	57.7	28.2
Change in Range between Heat and Cool (ΔR), (ºC)	-45.9	41.7	4.4

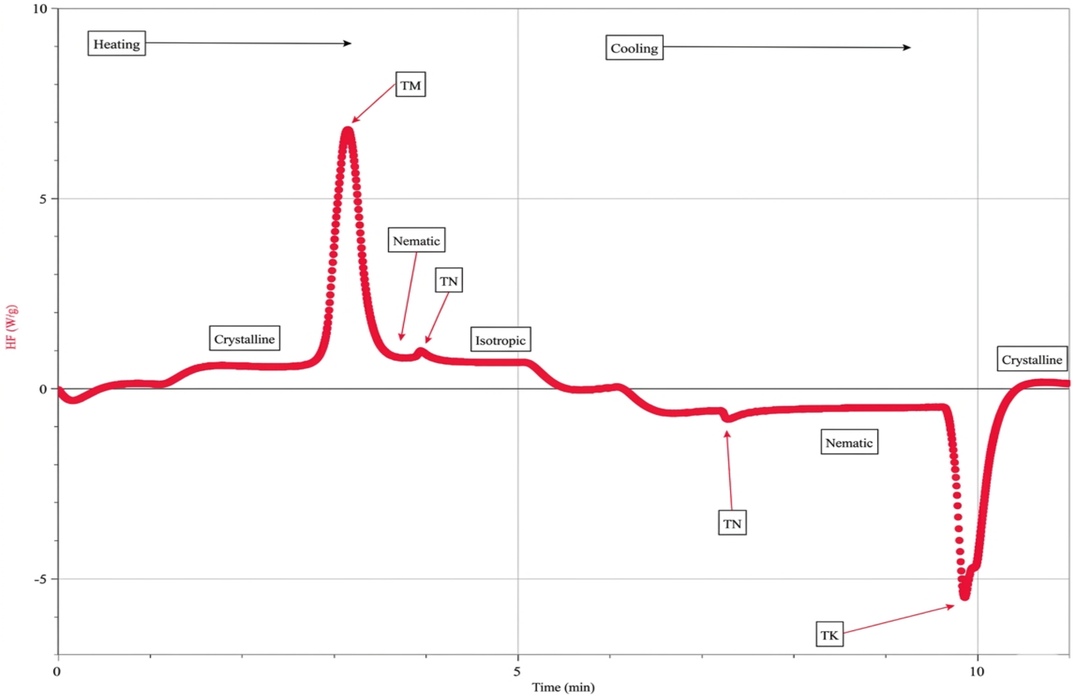


Fig. 5 Heat flow vs time plot for one heat and cool cycle, for 7OCB liquid crystal.

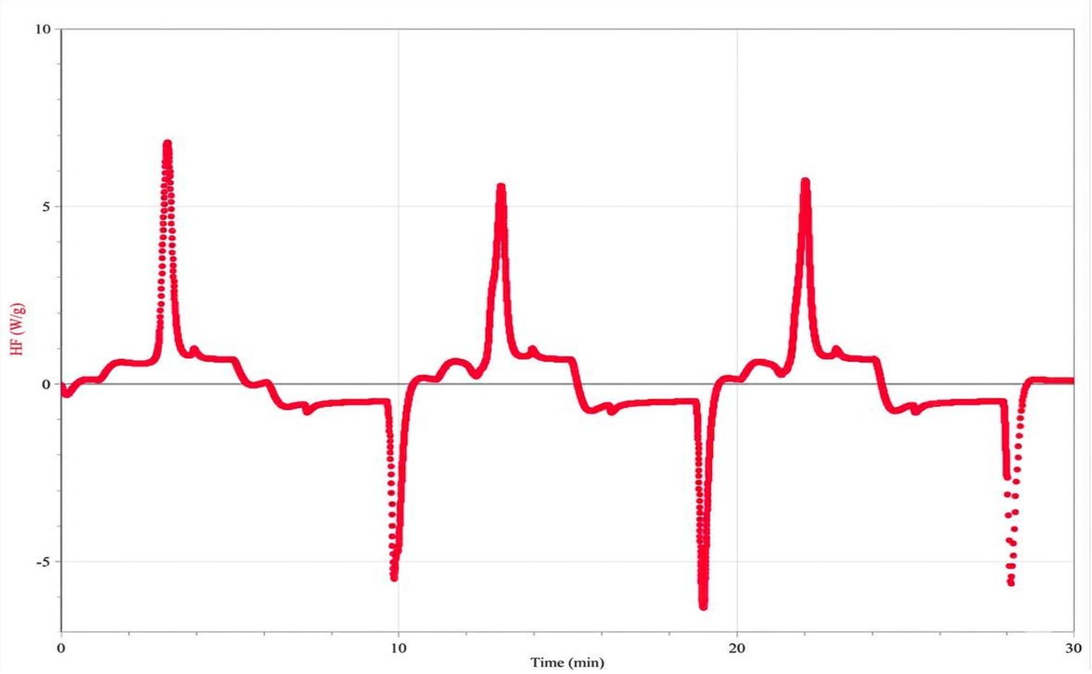


Fig. 6 Heat flow vs time plot for all three heat and cool cycles, for 7OCB liquid crystal.

Figure 5 shows a plot for heat flow versus time for 7OCB for heating and cooling for one cycle, whereas Figure 6 shows the same for three cycles together. The sharpest and tallest peak appeared is Tm (Crystalline in heating) and Tk (Crystalline in cooling), whereas the shortest peak appeared is Tn (nematic peak) in heating and cooling for each cycle. The data details of the time when these phase transitions appeared in Figures 4 and 5 are shown in Table 4.

Table 4. Details of the Time Range of each Transition of 7OCB.

HF vs Time	Crystalline range (min)	Nematic range (min)	Isotropic range (min)
Heating	1.5	0.8	1.08
Cooling	1.1	2.34	0.54
Change (ΔR)	0.4	1.54	0.54

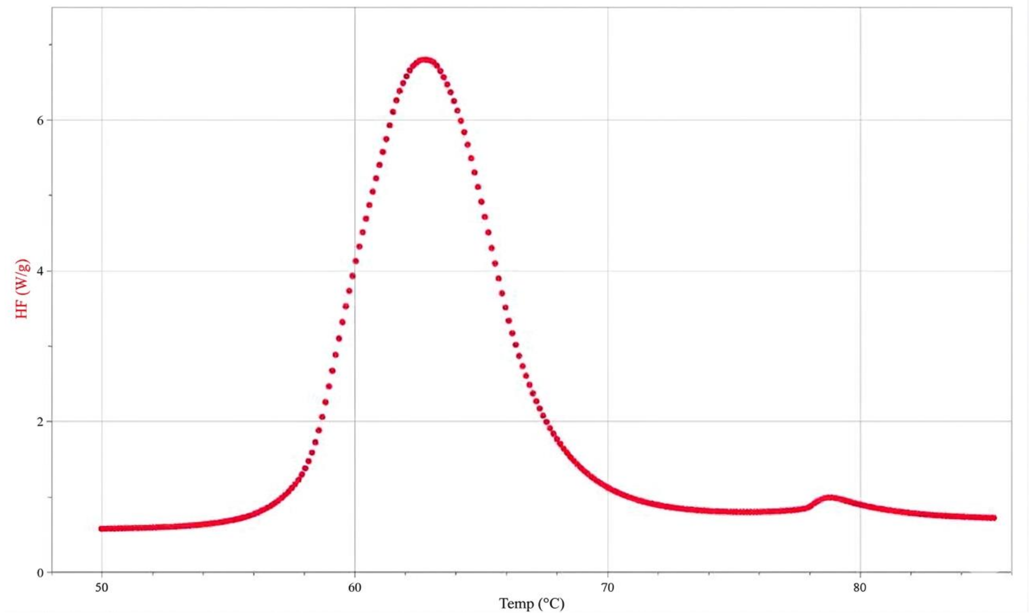


Fig. 7 Zoomed-in plot for heat only, for liquid crystal 7OCB.

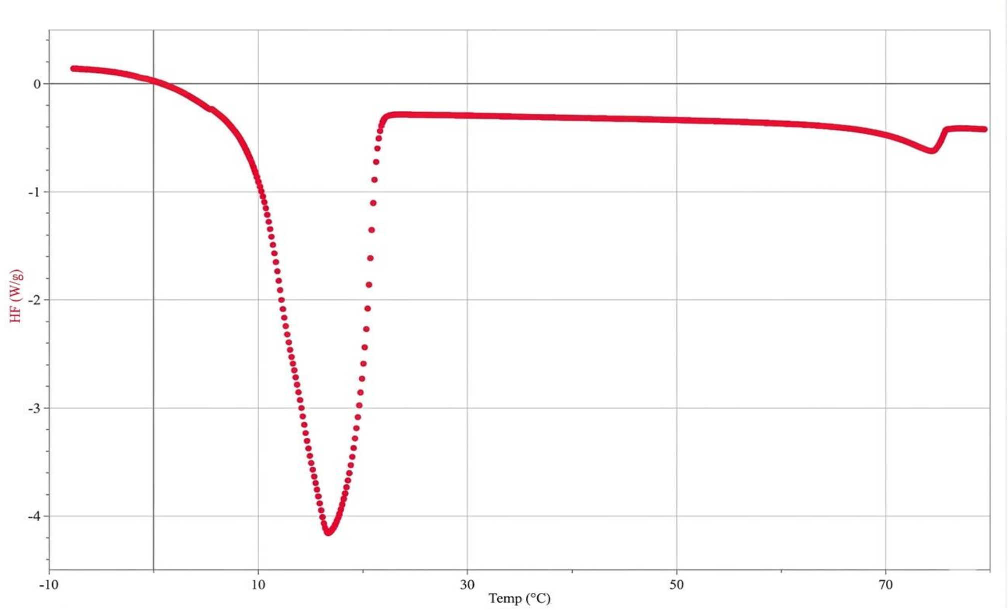


Fig. 8 Zoomed-in plot for cooling only, for liquid crystal 7OCB.

Figures 7 and 8 are the zoomed-in graphs for heating and cooling peaks. To see the peaks more clearly, zoomed-in graphs were plotted. Figure 7 shows the heating plot, and Figure 8 shows the cooling plot. The big peaks are the nematic to crystalline phase change peaks, and the small peaks are the phase change between the nematic and isotropic phases. The TK spike at 62ºC for heating shows the amount of heat the 7OCB crystal absorbs to change phases, while the TK spike at 18ºC for cooling shows the heat leaving the 7OCB molecule. The nematic phase for the heating plot is a much smaller range than for cooling, quickly transitioning into the isotropic phase. During cooling, the nematic phase lasts much longer.

For Figures 9-12, each peak was zoomed in on more, showing the heat flow into the molecule 7OCB for heating in Figures 9 and 10, and the heat flow out of the molecule for the cooling peaks, Figures 11 and 12. The peaks were singled out and enlarged in order to view them with greater ease. The wing gaps can be seen as the baseline of each peak changes after a phase transition.

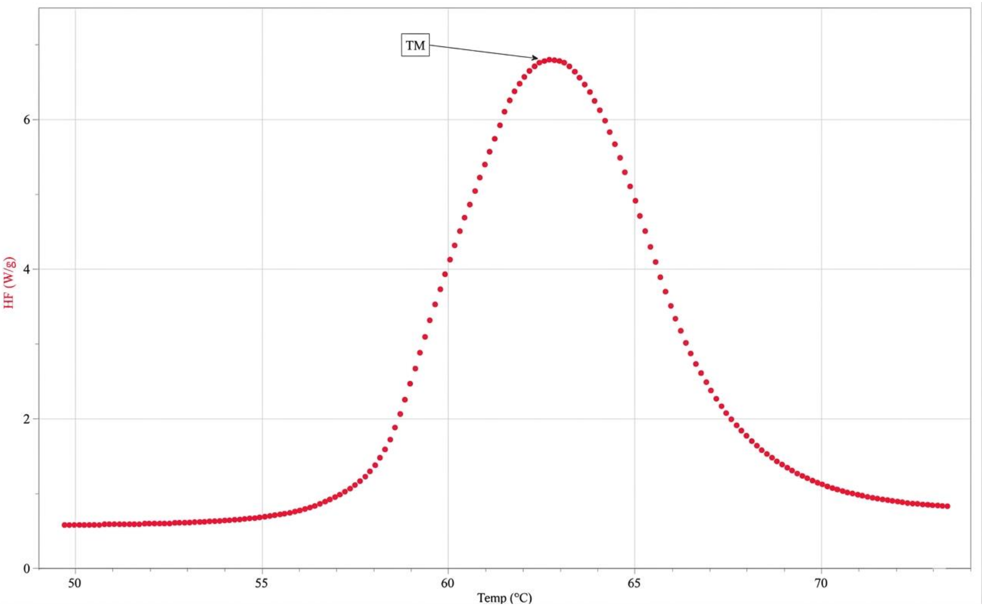


Fig. 9 Zoomed-in plot for the melting transition for heating, for 7OCB.

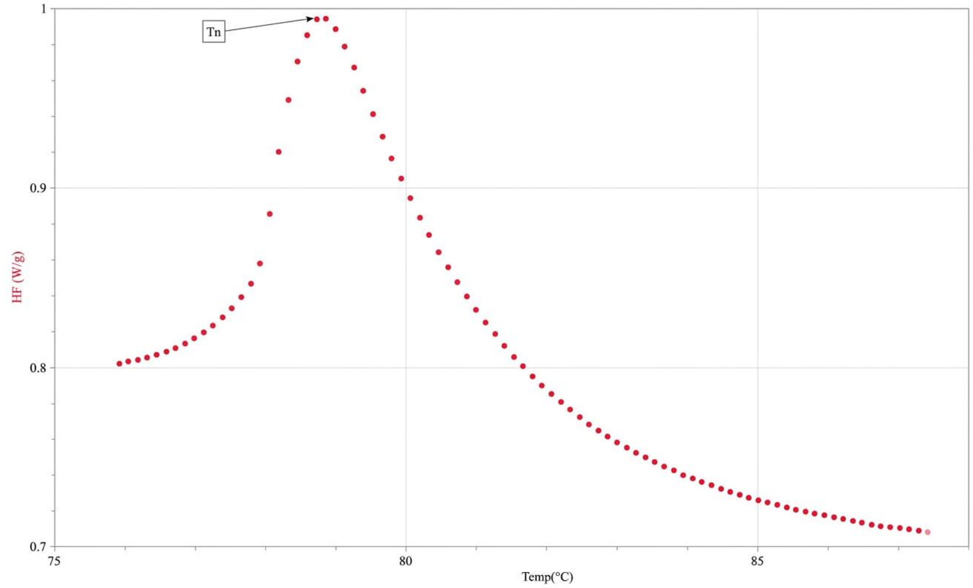


Fig. 10 Zoomed-in plot for nematic transition for heating, for 7OCB.

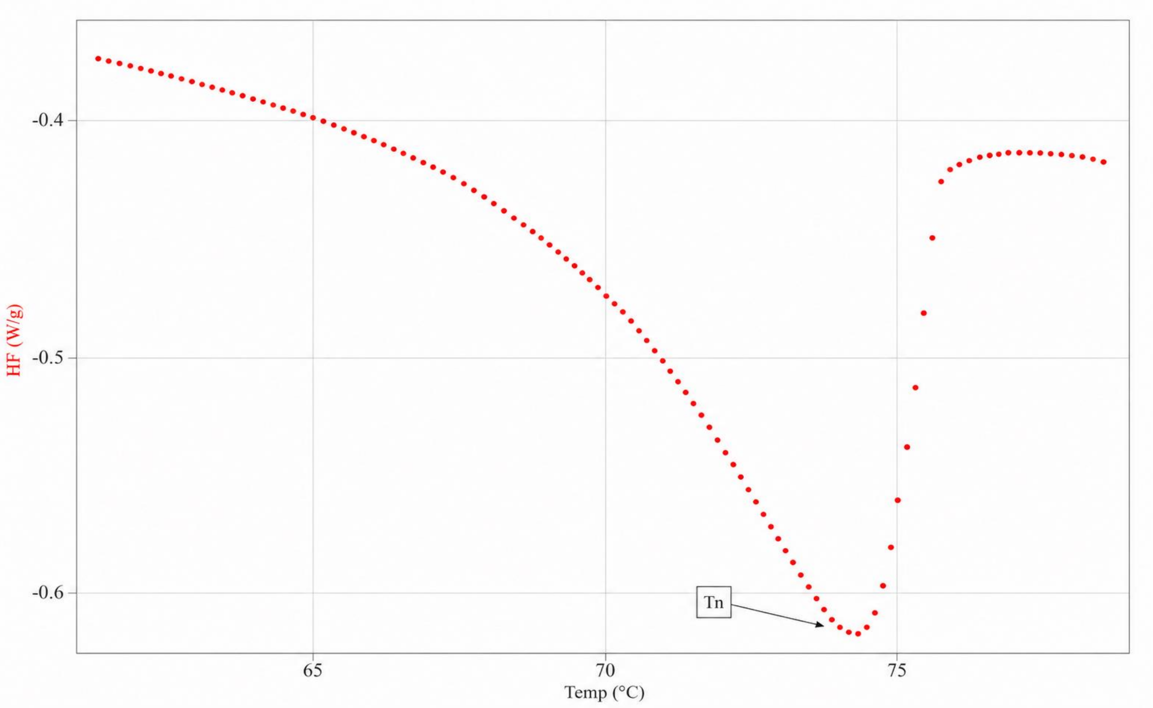


Fig. 11 Zoomed-in plot for nematic transition for cooling, for 7OCB.

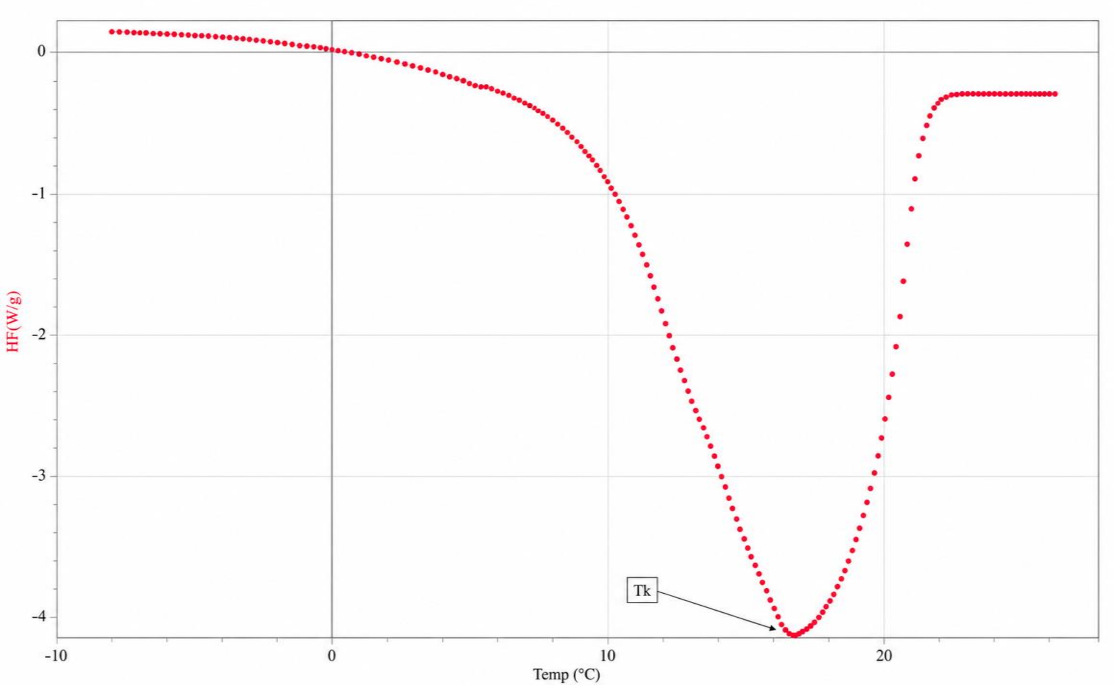


Fig. 12 Zoomed-in plot for crystalline transition for cooling, for 7OCB.

Figures 13 and 14 show the specific heat capacity of 7OCB for heat and cool separately, whereas Figure 15 shows the specific heat capacity of 7OCB for heat and cool together. The crystalline phase is much longer for heating than for cooling, taking more energy to get 7OCB to the nematic phase. During cooling, the nematic phase lasts much longer, having a much longer temperature range. The crystalline phase transition peak for cooling is much wider than it is for heating, showing how much longer it takes to return to a crystalline formation.

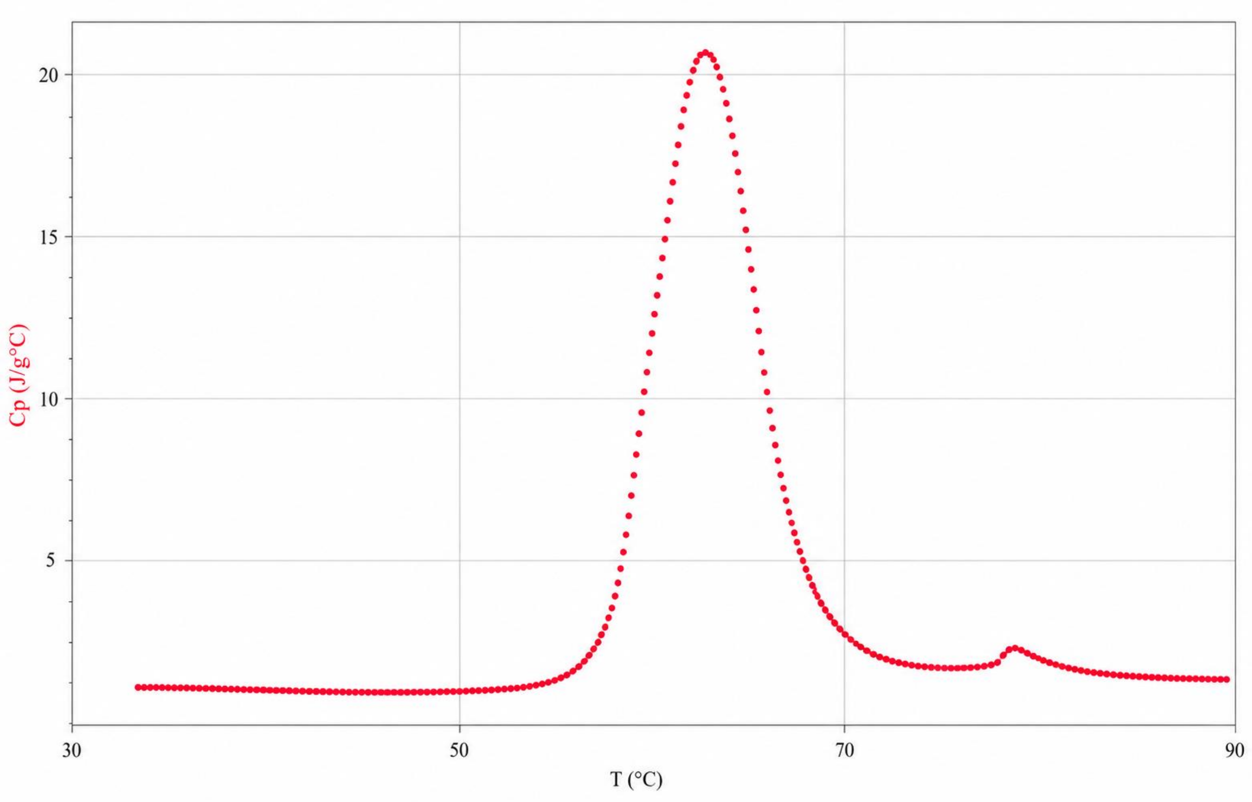


Fig. 13 Specific Heat Capacity (Cp) vs temperature (T) plot for heat only, for 7OCB.

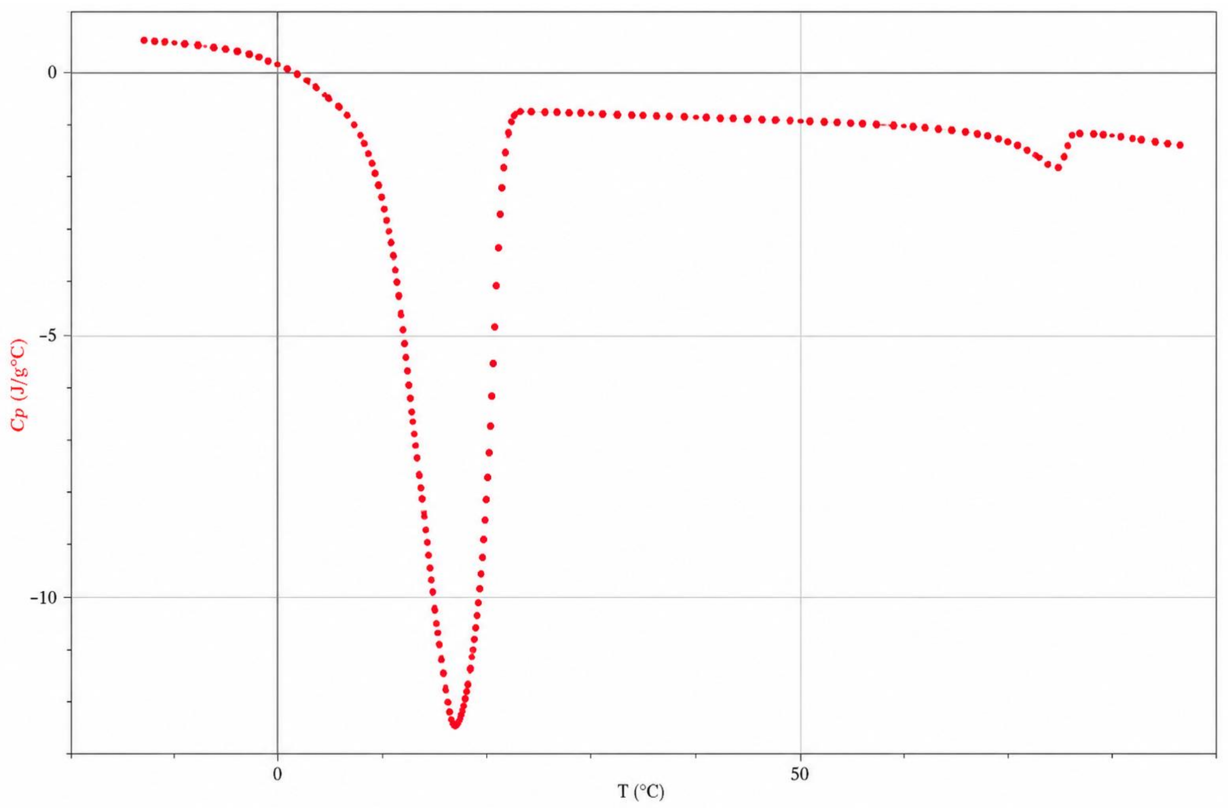


Fig. 14 Specific Heat Capacity (Cp) vs temperature (T) plot for cool only, for 7OCB.

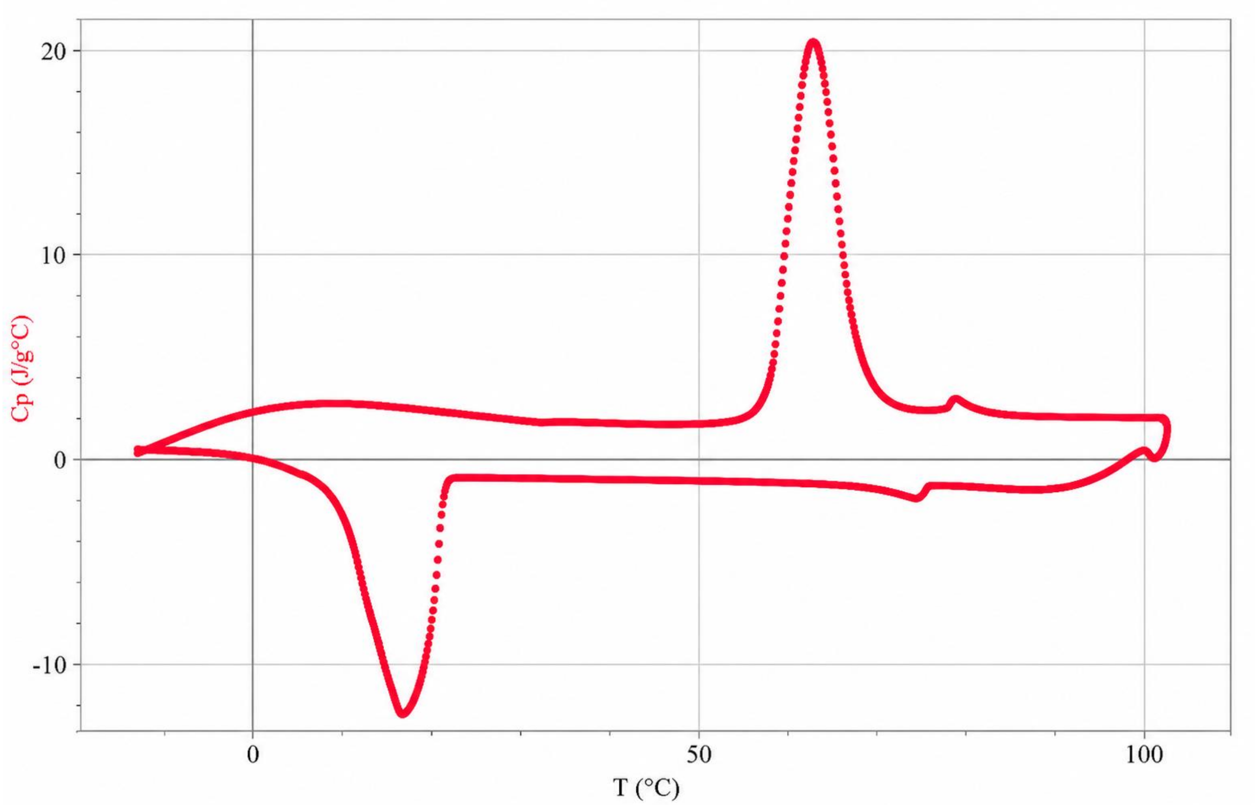


Fig. 15 Specific Heat Capacity (Cp) vs Temperature (T) plot for heat and cool together, for 7OCB.

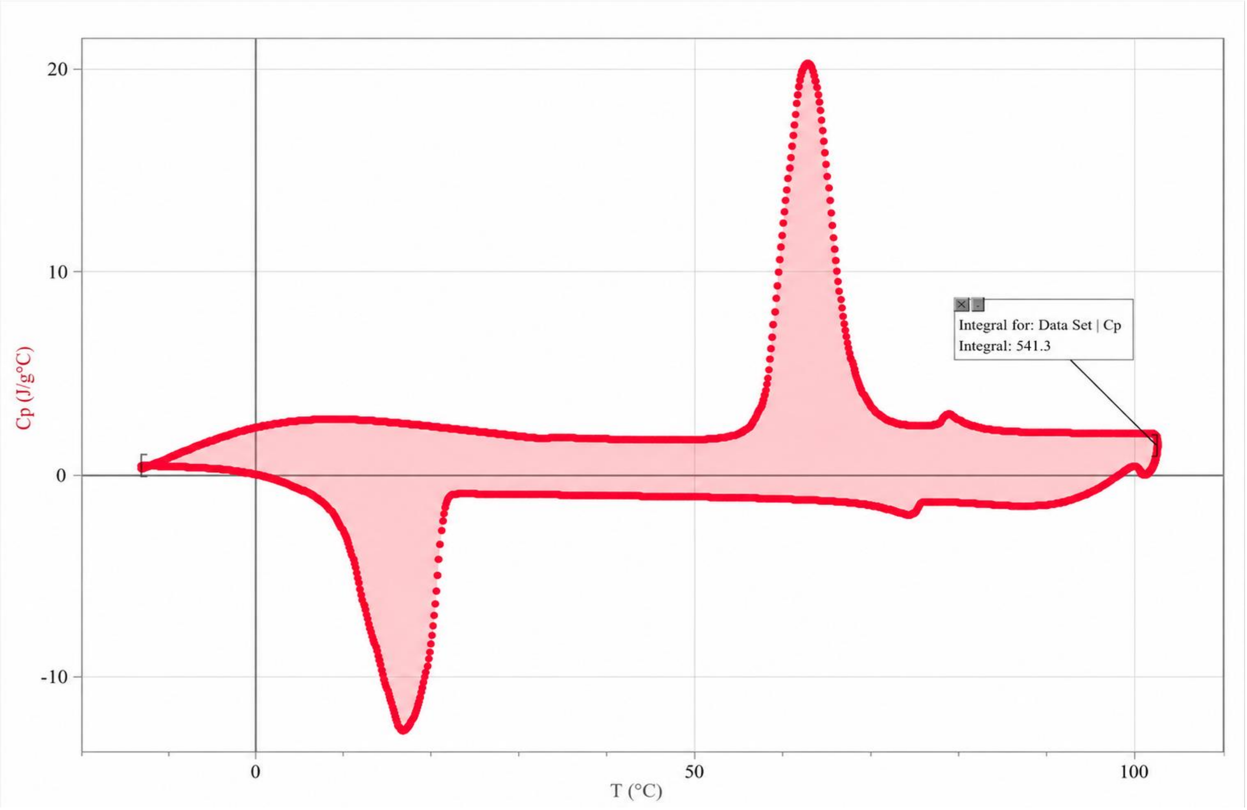


Fig. 16 Specific Heat Capacity (Cp) vs Temp (T) plot showing integral part with shaded area that is thermal energy trapped within 7OCB molecules during heating and cooling.

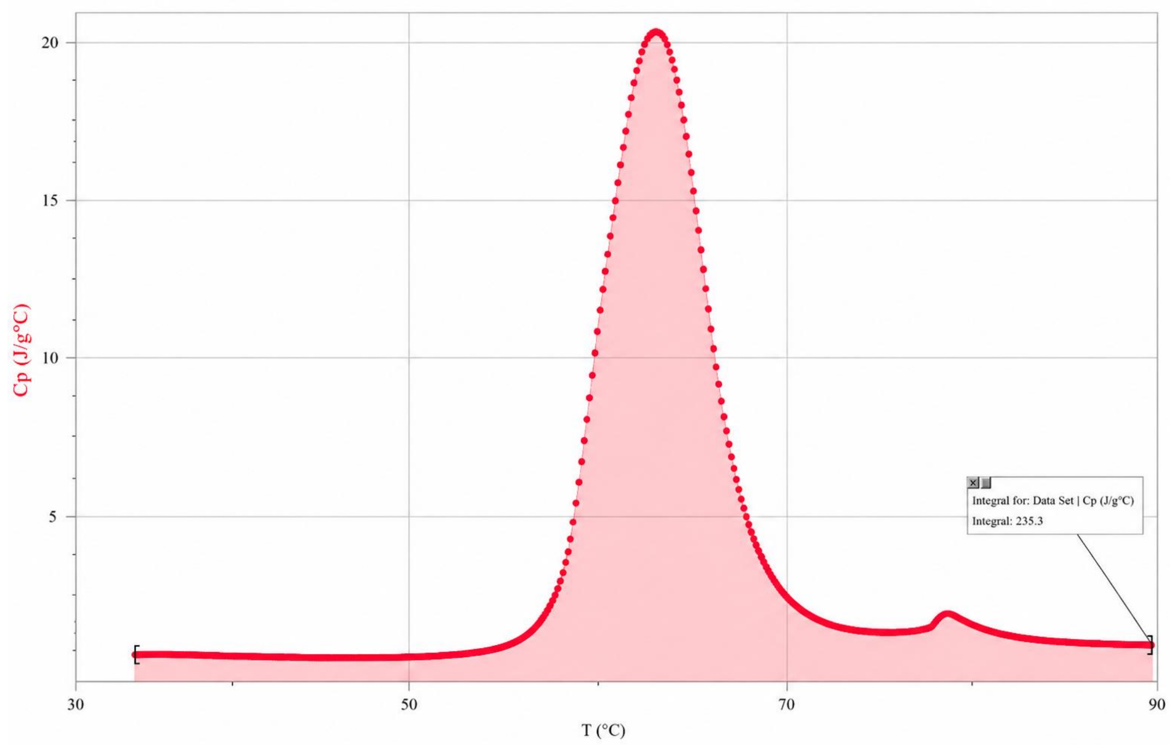


Fig. 17 Specific Heat capacity (Cp) vs Temp (T) integral, area under the curve for heating only

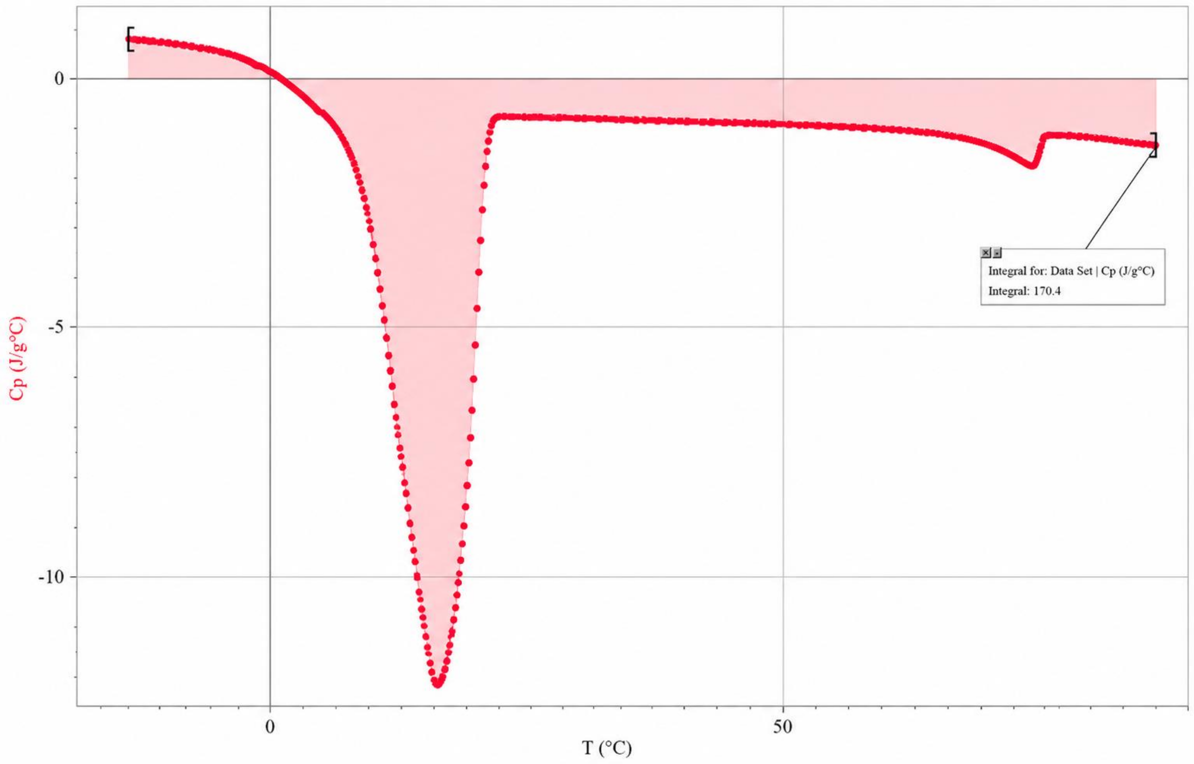


Fig. 18 Specific Heat capacity (Cp) vs Temp (T) integral, area under the curve for cooling only

For Figures 16-18, the full heat capacity vs. temperature was plotted for both heat and cool together, then heat and cool separately. To understand how much thermal energy can be absorbed and kept by the 7OCB molecule, the specific heat capacity vs temperature graph was integrated to find out the area under the heat and cool together, which gives how much thermal energy is kept between heat and cool, the enthalpy, the value of enthalpy being the integral of each peak. The heat and cool peaks were plotted alone, then integrated to find the enthalpy of the heat and cool cycles separately. The hysteresis present in Figure 16 shows how much energy is trapped in the 7OCB molecules during heating and cooling, whereas the area under each section of heating and cooling separately shows energy trapped for only heating and cooling in Figures 17 and 18. The data details of thermal energy, enthalpy, and specific heat capacity of 7OCB phase transitions can be seen in Table 5.

Table 5. (a) Enthalpy of each cycle. (b) Details of the Specific Heat Capacity of each transition for heating and cooling.

	Enthalpy (J/g)
Heat and cool	541.3
Heat only	235.3
Cool only	170.4
Change between heat and cool	64.9

Transition	**Cp (J/g^{o}C)**
T_m	20.33
T_n (Heating)	3.04
T_n (Cooling)	-1.87
T_k	-12.51

4. Derivative Graphs: Thermal Speed, Thermal Acceleration, and Thermal Jerk

To understand the details of the thermodynamics of the phase transitions of 7OCB, each phase transition of heat and cool was taken as 1st, 2nd, 3rd derivatives of Heat Flow to find out the thermal speed, thermal acceleration, and thermal jerk, respectively, of each peak. These results are shown in this section. The blue shows the 1st derivative, the green shows the 2nd, and the Orange shows the 3rd derivative of the heat flow of the transitions. This was plotted for each peak. Figures 19, 20, 21, and 22 show the derivatives of each peak, the first derivative showing thermal speed, highlighting the direction of heat. The second derivative shows the thermal acceleration, the spike showing how the heat is moving into or out of the 7OCB liquid crystal molecule. The third derivative shows the thermal jerk of the 7OCB molecule.

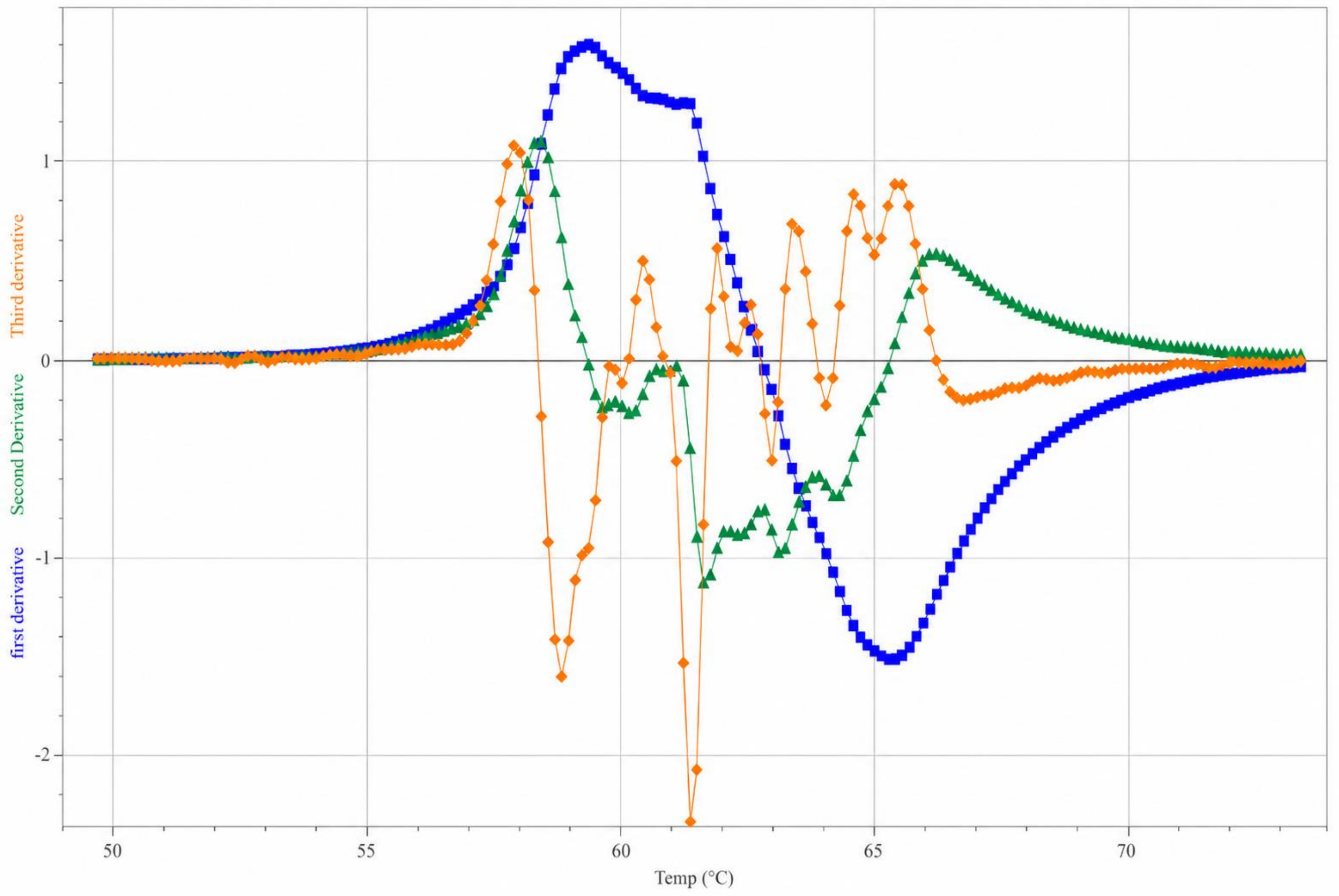


Fig. 19 Heating crystalline peak with three derivatives

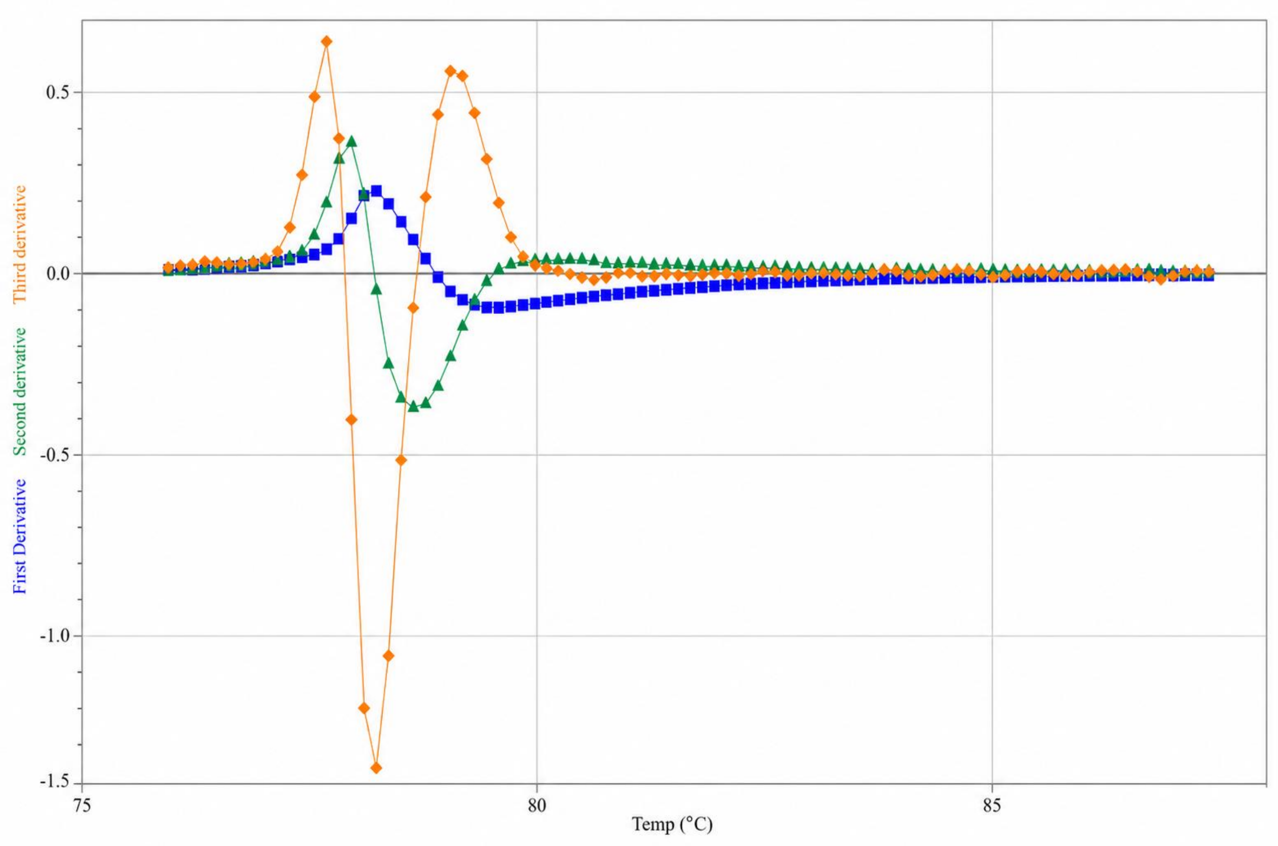


Fig. 20 Heating nematic peak with three derivatives.

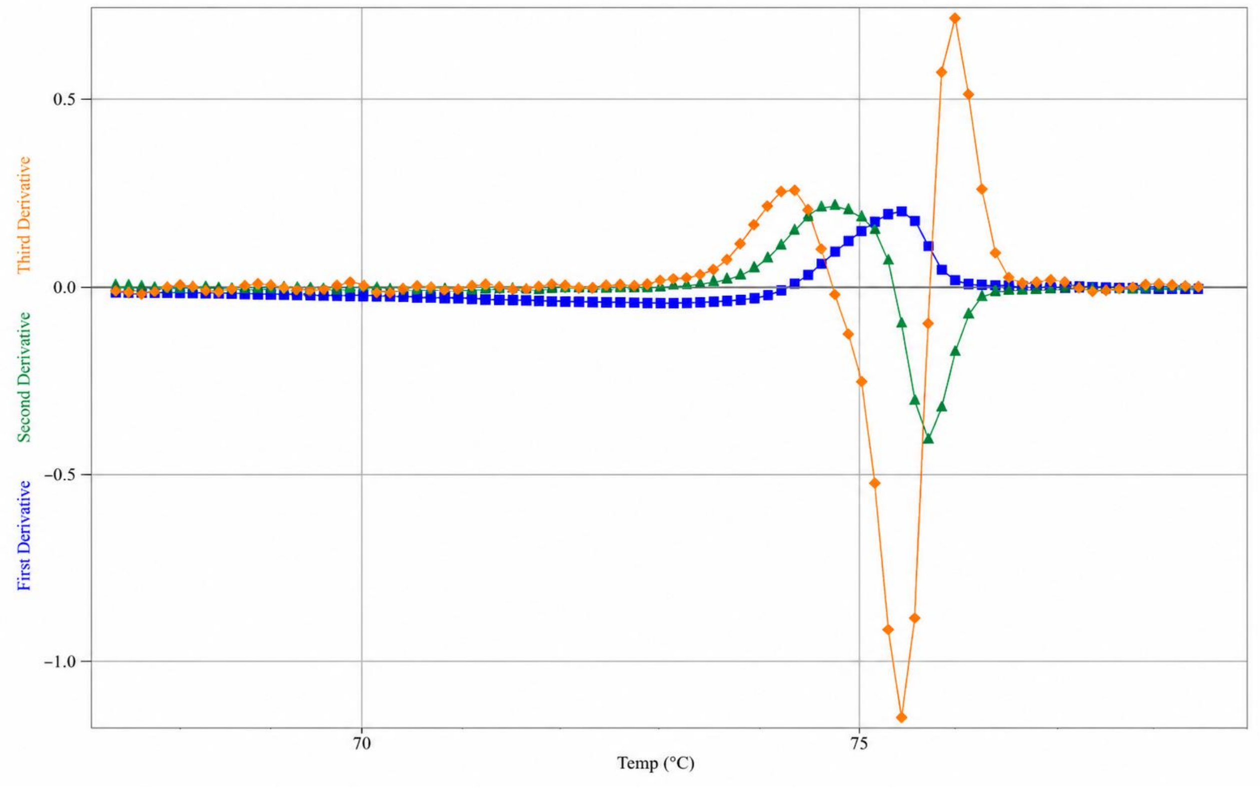


Fig. 21 Cooling nematic peak with three derivatives

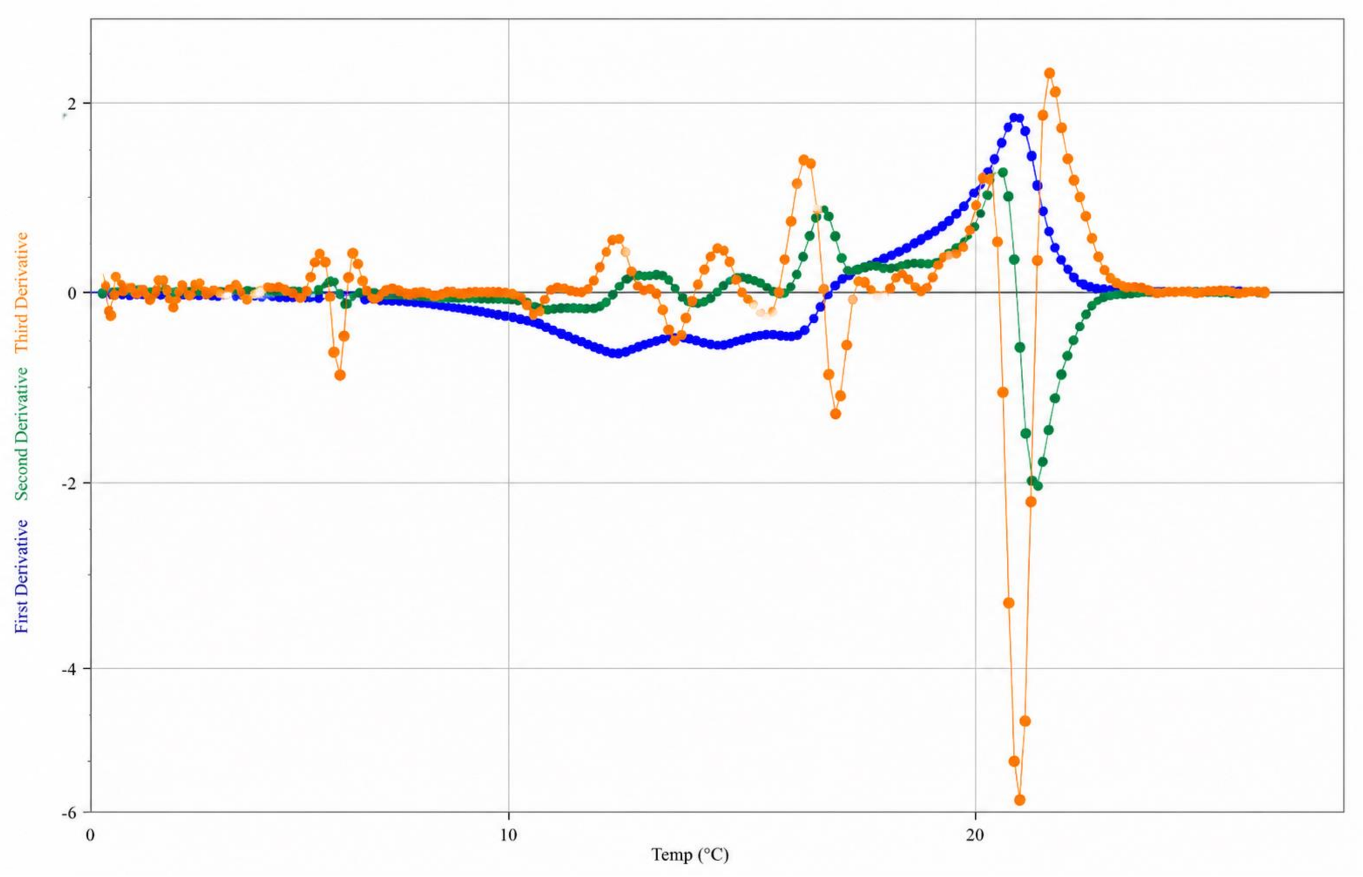


Fig. 22 Cooling crystalline peak with three derivatives.

The details of the data found from Figures 19-22, as Thermal speed, Thermal Acceleration, and Thermal Jerk of each phase transition of 7OCB, are shown in Table 6.

Table 6. Data details of Thermal Speed, Thermal acceleration, and Thermal jerk of each phase transition of 7OCB

Transition	Peak Numbers	Thermal speed		Thermal Acceleration		Thermal Jerk	
		T(°C)	v_ther (W/(g•s))	T(°C)	a_ther (W/(g•s^2))	T(°C)	J_ther (W/(g•s^3))
Crystalline Heating	1	59.36	1.612	58.47	1.100	57.93	1.085
	2	61.37	1.304	61.13	-0.036	60.47	0.495
	3			66.23	0.536	61.92	0.564
Crystalline Cooling	1	20.82	1.86	16.63	0.84	16.20	1.35
	2	20.82	1.86	20.47	1.22	20.13	1.14
	3	20.82	1.86			16.23	1.35
Nematic Heating	1	78.20	0.217	77.93	0.358	77.66	0.616
	2					79.00	0.557
Nematic cooling	1	75.45	0.218	74.78	0.232	76.01	0.773
	2					74.37	0.279

5. Discussion

7OCB is a thermotropic liquid crystal; it has a cyanobiphenyl group, with an alkoxy group with a chain of seven carbons extending out. The goal of this experiment was to test the number of phase transitions and the range of the nematic state in ºC. The 7OCB liquid crystal was run through a DSC, with a range from -20ºC to 100ºC, using a ramp rate of 20ºC/min. The total cycle was run 3 times for a total of 3 heating and 3 cooling cycles.

The heating and cooling crystallization peaks seen were strong first-order reactions, also seen by the number of spikes for each derivative. The isotropic peaks were weak first-order reactions. With one thermal speed peak, two thermal acceleration peaks, and three thermal jerk peaks each. The cooling crystalline peak is much wider than the heating crystalline peak, showing how much more heat needs to exit the molecule before it can enter the crystalline phase.

The purpose of plotting the derivatives of the heat flow is to see the thermal mechanics of liquid crystal 7OCB. The derivatives of the crystalline transitions show many peaks, while the nematic transitions have fewer peaks. The thermal speed peak of nematic heating is at 78.2 ºC, the acceleration peak is at 77.9 ºC, and the thermal jerk peak is at 77.7 ºC. This highlights the stability of the nematic phase, with much less deviation than the crystalline phase. The nematic phase also begins at a higher temperature, making it much stronger. Using 7OCB liquid crystal in smart devices is a possible future for manufacturing companies.

The nematic range during heating was very small due to the amount of energy needed to reach the crystalline transition peak, where 7OCB was readily able to transition to the isotropic phase once the nematic phase was reached. During cooling, the nematic range was much longer than heating, spanning over 57.7ºC, 41.7ºC larger than the heating phase nematic range.

The nOCB family of crystals is composed of the same groups, each with a different number of carbons in a chain from the end of the molecule. Each molecule in the nOCB family has a cyanobiphenyl group, with an alkyloxy group with a number of carbons extending out. The difference between the nCB and nOCB groups is Oxygen at the base of the carbon chain on nOCB molecules, which is absent in nCB molecules. [4-5] The 7OCB liquid crystal was analyzed through differential scanning calorimetry (DSC), in order to determine the details of temperature at each phase transition and the range of each phase, focusing specifically on the range of the nematic phase.

6. Conclusion

In this research, the liquid crystal 7OCB was studied in detail using the DSC technique to determine the unique results of each phase transition that occurred in the heating and cooling of 7OCB. From the results, it is clearly seen how the crystalline peak is much bigger in cooling than in heating. The thermal speed, thermal acceleration, and thermal jerk were also done to show how heat moved into and out of the 7OCB liquid crystal during each phase change. Derivative graphs from the nematic transition had fewer peaks than the crystalline transitions, showing less deviation, meaning a much more durable result in the nematic phase, which can be used in smart devices. The nematic phase was much wider in cooling than in heating as well, with an increase in range of 41.7ºC. The large range of the nematic phase during cooling is very useful; other liquid crystals with smaller nematic phase ranges fail if the climate is too hot or too cold, causing the liquid crystal to exit the nematic phase and either crystallize or enter the isotropic phase, causing the device to break. The high temperature of the nematic state, beginning at 78.7 ºC, is what allows it to withstand heat, being a very stable choice, with only 2 thermal jerk peaks for each nematic phase change. The wider range allows for use over a larger range of temperatures, in hot and cold climates, and withstands temperature changes between outdoors and indoors during cold or hot seasons.

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