



Multiphysical-Chemical Insights into Hailstone Growth from Isotopes, Droplet Freezing, and Insoluble Particles

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Abstract. Laboratory experiments play a pivotal role in understanding the microphysical mechanisms of hailstone formation. However, few studies have applied multiple physicochemical analyses to the same hailstone, resulting in a fragmented understanding of hailstone growth. Here, for the first time, we present a comprehensive investigation of an individual layered hailstone by combining three laboratory techniques: isotopic analyses, droplet-freezing experiments and species/size distribution of insoluble particles. Isotopic analyses revealed an up-down vertical growth trajectory, with the embryo exhibiting an exceptionally low freezing temperature of $-22.0\text{ }^{\circ}\text{C}$, as considerably lower than values reported in previous studies. Droplet-freezing experiments demonstrated three to four distinct ice-nucleating particle (INP) modes, and showed that initial freezing temperatures increased by $5\text{--}6\text{ }^{\circ}\text{C}$ from the embryo toward the outer shells. The size distributions and chemical compositions of insoluble particles including organics, dust, and bioprotein particles all varied among the different INP modes. Larger insoluble particles and higher particle concentrations were associated with freezing at higher temperatures. Notably, bioprotein particles larger than $6\text{ }\mu\text{m}$ were absent from the embryo but consistently present in the outer shells, providing a mechanistic explanation for the embryo's relatively low freezing temperature. By integrating isotopic, droplet-freezing, and insoluble particle species/size measurements on the same hailstone, this study provides new insights into the microphysical mechanisms governing hailstone growth and highlights the importance of insoluble particle characteristics in dominating hailstone ice nucleation.

1 Introduction

Hailstorms pose significant threats to public safety, economic development, and ecosystems (Allen et al., 2020; Cao, 2008; Kim et al., 2023; Raupach et al., 2021; Tippett et al., 2015; Zhang et al., 2026). The microphysical mechanisms governing hailstone formation and growth are highly complex, and a comprehensive understanding and accurate prediction of hailstorms remain challenging. Radar observations (Brook et al., 2021; Conway and Zrnić, 1993; Schlatter et al., 2008) and numerical simulations (Dennis and Kumjian, 2017; Kahraman et al., 2024; Liu et al., 2023; Ziegler et al., 1983) are conventional approaches for investigating hailstone growth. However, the inherent complexity of deep convective clouds limits the ability of both approaches to resolve the detailed physicochemical processes involved. Laboratory experiments therefore provide critical physicochemical insights into hailstone growth and are widely employed to investigate the microphysical mechanisms.

A fundamental component of these mechanisms is heterogeneous freezing, induced by ice nucleating particles (INPs), which plays a vital role in the initial formation of hailstone embryos (Allen et al., 2020). Immersion freezing, which is considered the dominant heterogeneous freezing process in mixed-phase clouds (Diehl and Grützun, 2018; Murray et al., 2012; Szakáll et al., 2021), is generally regarded as an important mechanism for hailstone embryo formation. Insoluble particles constitute a major source of atmospheric INPs (Lamb and Verlinde, 2011). Analyzing the insoluble particles that serve as primary INPs, including their composition, number concentration, and size distribution, can provide insights into their characteristics under



different atmospheric conditions and their potential influence on hailstone growth (Li et al., 2017; Michaud et al., 2014; Zhang et al., 2023; Zhao and Tian, 2026).

- 60 Previous studies have shown that larger insoluble particles generally exhibit greater ice-nucleating ability (Chen et al., 2021b; DeMott et al., 2010; Porter et al., 2020; Reicher et al., 2019), while their concentrations can vary by up to two orders of magnitude in different hailstorms (Zhang et al., 2023). Organics, dust, and bioprotein particles are crucial sources of atmospheric INPs. These particle species may contribute preferentially to ice formation over different temperature ranges, and thereby influence hailstone formation (Creamean et al., 2013; Ren et al., 2024; Suski et al., 2018; Zhang et al., 2023).
- 65 INPs in cloud hydrometeor samples often show similar dominant species to those from air samples: bioprotein particles, which are generally active at relatively high temperatures, often above approximately -15°C (e.g., Chen et al., 2021a, b; Hartmann et al., 2020; Hill et al., 2014; Joyce et al., 2019; Ladino et al., 2019; Šantl-Temkiv et al., 2019), and mineral dust, which commonly contributes to ice formation between -20°C and -30°C (Ladino et al., 2019; Murray et al., 2012; Si et al., 2019). Organics particles have been reported to contribute substantially to freezing over a broad temperature range (Testa et al., 2021); however, their role in hailstone formation through immersion freezing remains uncertain.
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- Laboratory experiments are essential for obtaining physicochemical information about hailstones and for advancing our understanding of hailstone microphysics. The layered structure of hailstones preserves information about the environmental conditions within the storm and the trajectory of hailstone growth (Knight, 1981; Lin et al., 2025; Michaud et al., 2014). Hailstone cross-sections can be classified into different shells according to their transparency, allowing the characteristics recorded in each individual shell to be examined in greater detail (Knight, 1981; Lin et al., 2025; Michaud et al., 2014; Zhang et al., 2023). Measurement of stable isotopes components, such as the deuterium (δD) and oxygen-18 ($\delta^{18}\text{O}$) ratios, can be used to trace the trajectories of hydrometeors within storms and provide information on hailstone formation temperatures and vertical growth paths (Li et al., 2020; Lin et al., 2025; Michaud et al., 2014; Tian et al., 2026). Droplet-freezing experiments quantify the immersion-freezing properties of hailstone solutions by recording the freezing events of supercooled droplets at temperatures below 0°C . These experiments provide temperature-resolved freezing spectra and are widely employed to assess the ice-nucleating ability of particles (Chen et al., 2021a; Reicher et al., 2019; Ren et al., 2024; Si et al., 2019; Wang et al., 2025).
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- Various laboratory techniques have been developed to characterize the composition, size, and concentration of insoluble particles in hailstones. Mandrioli et al. (1973) first confirmed the presence of viable biological entities in hailstone samples using colony-culture experiments. A comparative analyses of droplet-freezing experiments on hailstone-derived solutions before and after heat treatment further demonstrated that bioprotein particles can promote ice nucleation at relatively high temperatures (Michaud et al., 2014; Ren et al., 2024). Environmental scanning electron microscopy (ESEM) and energy dispersive X-ray spectroscopy (EDS) enable direct visualization and quantification of composition, size and concentration of insoluble particles (Bernal Ayala et al., 2024, 2025; Michaud et al., 2014; Zhang et al., 2023; Zhao and Tian, 2026).
- 85
- 90 However, the limited amount of material available from individual hailstones, together with sample consumption during laboratory analyses, has meant that most previous studies have applied only one or two techniques to each specimen. For



example, recent work on hailstones from the same Beijing storms involved separate samples for droplet-freezing experiments (Ren et al., 2024) and insoluble particles analyses (Zhang et al., 2023), respectively. Michaud et al. (2014) employed SEM and droplet-freezing experiments on distinct hailstone embryos collected from three separate Rocky Mountain storms to investigate their ice nucleation properties. Consequently, multi-method investigations of the same hailstone, particularly analyses that resolve differences among individual shells, remain scarce.

This study addresses this knowledge gap by conducting three complementary analyses on a single hailstone. After sectioning the hailstone into five shells, we employed isotopic analyses to reconstruct its vertical growth trajectory, droplet-freezing experiments to determine the temperature-dependent freezing spectra, and ESEM coupled with EDS to characterize the composition, size and concentration distribution of insoluble particles in each shell. By integrating these measurements on the same individual hailstone, we investigate the relationship among hailstone growth history, ice-nucleation activity, and the physicochemical characteristics of insoluble particles. The experimental methods are described in Sect. 2, the main results are presented in Sect. 3, and the conclusions are summarized in Sect. 4.

2 Method

2.1 Hailstone selection and processing

A hailstone with a large volume (diameter > 50 mm), five distinct shells and an intact morphology was selected for analyses to ensure sufficient material for all experiments. This ensures that the sample was representative while satisfying the constraints of factors including sample quantity, experimental setup and analyses duration. The hailstone was collected from a hailstorm in Yantai, Shandong, China (37.49°N, 121.45°E) at 14:12 local time on October 1, 2021, which was from the same hailstorm as in Lin et al. (2025), but approximately 1 km away from their sampling location (Fig. 1). It was immediately placed in a vacuum-sealed bag and stored in a freezer at -25 °C.

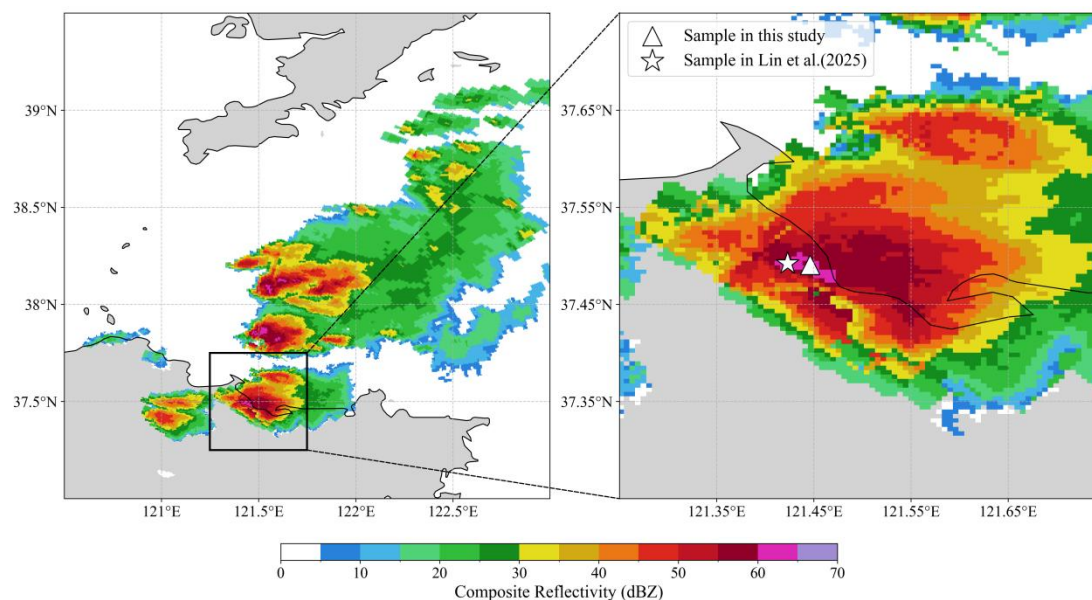


Figure 1: Composite radar reflectivity image of Yantai at 14:06 local time on October 1, 2021. Gray shading represents land; the triangle represents the hailstone sample location in this study; the star represents the sample location from Lin et al. (2025).

The analytical procedure is shown in Fig. 2. Surface contaminants, including grass, soil, and rainwater, were first removed by gently polishing the hailstone. The hailstone was then sectioned along its maximum dimension using an electrically heated Fe-Cr alloy wire. Based on their natural light transmittance, the cross-section was divided into alternating transparent shells and opaque shells. The individual shells were separated using the same heated-wire method. All the above operations were conducted at temperatures below -8°C to avoid unnecessary volume loss of the hailstone sample. Each shell was melted separately and transferred into a 2 mL glass sample vial. All vials were sealed and stored at 4°C until analyses.

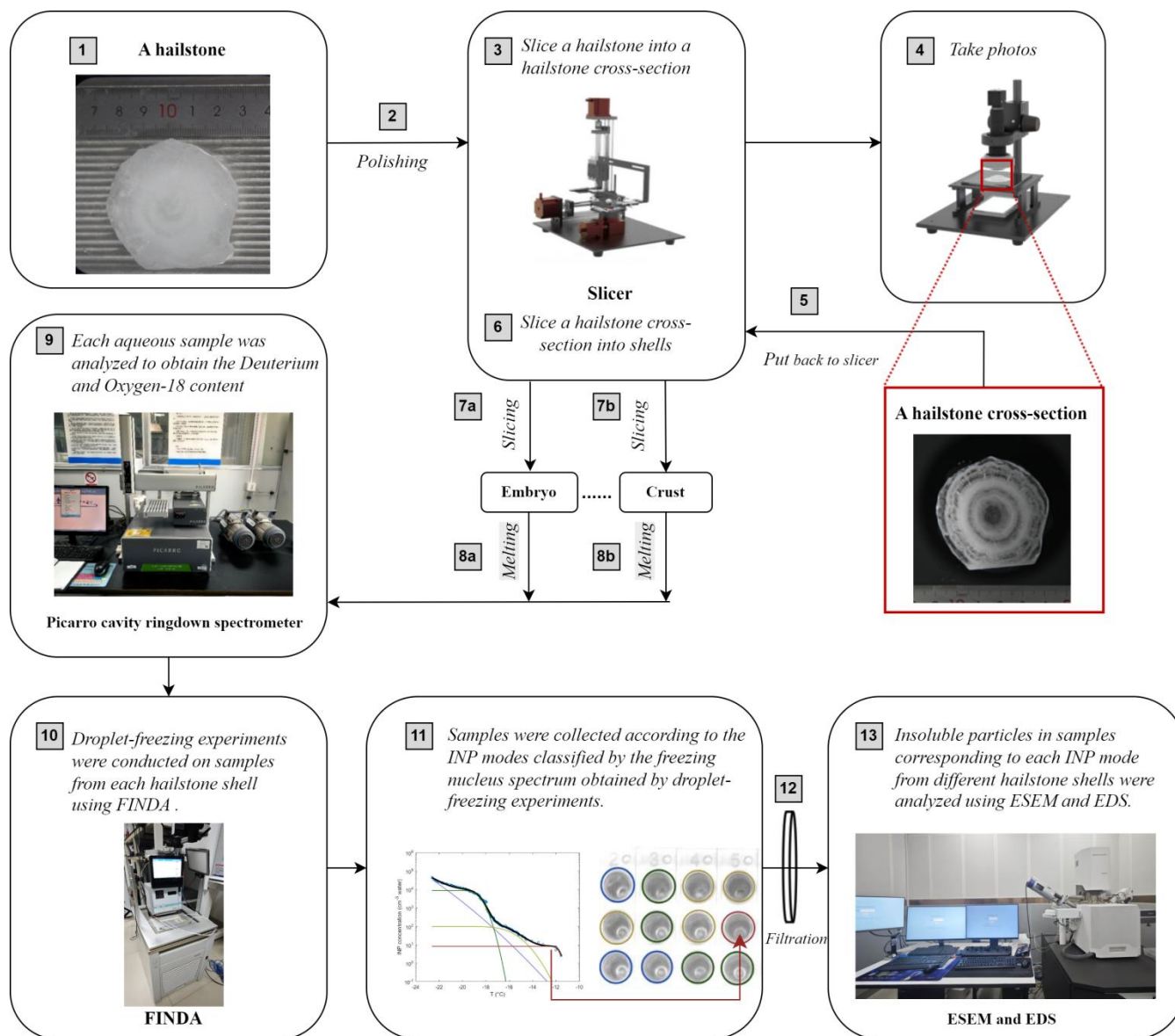


Figure 2: Diagram illustrating the analytical procedure (modified from Lin et al., 2025).

2.2 Hydrogen and Oxygen isotope measurement

125 The ratios of deuterium (D/H) and oxygen-18 ($^{18}\text{O}/^{16}\text{O}$) were measured using cavity ring-down spectroscopy (CRDS, L-2130i; Picarro, Santa Clara, CA, USA) and are reported as the differences relative to Vienna Standard Mean Ocean Water (VSMOW):

$$\delta\text{D} = \frac{(\text{D}/\text{H})_{\text{Sample}} - (\text{D}/\text{H})_{\text{VSMOW}}}{(\text{D}/\text{H})_{\text{VSMOW}}} \quad (1)$$



$$\delta^{18}\text{O} = \frac{(\frac{^{18}\text{O}}{^{16}\text{O}})_{\text{Sample}} - (\frac{^{18}\text{O}}{^{16}\text{O}})_{\text{VSMOW}}}{(\frac{^{18}\text{O}}{^{16}\text{O}})_{\text{VSMOW}}} \quad (2)$$

where VSMOW reference water contains 155.76 ± 0.05 ppm of D and 2005.20 ± 0.45 ppm of ^{18}O (Baertschi, 1976; Hagemann et al., 1970). Following the Adiabatic Model adopted by Lin et al. (2025), the isotope ratio $R_c(T)$ of the condensed water at various heights (i.e., at different temperatures T) can be expressed as:

$$R_c(T) = \frac{m_b R_b}{m_c(T) + m_v(T)/\alpha(T)} \quad (3)$$

where m_b and R_b represent the water content and its isotope ratio at the cloud base, respectively, and are assumed to remain constant for a given storm. The quantities $m_c(T)$ and $m_v(T)$ denote the contents of condensed water and water vapor, respectively, and $\alpha(T)$ is the isotope separation coefficient. The values of m_b , $m_c(T)$, and $m_v(T)$ can be given by the adiabatic curve derived from vertical profiles based on ERA5 reanalysis data, $\alpha(T)$ is given by an empirical formula derived by Majoube (1971), and R_b is calculated using the maximum and minimum isotope ratios measured in the hailstone samples, following Lin et al. (2025). The ambient temperature and altitude corresponding to each hailstone shell during growth were inferred by comparing the isotope composition of each shell of hailstone samples with the $R_c(T)$ profile. The vertical growth trajectory of the hailstone was then reconstructed from the inferred temperature-altitude relationship.

2.3 Droplet-freezing experiment

The Freezing Ice Nucleation Detector Array (FINDA) was used to perform the droplet-freezing experiment. To measure the INP number concentration at both higher and lower temperatures, each shell of hailstone solution was divided into the original solution group and the diluted solution group for the droplet-freezing experiment. The ultrapure water used for dilution was obtained by filtering distilled water with a $0.02 \mu\text{m}$ filter. Before conducting the hailstone solution experiment, a droplet-freezing experiment was first performed on ultrapure water to ensure that the dilution effect on the hailstone solution was negligible.

The experimental process of the droplet-freezing experiment was as follows: first, the experimental samples were mixed with a vortex oscillator for 1 minute to ensure uniform sampling. Then, the well-mixed hailstone solution was pipetted into droplets with a volume of $10 \mu\text{L}$ and added to the 92 available wells of the sampling plate. A total of 92 droplets were used for each set of experiments. Specifically, due to the limited sample quantities in the shell A group with a dilution factor of 2 and the shell C group with a dilution factor of 100, 78 droplets were used for these two sets of experiments. Next, the refrigeration system reduced the temperature of the sampling plate from room temperature (approximately 20°C) to 0°C . Because the plate lagged the set temperature during the cooling process, the system was maintained at 0°C for 10 minutes to allow the overall temperature of the enclosed space where the sample array was located to drop to 0°C . Then, the



155 temperature of the sample array decreased from 0 °C to -40 °C at a cooling rate of 1 °C/min. After the freezing process was completed, the sampling plate was removed, covered with an identical empty sampling plate on top and then placed into a sealed bag to minimize exposure of frozen droplets to the external environment. The sample was subsequently stored in a freezer maintained at -25 °C.

During the experiment, a charge-coupled device camera captured images of the droplets at 0.2-second intervals to monitor their phase state. A freezing event was identified as a sudden drop in the droplet luminance. Thus, the freezing ratio $f(T)$, which is defined as the fraction of frozen droplets $N_f(T)$ relative to the total number of droplets N_d at each temperature, can be obtained:

$$f(T) = \frac{N_f(T)}{N_d} \quad (4)$$

According to Vali (1971), the cumulative INP concentration active above temperature T can be calculated as

$$C_{INP}(T) = -\frac{\ln(1-f(T))}{V_d} \cdot DF \quad (5)$$

where V_d is the volume of each droplet with a value of 10 μL , and DF denotes the dilution factor.

165 The freezing nucleus spectrum is fitted to directly characterize INP modes. Following de Almeida Ribeiro et al. (2023), the differential freezing nucleus spectrum of each INP mode can be represented by a Gaussian distribution,

$$D_{INP,i}(T) = \frac{N_i}{\sigma_i \sqrt{2\pi}} \exp \left[-\frac{(T-T_i)^2}{2\sigma_i^2} \right] \quad (6)$$

where the subscript i denotes the i_{th} mode; N_i represents the total INP concentration of the INP mode; T_i is the characteristic temperature corresponding to the most probable freezing temperature, where the freezing ratio increases most rapidly; σ_i quantifies the dispersion of the freezing temperature distribution. The cumulative freezing nucleus spectrum for the i_{th} mode is calculated as

$$C_{INP,i}(T) = \int_T^{T_0} D_{INP,i}(T) \quad (7)$$

where $T_0 = 0$ °C. The cumulative freezing nucleus spectrum for all INP modes is calculated as

$$C_{INP_ALL,k}(T) = \sum_{i=1}^k C_{INP,i}(T) \quad (8)$$

where k is the total number of INP modes.

The number of INP modes corresponding to each hailstone shell can be determined from the scatter plot of $C_{INP}(T)$ obtained through the droplet-freezing experiments. The experimental data are fitted according to Eq. (6)-(8) to obtain the three best-fit



175 parameters N_i , σ_i and T_i corresponding to the i_{th} INP mode, so that the fitting curve of each mode and the overall curve of each hailstone shell are obtained.

An INP mode was considered dominant when it contributed the largest fraction to the cumulative INP concentration at a given temperature (Ren et al., 2024). The intersections of the fitting curves for different INP modes were used to divide the freezing nucleus spectrum into several temperature intervals, each dominated by a different INP mode. Accordingly, each
180 frozen droplet was assigned to the INP mode corresponding to the temperature interval in which it froze.

2.4 Measurements of insoluble particle species/size distribution

After the fitting curves of the freezing nucleus spectra were determined, all the droplets assigned to the same INP mode within each hailstone shell were melted and collected separately from the sample plate using a pipette. For each dilution factor, the masses of the collected samples belonging to the same INP mode were measured and converted to the volumes of
185 the original hailstone solution before dilution. The solution corresponding to each INP mode was then filtered using a syringe through a mixed cellulose ester filter membrane (composed of C, H, N, and O) with a diameter of 13 mm and a pore size of 25 nm. To maximize the recovery of insoluble particles onto the membrane, the syringe and membrane were rinsed three times with 1 mL of ultrapure water per rinse, resulting in a total rinsing volume of 3 mL. The membranes were subsequently dried at approximately 40 °C.

190 The number, elemental composition, and sizes of insoluble particles were determined by examining filter membranes using ESEM and EDS following the method described in Zhang et al. (2023). Due to the large number of insoluble particles on each membrane, random fields of view were selected for scanning to improve analytical efficiency while maintaining sample representativeness. The scanning time for each filter membrane was set to 3 hours.

Following the classification method of Zhang et al. (2023), the insoluble particles were classified into four categories:
195 organics, dust, bioprotein, and Fe–Cr alloys particles. Among these, Fe–Cr alloys particles were attributed to contamination introduced during hailstone sectioning and were therefore excluded from subsequent analyses. The number concentration of insoluble particles n_{hail} was calculated as

$$n_{hail} = \frac{S_{membrane}}{N_{site} \cdot W_{site} \cdot L_{site}} \frac{N_{scanned}}{V} \quad (9)$$

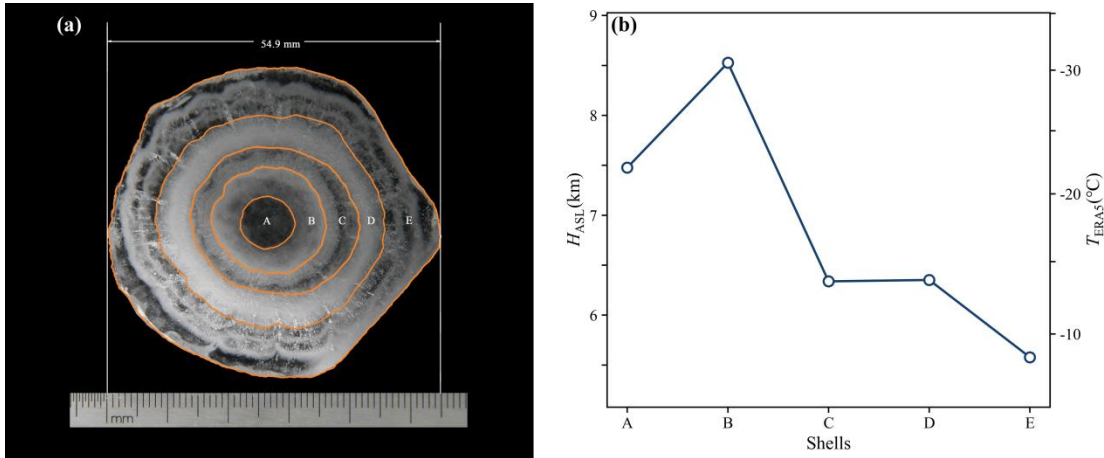
where $S_{membrane}$ is the area of the filter membrane, N_{site} is the number of scanned fields of view, W_{site} and L_{site} are width and length, respectively, of a single scanned field of view, $N_{scanned}$ is the number of scanned particles, and V is the volume of the
200 original hailstone solution corresponding to the sample solutions. Since the solutions subjected to filtration were prepared using different dilution factors, V was calculated from the volume of each diluted solution V_i and its corresponding dilution factor DF_i , as follows

$$V = \sum_i \frac{V_i}{DF_i} \quad (10)$$

3 Results

3.1 Sample information

205 The hailstone cross-section under natural light (Fig. 3a) reveals a transparent frozen droplet embryo at the center. Five shells were distinguished based on their transparency or opacity, and were sequentially labeled with shell A to shell E from the embryo to the edge.



210 **Figure 3: Photograph of the hailstone cross-section (a) and vertical growth trajectory (b) reconstructed from isotopic analyses. Orange lines in (a) divide the hailstone shells with annotations from A-E. Left and right ordinates in (b) are height above sea level (H_{ASL}) and temperature (T_{ERAS}).**

3.2 Isotopic analyses

The vertical growth trajectory of the hailstone was reconstructed by comparing the measured δD and $\delta^{18}O$ of each shell with their profiles derived from the Adiabatic Model (Lin et al., 2025). D/H value was used to estimate the growth altitude of each shell because it has a smaller relative uncertainty than $^{18}O/^{16}O$ (Lin et al., 2025). The results are shown in Table 1.

Table 1. Results of stable isotope analyses of hailstone shells and vertical growth trajectory of the hailstone.

	$\delta D(\text{‰})$	$\delta^{18}O(\text{‰})$	$T_{ERAS}(\text{°C})$	$H_{ASL}(\text{m})$
Shell A	-104.23	-15.10	-22.0	7477
Shell B	-110.24	-15.89	-30.7	8527
Shell C	-95.79	-14.00	-13.6	6338
Shell D	-95.90	-14.08	-13.7	6352
Shell E	-89.42	-13.25	-8.5	5578



220 The growth zone of the hailstone spanned a wide temperature range from -8.5°C to -30.7°C , corresponding to an
 approximate vertical range of 3 km. Overall, the hailstone embryo formed at an altitude of approximately 7.5 km, and
 subsequently developed along an up-down trajectory (Fig. 3b). According to Lin et al. (2025), the ascending growth
 trajectory is essential for the generation of large hailstones. This is consistent with the growth trajectory reconstructed for the
 hailstone examined in this study, which had a maximum hail dimension of 54.9 mm. Notably, the inferred formation
 225 temperature of the hailstone embryo was -22.0°C , considerably lower than the corresponding temperature of the other three
 hailstones collected from the same storm analyzed by Lin et al. (2025) (Fig. 1), which were approximately -10°C . The
 following droplet-freezing experiments and insoluble particles characterization provide microphysical insights and
 explanations for this observation.

3.3 Droplet-freezing experiment

230 3.3.1 Freezing temperature

The relationship between the freezing ratio and temperature obtained from all droplet-freezing experiments is shown in Fig.
 4. Freezing events in the ultrapure water sample occurred within a low temperature range from -19.35°C to -27.69°C . Only
 one of the 92 droplets froze at a temperature above -20.00°C , at -19.35°C . This result is consistent with the freezing
 temperature range reported for the ultrapure water used by Ren et al. (2024) ($-21.0\pm 2.0^{\circ}\text{C}$ to $-28.5\pm 0.5^{\circ}\text{C}$). For the hailstone
 235 samples at different dilution ratios, the onset temperatures of freezing ranged from -16.34°C to -5.40°C , and the termination
 temperatures ranged from -25.76°C to -13.75°C . The freezing temperatures of the hailstone samples were substantially
 higher than those of ultrapure water (Fig. 4), indicating that the effect of the ultrapure water used for dilution in this
 experiment was negligible. Notably, the initial freezing temperature of the embryo sample (shell A) was -11.56°C ,
 substantially lower than those of outer shells ranging from -6.25°C to -5.40°C .

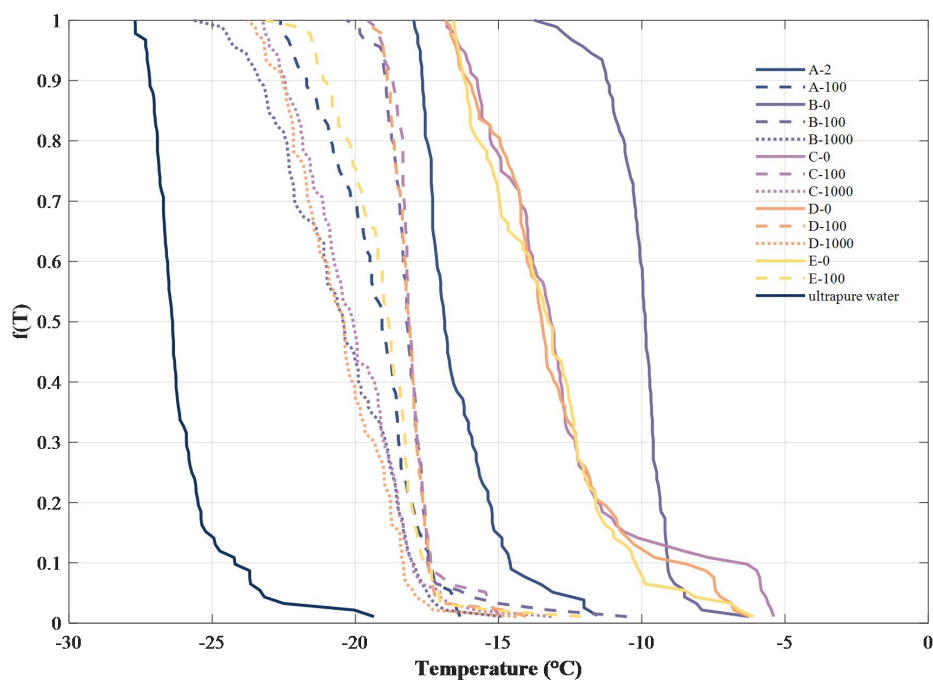


Figure 4: The relationship between the freezing ratio and temperature obtained from all droplet-freezing experiments. The letters A-E in the legend represent the hailstone shells shown in Fig. 3a. The following numbers represent the dilution factor.

3.3.2 Freezing nucleus spectra and INP modes

Following the fitting method in Sect. 2.3, freezing nucleus spectra for the five hailstone shells were obtained over a wide temperature range (Fig. 5). The fitting parameters for each INP mode of the corresponding hailstone shell are presented in Table 2.

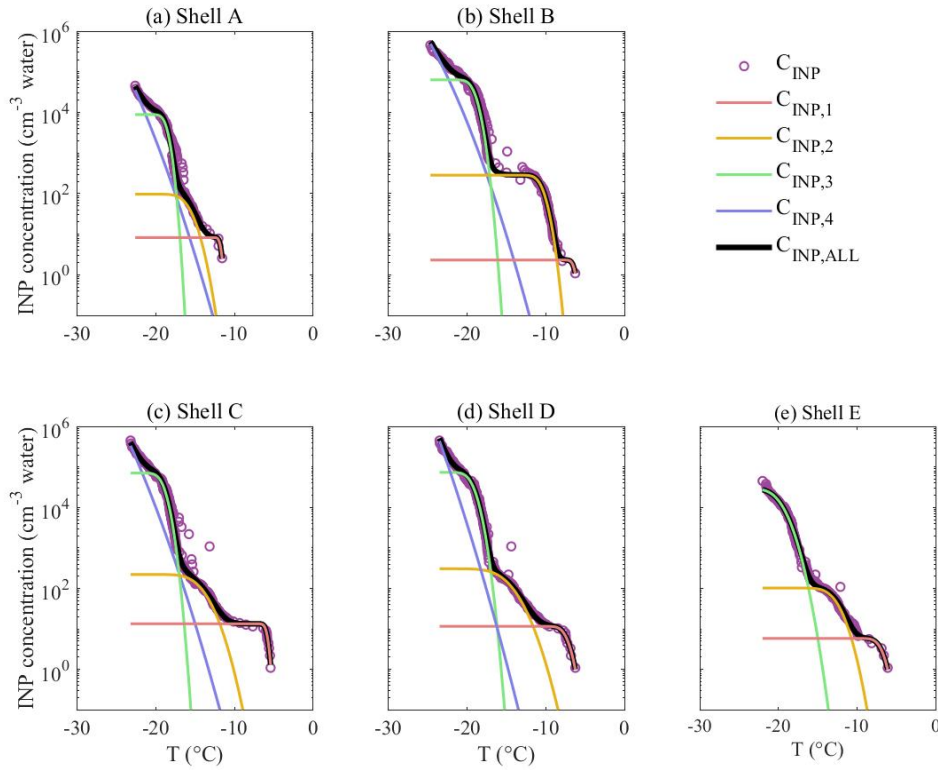


Figure 5: The freezing nucleus spectra for the five hailstone shells. Data points of measured cumulative INP concentrations are marked with purple circles. Lines are the best-fit freezing nucleus spectra, including cumulative freezing nucleus spectra for each INP mode (red, yellow, green and blue lines) arranged by temperature in descending order, and cumulative freezing nucleus spectra for all INP modes (black lines).

Four INP modes were required to achieve good fits to the freezing nucleus spectra of hailstone shells A-D (Fig. 5a-d), whereas a maximum of three INP modes was sufficient to fit the cumulative freezing spectra of all hailstones in Ren et al. (2024). This suggests that analyzing individual hailstone shells can resolve additional INP modes and reveal more detailed information about the freezing history of hailstone formation. In addition, shell E displays only three INP modes (Fig. 5e), indicating that the number of INP modes varied among the different hailstone shells. The INP modes are numbered by their dominant temperature range from highest to lowest temperature (Fig. 5 and 6).

The total INP concentrations, N_i , of Mode 1 were all no greater than 10 cm^{-3} , and correspondingly few droplets froze within this temperature range during the experiment. Based on these characteristics, Mode 1 of shells A-E was classified as the high-temperature (HT) INP mode. Mode 2 had the total INP concentrations range from 10^1 to 10^2 cm^{-3} and characteristic temperatures ranging from $-15.90 \text{ }^\circ\text{C}$ to $-10.51 \text{ }^\circ\text{C}$. It was therefore classified as the medium-high-temperature (MHT) INP mode. Mode 3 had the total INP concentrations in the range from 10^3 to 10^4 cm^{-3} and characteristic temperatures ranging



from -20.00 °C to -18.67 °C, and was classified as the medium-low-temperature (MLT) INP mode. Mode 4, which was identified in shells A-D, had total INP concentrations greater than 10^8 cm^{-3} and characteristic temperatures below -30 °C. It was therefore classified as the low-temperature (LT) INP mode. Notably, the characteristic temperatures T_l , for the HT mode were above -10 °C in all shells except shell A (embryo). This pattern is consistent with the approximately 5-6 °C increase in onset freezing temperatures from embryo to outer shells.

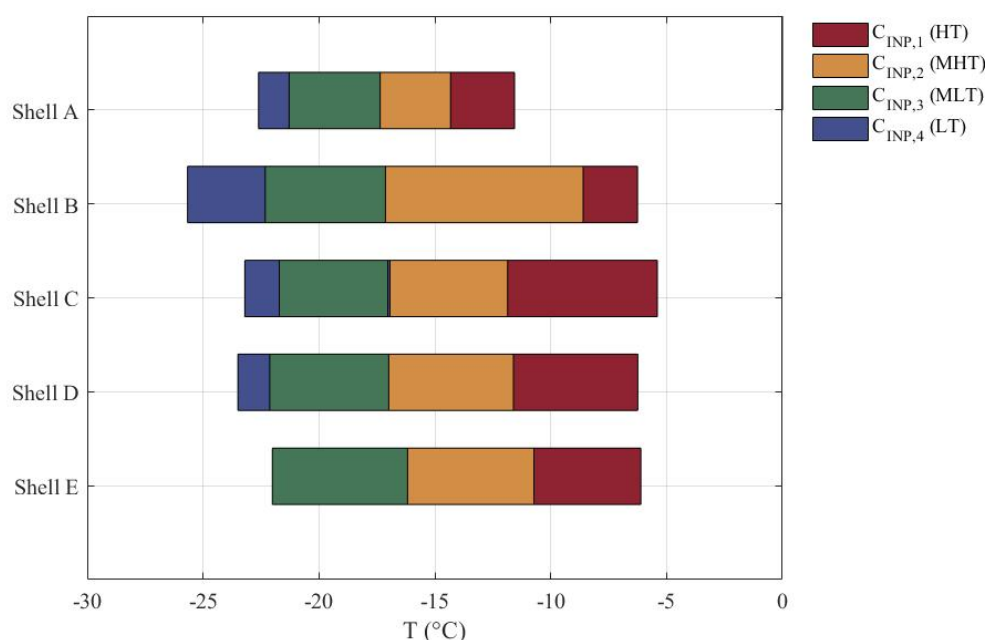


Figure 6: The dominant INP mode in cumulative freezing nucleus spectra over the experiment temperature range for the five hailstone shells. The little gap in shell C between MHT mode and MLT mode is the blue shaded LT mode.

The total INP concentration of the LT mode is at least four orders of magnitude higher than those of the other modes. According to Eq. (5), the cumulative INP concentration, C_{INP} , increases with the dilution factor. Therefore, when $C_{INP} \gg 10^4 \text{ cm}^{-3}$, the freezing spectrum may be dominated by the LT mode, consequently the other modes may no longer be resolved. This suggests that the LT mode is likely an essential component of the complete freezing spectrum, and that the mode not resolved for shell E may occur if experimental data with higher dilution factor are included. The high INP concentration of LT mode may also obscure other INP modes. For example, the LT mode identified in shell C dominated a narrow temperature range of approximately 0.1 °C between the two medium-temperature INP modes (-17.03 °C to -16.93 °C) (Fig. 6).

The combined freezing spectra of the five hailstone shells are shown in Fig. 7. The freezing spectra of shells C and D show substantial overlap, especially at temperatures below -10 °C. This is consistent with the nearly identical formation temperatures of shells C and D obtained by isotopic analyses in Sect. 3.2 (-13.6 °C and -13.7 °C, respectively), indicating



that they experienced similar growth environments. In addition, the relatively low onset freezing temperature of shell A (-
285 11.56 °C) is consistent with its relatively low formation temperature (-22.0 °C), as determined in Sect. 3.2.

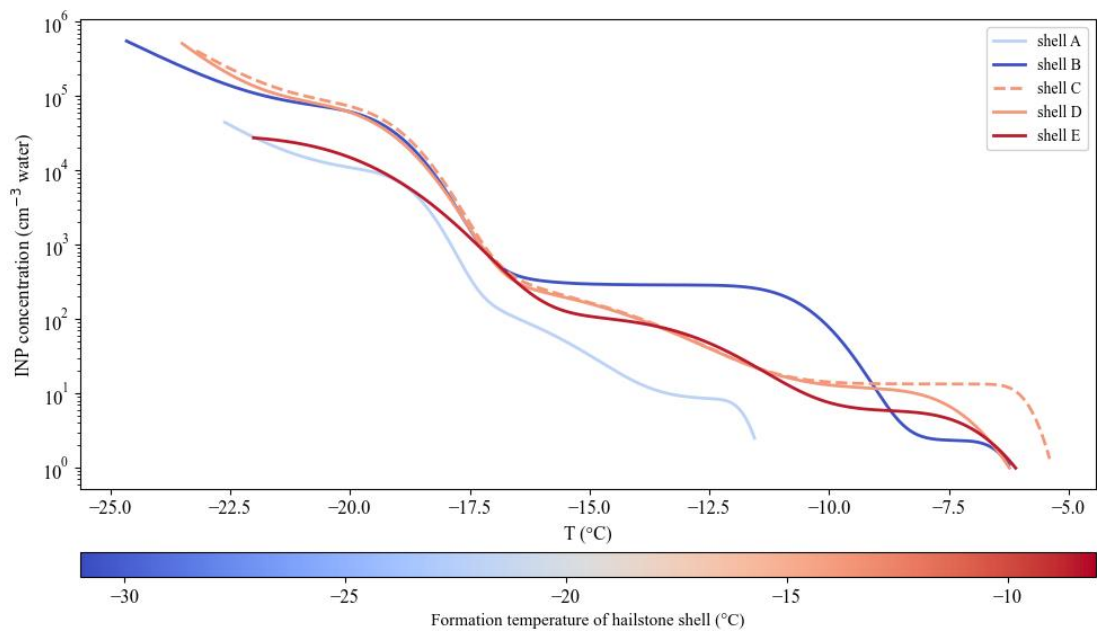


Figure 7: The combined freezing nucleus spectra of five hailstone shells. The colors represent the formation temperature of each hailstone shell, derived from isotopic analyses.

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Table 2 Fitting parameters for each INP mode of hailstone shells

		Mode 1	Mode 2	Mode 3	Mode 4
Shell A***	N_i	8.32	9.73×10^1	8.88×10^3	6.62×10^8
	σ_i	0.27	1.16	0.56	4.07
	T_i (°C)	-11.71	-15.90	-18.67	-38.36
Shell B***	N_i	2.32	2.85×10^2	6.34×10^4	1.24×10^8
	σ_i	0.38	0.80	0.76	3.73
	T_i (°C)	-6.28	-10.51	-19.12	-34.56
Shell C***	N_i	1.34×10^1	2.22×10^2	7.10×10^4	7.73×10^9
	σ_i	0.33	1.66	0.76	4.14
	T_i (°C)	-5.83	-14.43	-19.09	-39.43
Shell D***	N_i	1.16×10^1	3.06×10^2	7.41×10^4	4.72×10^{12}
	σ_i	0.80	1.95	0.88	4.29



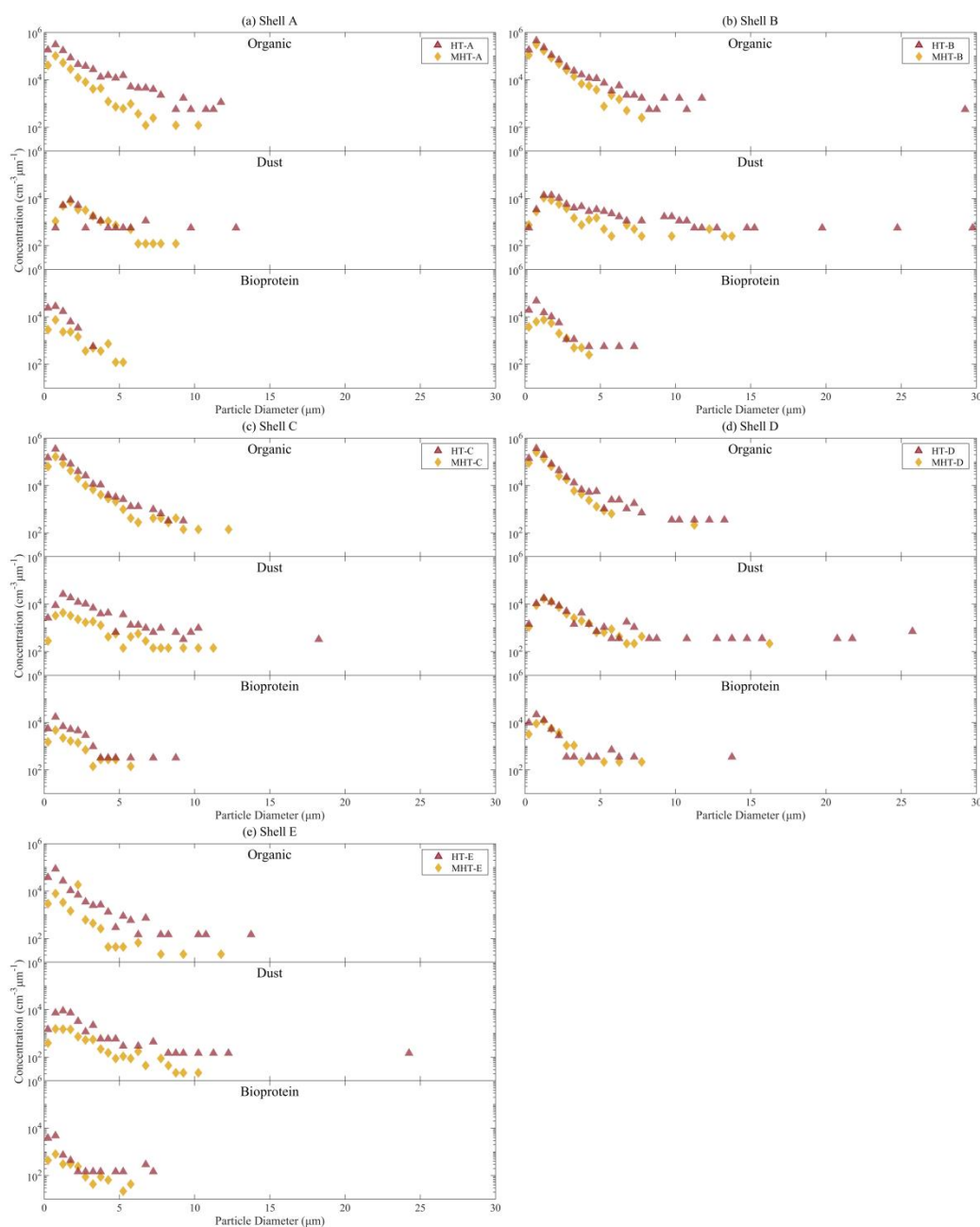
	T_i (°C)	-7.33	-15.07	-19.36	-45.85
	N_i	5.81	1.02×10^2	2.97×10^4	
Shell E***	σ_i	0.80	1.32	1.41	
	T_i (°C)	-6.87	-12.81	-19.99	

*** $p < .001$ based on an overall F-test on the log scale

3.4 Characteristics of insoluble particles

295 A total of 36,411 insoluble particles in samples from each mode (HT, MHT, MLT and LT) of five hailstone shells were analyzed using ESEM and EDS. Following the method of Zhang et al. (2023), a random forest algorithm was used to classify the particles into four types: Fe-Cr alloy particles, organics, dust, and bioprotein particles. Excluding the 663 Fe-Cr alloy particles, the remaining particles include 30,865 organics, 2,853 dust, and 2,030 bioprotein particles.

300 In the droplet-freezing experiment, the original hailstone solutions froze completely within the temperature ranges dominated by the HT and MHT modes. These two modes therefore primarily represented the original solution and best reflected its particle characteristics. In contrast, the samples corresponding to the MLT mode and the LT mode were subject to greater uncertainty due to dilution. Therefore, we analyzed the size distribution of organics, dust and bioprotein particles from the HT and MHT modes of each hailstone shell, as shown in Fig. 8.



305 **Figure 8: Size-resolved particle number concentration distributions of organics, dust, and bioprotein particles within hailstone shell A-E. Purple triangles represent the HT mode; yellow diamonds represent the MHT mode.**

The number concentrations of insoluble particles in the HT and MHT modes range from 10^4 to 10^6 cm^{-3} , and the differences among the hailstone shells were within one order of magnitude. Overall, the insoluble particles in the HT mode exhibited



higher or comparable number concentrations and larger sizes than those in the MHT mode. This suggests that higher number concentrations and larger sizes of insoluble particles promoted freezing at relatively high temperatures.

This size- and concentration-dependent effect extended to lower temperatures. At approximately -20°C , a regime in which dust is generally considered the dominant immersion-freezing type of INP at temperatures, (Ladino et al., 2019; Murray et al., 2012; Si et al., 2019). The cumulative INP concentrations of shells B, C, and D were substantially higher than those of shells A and E (Fig. 7). Consistent with this, shells B, C, and D exhibited higher number concentrations of dust particles and a greater abundance of large dust particles ($> 10\ \mu\text{m}$) compared to shells A and E (Fig. 9a). These findings suggest that dust with larger particle sizes and higher number concentrations may enhance the ice nucleation efficiency of supercooled water droplets at approximately -20°C .

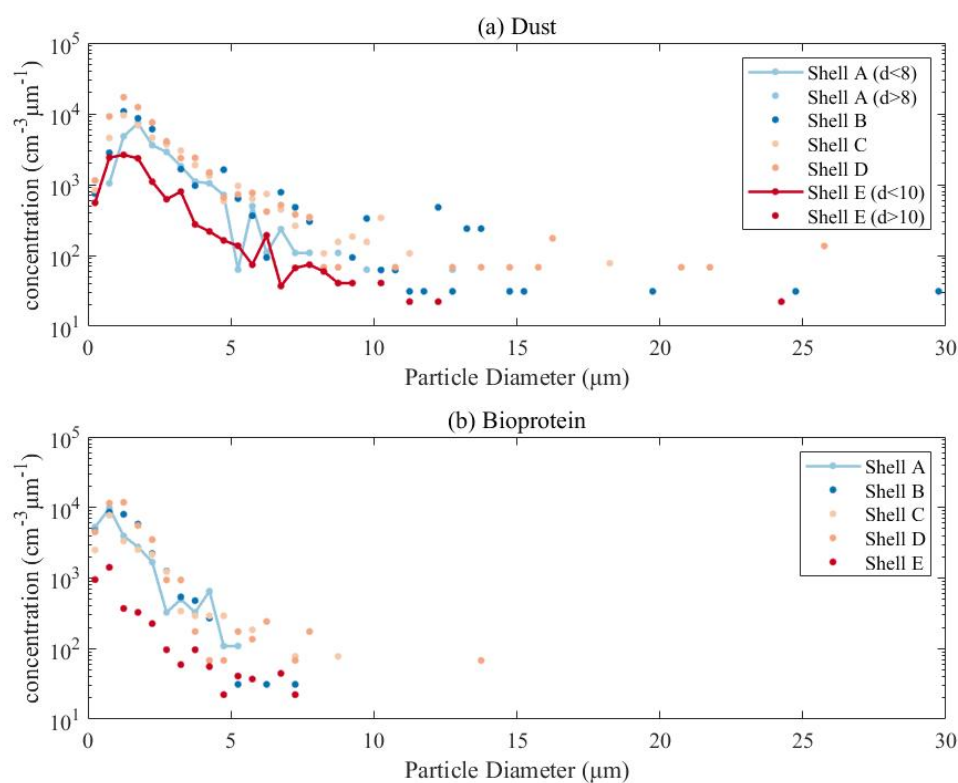


Figure 9: Size distribution of (a) dust and (b) bioprotein particles in the original solutions from each hailstone shell. In (a), dust particle concentrations for Shell A (at intervals of $0.5\ \mu\text{m}$ below $8\ \mu\text{m}$) are represented by a light blue line connecting data points, while individual light blue dots denote the presence of particles with diameters larger than $8\ \mu\text{m}$. Similarly, for Shell E, a red line connecting data points represents particles with diameters smaller than $10\ \mu\text{m}$, and individual red dots show larger particles ($d > 10\ \mu\text{m}$). Data for Shells B (dark blue), C (light orange), and D (orange) are plotted as data points throughout the entire size range. In (b), data for Shell A are connected by a line, with data for all other shells shown as dots.



In the hailstone embryo (shell A), the largest bioprotein particle observed in the HT mode was only 3.14 μm in diameter, and only one particle larger than 5 μm (5.32 μm) was detected in the MHT mode. In contrast, bioprotein particles exceeding 5 μm were consistently observed in both INP modes of the outer shells. Given that bioprotein particles are widely recognized as the dominant type of immersion freezing INP at temperatures above -15 $^{\circ}\text{C}$ (Chen et al., 2021a, b; Hartmann et al., 2020; Hill et al., 2014; Joyce et al., 2019; Ladino et al., 2019; Šantl-Temkiv et al., 2019), the scarcity of large bioprotein particles in the hailstone embryo may partly explain both its relatively low onset freezing temperature in the droplet-freezing experiments and its relatively low formation temperature derived from isotopic analyses.

4 Conclusions

Owing to the challenge of directly tracking and observing hailstones which form in the extreme environment of strong convective systems, current understanding of hailstone freezing dynamics remains constrained. This study presents the first combined laboratory investigation integrating isotopic analyses, droplet-freezing experiments and insoluble particle species/size distribution on an individual hailstone, thereby providing a comprehensive assessment of hailstone freezing characteristics and the role of insoluble particles therein.

A large hailstone sample (frozen droplet embryo) with a diameter of 54.9 mm collected from Yantai was sliced and divided into five shells. Isotopic analyses revealed that the hailstone grew over a wide temperature range from -30.7 $^{\circ}\text{C}$ to -8.5 $^{\circ}\text{C}$, exhibiting an alternating up-down trajectory. Notably, the formation temperature of the hailstone embryo was -22.0 $^{\circ}\text{C}$, which is relatively low compared to other embryos from the same hailstorm (around -10 $^{\circ}\text{C}$) reported by Lin et al. (2025).

The cumulative freezing nucleus spectra of hailstone shells required four INP modes to achieve satisfactory fits, in contrast to a maximum of three modes reported in Ren et al. (2024). This indicates that analyzing individual hailstone shells separately makes it possible to capture more detailed freezing characteristics. The onset freezing temperature of the embryo sample was -11.56 $^{\circ}\text{C}$, showing 5-6 $^{\circ}\text{C}$ lower than the outer shells, which corresponds with the low formation temperature (-22.0 $^{\circ}\text{C}$) derived from isotopic analyses.

Analyses of the insoluble particles in the HT and MHT modes of each hailstone shell were carried out to obtain the size-resolved number concentration of three particle species: organics, dust, and bioprotein particles, all of which varied across different INP modes. Large particles sizes and higher concentrations were observed in HT mode, suggesting that these particles promote hailstone freezing at higher temperatures. The hailstone embryo lacked bioprotein particles with diameters larger than 6 μm , which may explain the low onset freezing temperature in the droplet-freezing experiment and the low formation-temperature inferred from isotopic analyses. The consistent results from isotopic analyses, droplet-freezing experiments, and insoluble particle characterization collectively validate the integrated experimental approach for unraveling the microphysical processes of hailstone formation.

Despite the study's limitation to a single hailstone sample, which constrains the generality of its conclusions, the results demonstrate the potential of combined experimental approaches to provide multidimensional insights into the microphysical processes of hailstone formation. Future work should extend such combined approaches to a broader range of hailstorms



across different countries and regions, which is essential for statistically establishing the relationship between hailstone
360 growth mechanisms and the role of insoluble particles. In addition, the impact of organics on freezing appeared to be limited
under the experimental conditions of this study, with no clear effect detectable. Further combined-technique research is
required to better understand the influence of insoluble particles on ice nucleation.

Code and data availability

The code implementing the Adiabatic Model used to reconstruct the vertical growth trajectories of hailstones (Sect. 2.2) and
365 the random forest algorithm used in Sect. 3.4 are available upon request from the corresponding authors. All data are
likewise available upon request from the corresponding authors, except ERA5 reanalysis data, which are publicly available
from the Copernicus Climate Data Store (<https://cds.climate.copernicus.eu>).

Author contributions

QZ conceived the study, ZW and XYL designed the experiments and carried them out. XFL, KB, HZ and HX provided the
370 resources required for the laboratory analyses. ZW wrote the manuscript draft. QZ, XYL and JS reviewed and edited the
manuscript.

Competing interests

The contact author has declared that none of the authors has any competing interests.

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