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Variation of Heating Rate in Determining Melting Temperature Using Differential Scanning Calorimetry

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Abstract. An experimental study on the variation of heating rates in determining melting temperature using Differential Scanning Calorimetry (DSC) was conducted. This research aimed to investigate the effect of different heating rates on the determination of melting temperature using DSC, employing Indium Standard Reference Material (SRM) Lot BD147. The standard and sample were analyzed at heating rates of 5, 10, 15, 20, and 25°C/min. The resulted melting temperatures were analyzed using ANOVA, which revealed statistically significant differences. Heating rates of 5, 15, 20, and 25°C/min showed significant deviations from the standard rate of 10°C/min, indicating that 10°C/min is the most suitable rate for accurate melting temperature analysis based on ISO 11357-3.

Keywords: differential scanning calorimetry, heating rates, melting temperature, variation

1. Introduction

Differential Scanning Calorimetry (DSC) is a thermal analysis technique that measures the difference of heat flow between a sample and a reference under identical temperature conditions. Both the sample and reference are exposed to a controlled temperature program within a symmetric measurement system, allowing observation of heat flow as a function of time and temperature (Menczel et al., 2023). This technique is widely used to evaluate thermal properties such as heat capacity and enthalpy in materials such metals, alloys, and ceramics (Szécsényi & Menczel, 2023). During the analysis, a sample of known mass is subjected to heating or cooling, and variations in its heat capacity are reflected as changes in heat flow. DSC provides critical information on thermal transitions, including melting points, glass transitions, and curing behaviour. In material processing, enthalpy data derived from DSC can serve as an indicator of efficiency process (Sedov et al., 2016; Szécsényi & Holló, 2023). Due to its versatility and reliability, DSC become the most frequently used in thermal analysis laboratory. For accurate Differential Scanning Calorimetry

(DSC) results, the selection of sample mass and the precise control of heating and cooling rates are critical parameters that must be carefully managed.

The melting point, which can be determined using DSC, is a critical parameter in the characterization of substances, particularly in the identification and purity analysis of compounds. Various factors can affect the accuracy, particularly the heating rate during analysis. This study aims to investigate the effect of heating rate on melting point determination for optimizing analytical conditions for more reliable and reproducible results.

2. Methods

2.1. Materials

This research work uses standard aluminum pans and covers, SRM indium standard (lot number BD147), and ultra-high purity (UHP) nitrogen gas.

2.2. Instrument

To support the experiment, we uses some instruments such as Differential Scanning Calorimeter (PerkinElmer DSC 4000), an analytical balance, a chiller, a crimper, scissors, and tweezers.

2.3. Preparation of DSC

Nitrogen gas was flowed at a rate of 19.8 mL/min. The Differential Scanning Calorimeter (DSC) instrument and connected computer were powered on and allowed to stabilize for 30 minutes. Afterward, the chiller was activated and set to maintain a temperature of approximately 20ºC.

2.4. Sample analysis

The indium standard was weighed within the range of 5-10 mg then pressed using a crimper and placed into the DSC instrument (Figure 1a) along with an empty reference pan (Figure 1b), which had also been pressed. The indium standard (SRM) was analyzed with varying heating rates of 5, 10 (as per ISO standards), 15, 20, and 25ºC/min, over a temperature range from 100ºC to 250ºC. This experimental procedure adheres to ISO 11357-3:2018, which provides guidelines for determining melting/crystallization temperatures and associated enthalpy changes.

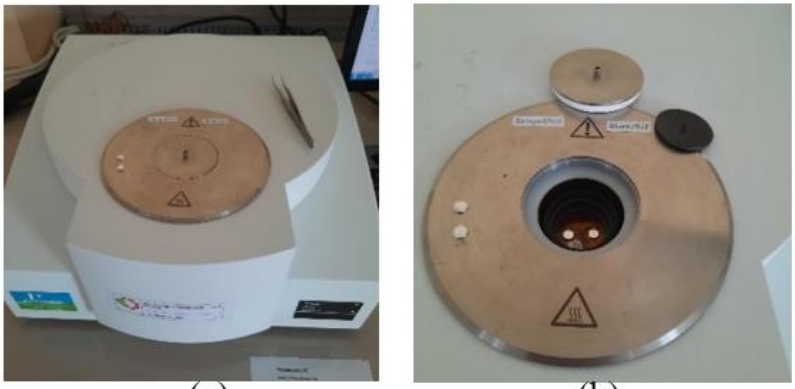

Figure 1. (a) Differential Sacanning Calorimetry; (b) Pan holder

3. Result and Discussion

This research work use ISO 11357-3:2018 as the guidance for experimental design, which outlines the determination of melting/crystallization temperatures and enthalpy changes. We use the heating rate of DSC testing of 10ºC/min as the verified value. This study was conducted to investigate the effect of heating rate variation on the melting point, using the indium standard. After conducting experiments with different heating rates, the results obtained significant differences as illustrated in Table 1.

Table 1. Melting Point Measurements (°C) at Different Heating Rates

Sample	5°C/min	10°C/min	15°C/min	20°C/min	25°C/min
1	155.91	156.43	157.33	158.03	158.91
2	155.79	156.54	157.31	158.03	158.87
3	155.76	156.68	157.29	158.14	158.80
Mean	155.82	156.55	157.31	158.07	158.86

A clear upward trend in the measured melting point was observed as the heating rate increased. The variance of each heating rate is tabulated in Table 2. To determine whether these differences were statistically significant, a one-way analysis of variance (ANOVA) was conducted.

Table 2. Descriptive Statistics by Heating Rate

Heating Rate	N	Mean	Variance
5°C/min	3	155.82	0.0063
10°C/min	3	156.55	0.0074
15°C/min	3	157.31	0.0004
20°C/min	3	158.10	0.0034
25°C/min	3	158.90	0.0007

The results of the one-way analysis of variance (ANOVA) are summarized in Table 3. The analysis revealed a statistically significant effect of heating rate on melting point, $F(4, 10) = 1218.66$, $p < .001$. The calculated F-value was substantially greater than the critical value of 3.48, suggesting that the observed differences in mean melting points across heating rate conditions are highly unlikely to have occurred by chance. This indicates a strong influence of heating rates on the melting point.

Based on Table 3, hypothesis testing can be conducted in two ways: first, by comparing the calculated F value with the value of F table, and second, by comparing the p-value with the significance level (error rate). In the first method, if the calculated F value is greater than the value of F table, then the null hypothesis (Ho) will be rejected. Conversely, if the calculated F value is

smaller than the value of F table, then Ho will be accepted (Suherman et al., 2020). In this study, the calculated F value was found to be 1218.656, which is larger than the value of F table which is 3.4781, thus it leads to the acceptance of H1. The second method involves comparing the p-value at a 95% confidence level to 0.05; if the p-value is smaller than 0.05, then Ho will be rejected. In this case, the p-value was indeed smaller than 0.05, hence H1 is accepted. Both methods of hypothesis testing support the conclusion that there is a significant difference in the effect of heating rate variation on melting point determination using DSC. Regarding to these results, a paired t-test was performed to compare each heating rate against the 10°C/min rate, which was used as the reference in line with ISO 11357 standards.

Table 3. One-Way ANOVA Summary

Source	SS	df	MS	F	*p*
Between groups	17.81	4	4.45	1218.66	< .001
Within groups	0.04	10	0.004		
Total	17.85	14			

3.1. Comparison of paired t-test results for different heating rates

As illustrated in Table 4, the hypothesis testing using the paired t-test was conducted in two ways. The first method compares the calculated t-value with the critical value from the t-distribution table. If the calculated t-value is greater than the critical value, then the null hypothesis (H_0) will be rejected. If it is smaller, then H_0 will be accepted. In this study, the calculated t-value exceeded the critical value, indicating that the alternative hypothesis (H_1) is supported.

The second method evaluates the p-value in relation to the significance level ($\alpha = 0.05$). If the p-value is smaller than 0.05, hence H_0 will be rejected; otherwise, it will be accepted. The results indicated that a p-value is less than 0.05, which again supports the acceptance of H_1. Both methods

led to the same conclusion which is a significant difference between each heating rate and the reference heating rate.

The variation in melting temperature observed with different heating rates is primarily attributed to thermal lag at higher rates, where the furnace temperature rises faster than the sample can absorb heat, resulting in an overestimation of the melting point. Conversely, very low heating rates may cause baseline drift and reduce measurement resolution. This phenomenon has also been reported in several previous studies.

Table 4. Results of paired t-tests comparing 10°C/min with other heating rates

Comparison	Calculated t-value	Critical t-value (two-tailed)	p-value	t-value vs. t-critical	p-value vs. α (0.05)	Interpretation
10°C/min vs 5°C/min	7.6968	4.3026	0.0165	t > t-critical	p < 0.05	Significant
10°C/min vs 15°C/min	12.3655	4.3026	0.0065	t > t-critical	p < 0.05	Significant
10°C/min vs 20°C/min	25.6826	4.3026	0.0015	t > t-critical	p < 0.05	Significant
10°C/min vs 25°C/min	38.6953	4.3026	0.0007	t > t-critical	p < 0.05	Significant

Toda (2016) demonstrated that higher heating rates in DSC analysis consistently shifted the melting peak to higher temperatures due to delayed heat penetration, which supports the present study's findings. Similarly, Vyazovkin (2020) evaluated the kinetic parameters of melting and crystallization in polymers and concluded that moderate heating rates, such as 10°C/min, strike a balance between accuracy and thermal responsiveness, especially for materials with complex crystallinity. This phenomenon is strongly aligned with the current result, which supports the ISO 11357-3:2018 recommendation of using a 10°C/min heating rate for reliable melting temperature determination.

In addition, Furushima et al. (2018) utilized fast-scanning calorimetry to investigate melting and recrystallization kinetics, revealing that deviations from optimal heating conditions could lead to distorted thermal events and misinterpretation of phase transition data.

In conclution, these findings reinforce that the selection of heating rate plays a critical role in the accuracy of DSC measurements. Consistent with both ISO guidelines and prior research, this study confirms that a heating rate of 10°C/min provides the most accurate and reproducible results, minimizing thermal lag while maintaining adequate resolution for detecting melting transitions.

4. Conclusion

This study concludes that a heating rate of 10°C/min is the most accurate and reliable for determining melting temperature using DSC, which is suitable with ISO 11357-3:2018. Other rates produced significantly different results, confirming the importance of adhering to the standard for consistent and comparable measurements.

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