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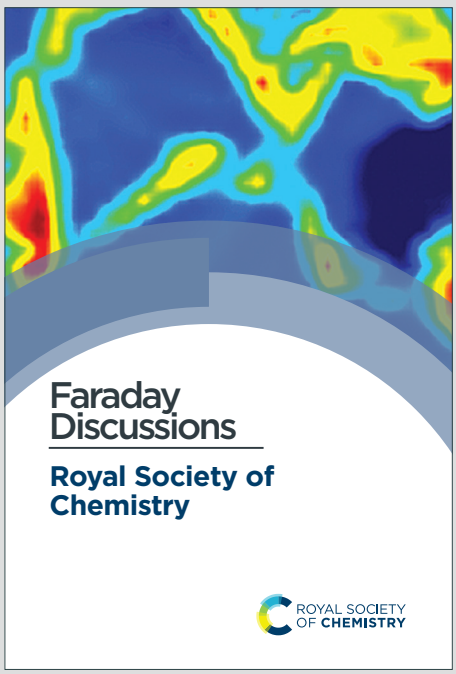

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PAPER

Decoupling Amine Evolution from Sulfur Delivery in High-Temperature Metal Sulfide Nanocrystal Synthesis

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Thiourea and its N,N′-substituted derivatives are widely employed as sulfur precursors in the colloidal synthesis of metal sulfide nanomaterials. Here we demonstrate that these precursors exhibit an intrinsic limitation at elevated temperatures: prior to H_2S evolution, they release reactive amines that perturb nucleation and growth. In situ mass spectrometry reveals that above 160 °C, amine evolution precedes sulfur delivery and coincides with dissolution or suppression of crystalline SrS formation in a model reaction. Mechanistic analysis further shows that all investigated thioureas converge to N,N′-dioleylthiourea (DOlTU) as a common intermediate before H_2S release. By directly employing DOlTU as a sulfur precursor, we decouple amine evolution from sulfur supply and establish a continuous, temperature-dependent nanocrystal growth profile up to 250 °C. These findings identify precursor decomposition chemistry as a decisive parameter in high-temperature metal sulfide synthesis and provide a rational strategy for designing sulfur sources that enable predictable and reproducible nanocrystal formation for optoelectronic materials.

Introduction

Understanding and controlling precursor reactivity is central to the synthesis of nanomaterials with tailored structural and optoelectronic properties. In colloidal routes toward metal sulfide nanocrystals, the temporal availability of reactive sulfur species governs nucleation kinetics, growth pathways, and ultimately phase, morphology, and defect density. Despite this central role, sulfur precursor selection is often guided by empirical considerations such as solubility or convenience, while the detailed thermal decomposition chemistry of the precursor receives comparatively little attention.

Thiourea and its N,N′-substituted derivatives are among the most widely employed sulfur precursors in colloidal metal sulfide synthesis. Their appeal stems from the formal –II oxidation state of sulfur, synthetic accessibility, and compatibility with coordinating solvents such as amines. However, in non-aqueous syntheses, thioureas do not directly deliver free sulfide ions. [1] Instead, sulfur release is governed by multistep thermal decomposition pathways that generate transient intermediates and gaseous byproducts that exhibit different chemistry than other precursors like elemental sulfur,[2] thiols,[3] thiocarbamates,[4] or other sulfur containing precursors.[5] Because these precursors are typically used in excess, their decomposition chemistry effectively defines the reactive window of the entire synthesis.

Though there are numerous studies using thiourea[6] and some derivatives like N,N′-diethylthiourea,[7] N,N′-dibutylthiourea,[8] N,N′-diisopropylthiourea,[9] a largely overlooked aspect of thiourea-based systems is the evolution of amines during thermal decomposition at elevated temperatures. In strongly coordinating media such as oleylamine, the release of additional amine species introduces competing equilibria that can alter metal–ligand interactions, solubility of intermediates, and surface passivation of nascent nanocrystals. Such perturbations are expected to influence nucleation and growth, particularly in high-temperature syntheses where kinetic control is critical for achieving phase-pure and defect-controlled materials relevant to optoelectronic applications. Yet the direct impact of precursor-derived amine evolution on nanocrystal formation remains insufficiently understood.

In this work, we examine the decomposition behaviour of thiourea and representative N,N′-substituted derivatives under conditions relevant to high-temperature colloidal synthesis. Using in situ mass spectrometry, we show that above 160 °C these precursors release reactive amines prior to H_2S evolution. Employing strontium sulfide (SrS) as a model system with potential uses in energy storage [10] and optoelectronics [11,12], we demonstrate that this amine evolution coincides with suppression or dissolution

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of crystalline material, leading to discontinuous and poorly reproducible growth behaviour. Combined with mechanistic studies of thiourea decomposition in oleylamine in the literature, [13] these observations support a model in which the investigated thioureas converge through N,N′-dioleylthiourea (DOlTU) prior to the later sulfur-release step. Recognizing DOlTU as the decisive intermediate enables a rational redesign strategy: by directly employing DOlTU as the sulfur precursor, amine evolution is decoupled from sulfur delivery. We show that this approach establishes a continuous and temperature-dependent nanocrystal growth profile across an extended reaction window. More broadly, our findings identify precursor decomposition pathway engineering as a key lever for achieving predictable and reproducible high-temperature synthesis of metal sulfide nanomaterials, with direct implications for the controlled fabrication of emerging optoelectronic materials.

Methods

Materials. Thiourea (99%) was purchased from Carl Roth. N,N′-dimethylthiourea (99%), oleylamine (technical grade), oleic acid (technical grade), toluene (99.5%), hexane (99%), and ethanol (99%) were purchased from Sigma-Aldrich. Strontium iodide (99.99%) was purchased from Thermo Fisher Scientific. All chemicals were used as received without further purification. Silicon wafers with a native silicon oxide layer were purchased from Silicon Materials, and microscope glass slides are from Epredia.

N,N′-dioleylthiourea synthesis. Oleylamine (125 mL, 0.384 mol) and N,N′-dimethylthiourea (20.0 g, 0.192 mol) were combined in a 250 mL round-bottom flask equipped with a magnetic stir bar, thermocouple, and a gas outlet connected to a bubbler containing 2 M HCl to trap released methylamine. The reaction mixture was purged with nitrogen three times and subsequently heated to 225 °C under a nitrogen atmosphere with continuous stirring. The reaction was maintained at this temperature for 6 h.

After cooling to room temperature, the resulting yellow–brown viscous mixture was treated with excess methanol (1:10 v/v relative to the reaction mixture) and heated to 60 °C while stirring for 30 min to extract DOlTU into the methanol phase. The methanol phase was separated and cooled in a freezer, resulting in the formation of a white crystalline solid. The product was collected by filtration and dried under vacuum. The extraction procedure with methanol was repeated until no additional DOlTU was recovered from the oleylamine phase.

Strontium sulfide nanomaterial synthesis. In a typical synthesis, SrI_2 (425.0 mg, 1.25 mmol) and the sulfur precursor (thiourea: 475.8 mg, 6.25 mmol; or N,N′-dimethylthiourea: 651.1 mg, 6.25 mmol) were dispersed in oleylamine (7.5 mL) in a 25 mL three-neck round-bottom flask equipped with a thermocouple, septum, and stopcock. The reaction mixture was purged with nitrogen and heated to 200 °C at a ramp rate of 75 °C min^{-1}, unless otherwise specified, under a nitrogen atmosphere, where it was maintained for 5 min (or longer reaction times and higher temperatures as specified in the text). During heating, the mixture formed a white suspension.

After the reaction, the mixture was allowed to cool naturally to 80 °C, at which point oleic acid (3 mL) was added under nitrogen. The reaction was then further cooled to room temperature. The nanocrystals were isolated by precipitation through the addition of toluene as solvent and ethanol as antisolvent, followed by centrifugation at 7500 rpm (RCF ≈ 7560 m s^{-2}). This washing procedure was repeated twice. The final precipitate was redispersed in hexane (4 mL) and stored under nitrogen for further characterization.

Nuclear magnetic resonance (NMR) spectroscopy. ^{1}H NMR spectra were recorded on a Bruker Avance 400 MHz spectrometer equipped with a 9.4 T wide-bore magnet and an ATM broadband probe. Samples were dissolved in DMSO-d_6 and measured at room temperature.

X-ray diffraction (XRD). Powder X-ray diffraction patterns were recorded using a Bruker D8 Advance diffractometer equipped with a Cu Kα radiation source (λ = 1.5418 Å) operating in Bragg–Brentano geometry. The instrument was configured with primary optics providing a fixed sample illumination of 8 mm and a secondary optics slit size of 9.5 mm. Samples were prepared by drop-casting nanocrystal dispersions onto glass substrates and allowing the solvent to evaporate under ambient conditions.

Scanning electron microscopy (SEM). SEM images were acquired using a JEOL JSM-F100 microscope operated at an acceleration voltage of 10 kV. Samples were prepared by drop-casting diluted nanocrystal dispersions onto silicon wafers with a native oxide layer. Particle sizes were estimated manually using ImageJ. Because the nanocrystals often exhibit faceted or otherwise non-spherical morphologies, the reported sizes should be regarded as approximate values and are used primarily to compare relative growth trends between conditions rather than to assign precise geometric diameters.

Scanning transmission electron microscopy (STEM). STEM images were obtained using a JEOL JSM-F100 microscope operated at an acceleration voltage of 30 kV. Samples were prepared by drop-casting diluted nanocrystal dispersions onto Formvar-coated carbon films supported on 200-mesh copper TEM grids (Plano).

Transmission electron microscopy (TEM). TEM measurements were performed using a Philips CM30 microscope operated at an acceleration voltage of 300 kV. Samples were prepared by drop-casting diluted nanocrystal dispersions onto Formvar-coated carbon films on 200-mesh copper TEM grids (Plano).

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In situ quadrupole mass spectrometry (QMS). In situ mass spectrometry measurements were performed using a Hiden HPR-40 quadrupole mass spectrometer equipped with a heated QIC capillary inlet. Reaction mixtures were prepared by combining the respective thiourea precursor with oleylamine in a 1:4 ratio in a 25 mL three-neck round-bottom flask equipped with a thermocouple, septum, and stopcock. The flask was continuously flushed with N_2 during the experiment. Gaseous reaction products were monitored under standard operating conditions (70 eV ionization energy and 500 µA filament emission current) using a Faraday cup detector, as described previously.[14] Prior to entering the mass spectrometer, the hot gas stream was diluted with cold N_2 to prevent condensation within the capillary or instrument.

Results and Discussion

Thermal Decomposition Pathways of Thiourea Precursors. We first examined the thermal decomposition behaviour of thiourea and representative N,N′-substituted derivatives in oleylamine to establish the sequence of reactive species formation under conditions relevant to high-temperature nanocrystal synthesis. In situ quadrupole mass spectrometry reveals two distinct initial decomposition regimes depending on substitution pattern (Figure 1; detailed pathways in Figure S2). In the discussion below, the gas-evolution sequences are directly supported by the present QMS data, whereas the detailed condensed-phase sequence leading to DOlTU is a mechanistic assignment based on the combined evidence from this work and our previous complementary decomposition studies of thiourea derivatives in oleylamine. [13]

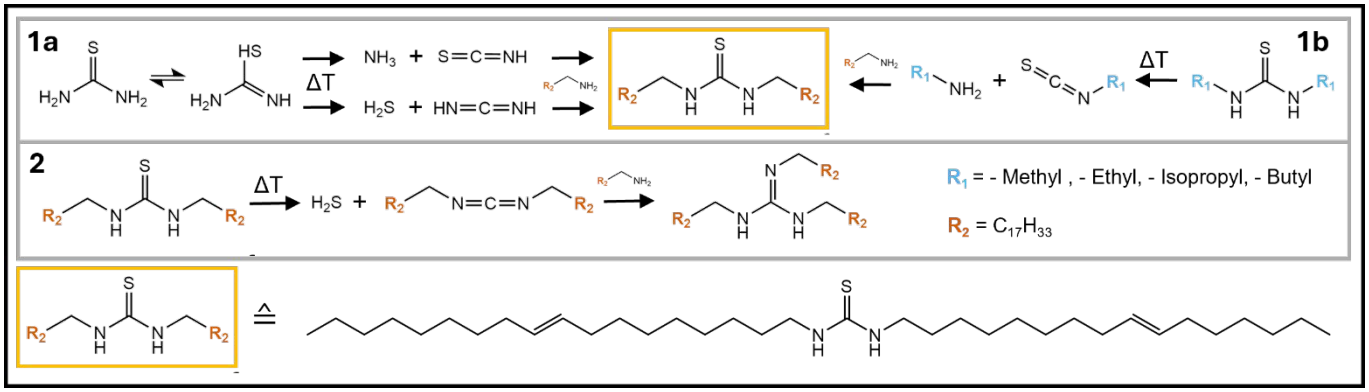

Figure 1. Proposed thermal decomposition pathways of thiourea (a) and N,N′-substituted thiourea derivatives (b) in oleylamine. While unsubstituted thiourea undergoes isomerization followed by simultaneous NH_3 and H_2S evolution, N,N′-substituted derivatives decompose stepwise, first releasing the corresponding amine (160–200 °C) prior to H_2S formation (~260 °C). Despite distinct initial steps, both pathways converge to N,N′-dioleylthiourea (DOlTU, highlighted), identified as the common intermediate preceding sulfur release. Subsequent decomposition of DOlTU generates H_2S and dioleylcarbodiimide, the latter reacting further to form 1,2,3-trioleylguanidine. Detailed pathways are provided in the Supporting Information.

Unsubstituted thiourea undergoes isomerization to isothiocyanic acid and decomposes near 200 °C with the simultaneous evolution of ammonia and H_2S, followed by a second H_2S release event at higher temperatures. In contrast, N,N′-substituted thioureas exhibit stepwise decomposition: cleavage of the C–N bond between 160–200 °C yields the corresponding amine (e.g., methylamine for N,N′-dimethylthiourea), while sulfur release in the form of H_2S occurs only at ~260 °C.

Despite these distinct initial steps, the available evidence supports mechanistic convergence of both precursor classes under the present reaction conditions. In this framework, the initially formed thiourea-derived fragments react with oleylamine to generate oleyl-substituted thiourea species that progress to N,N′-dioleylthiourea (DOlTU). DOlTU then enters the later high-temperature decomposition regime that releases H_2S and forms dioleylcarbodiimide, the latter reacting further with oleylamine to produce 1,2,3-trioleylguanidine. Accordingly, the combined data support DOlTU as the key operative intermediate preceding sulfur delivery. This convergence suggests that the decisive step governing sulfur release is not the initial thiourea structure itself, but the chemistry of DOlTU formed in situ.

Amine Evolution as a Perturbation of Nanocrystal Growth. To assess the synthetic consequences of these decomposition pathways, we employed strontium sulfide (SrS) nanocrystal formation as a model reaction. Strontium sulfide was selected as a model system because its nucleation and growth are highly sensitive to sulfur activity, amine chemistry, and ligand environment, allowing perturbations in precursor decomposition chemistry to be directly and easily observed. In situ mass spectrometry during

nanocrystal synthesis confirms that the decomposition behaviour observed in neat oleylamine persists in the presence of metal precursor (Figure S3).

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For both thiourea and N,N'-dimethylthiourea, peak amine evolution occurs prior to H_2S release. The temperature-dependent synthesis results closely follow the decomposition windows identified by QMS. For conventional thioureas, the lower-temperature regime is dominated by precursor-derived amine evolution, whereas substantial sulfur release occurs only at higher temperature. In the short-time SrS syntheses, this same temperature window corresponds to suppression or disappearance of crystalline SrS, indicating that perturbation of the ligand and coordination environment precedes effective sulfide delivery. This interpretation is consistent with complementary decomposition studies in which precursor-dependent amine and H_2S evolution windows were correlated with SrS nanoparticle formation across multiple thiourea classes. Critically, this amine-evolution regime coincides with suppression or disappearance of crystalline SrS formation in short-time (5 min) syntheses (Figure 2; XRD in Figure S4). Even in cases where crystalline material had already nucleated at lower temperatures, entry into this regime is associated with dissolution of previously formed nanocrystals, consistent with the precursor-dependent growth interruptions observed in the broader thiourea decomposition study.

These observations indicate that precursor-derived amines act as chemically active perturbations within the reaction medium. In strongly coordinating environments such as oleylamine, additional amine species alter ligand equilibria, metal complex stability, and solubility of sulfide intermediates. The temporal mismatch between amine evolution and sulfur release introduces competing processes: dissolution driven by coordination equilibria versus nucleation driven by sulfide supply.

For unsubstituted thiourea, the simultaneous release of ammonia and H_2S further exacerbates this competition, rendering the effective sulfur activity highly sensitive to external parameters such as nitrogen flow. The result is discontinuous growth behaviour and substantial batch-to-batch variability. These findings identify amine evolution as a hidden but decisive variable in thiourea-based nanocrystal synthesis.

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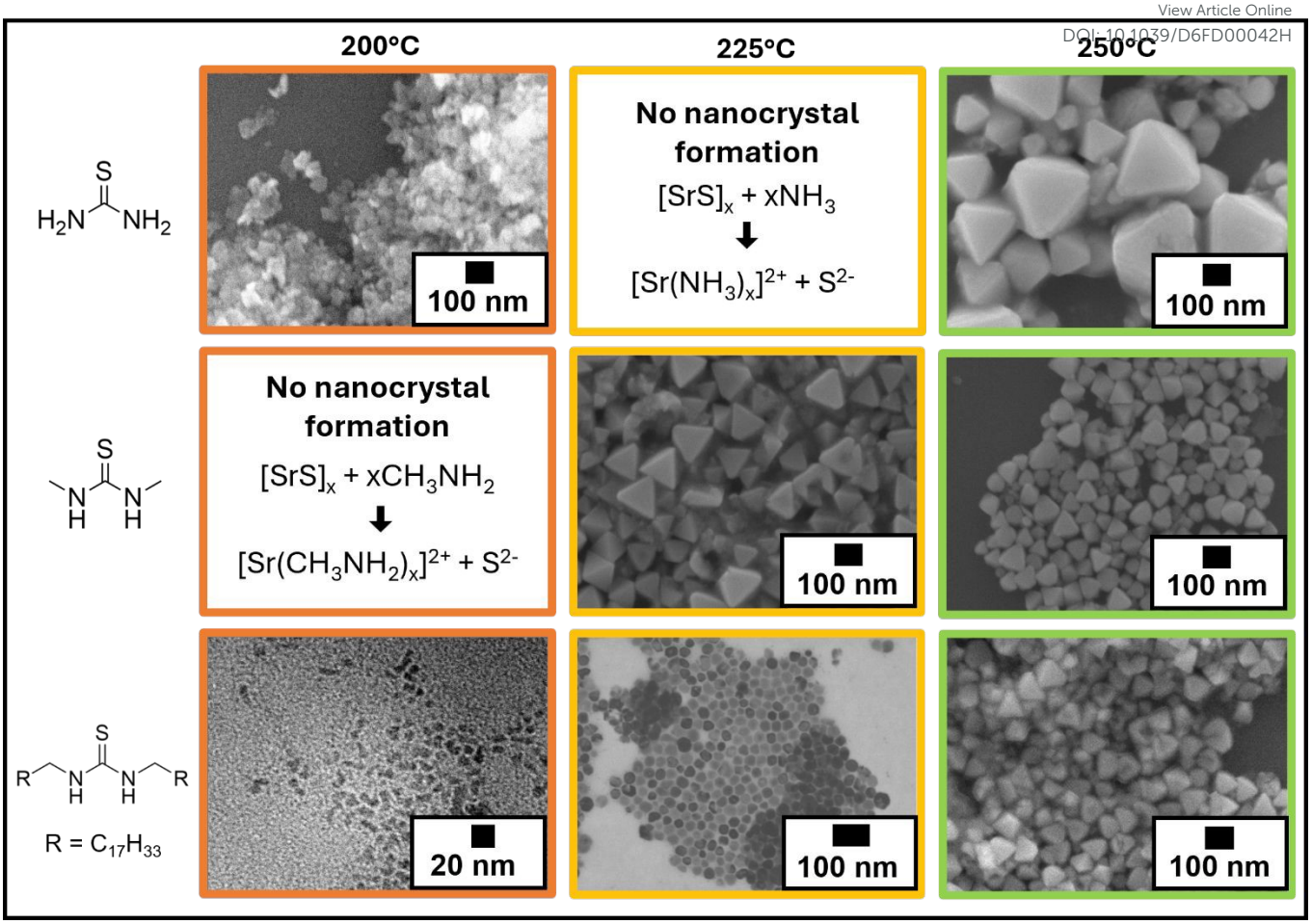

Figure 2. Electron microscopy analysis of SrS nanocrystals synthesized at 200, 225, and 250 °C using thiourea (top row), N,N'-dimethylthiourea (middle row), and N,N'-dioleylthiourea (DOlTU, bottom row) as sulfur precursors. For thiourea and N,N'-dimethylthiourea, nanocrystal formation is suppressed or reversed at temperatures corresponding to peak amine evolution, resulting in discontinuous growth behaviour and absence or dissolution of crystalline material. In contrast, DOlTU enables a continuous, temperature-dependent growth profile: nanoparticles of 6–9 nm at 200 °C evolve into 20–30 nm particles at 225 °C and well-defined octahedral crystals of 50–90 nm at 250 °C. The steady increase in particle size and morphological definition with DOlTU is consistent with decoupling of early amine evolution from sulfur delivery, thereby avoiding the disruptive coordination-environment perturbation observed for conventional thioureas.

Mechanistic Assignment of DOlTU as the Governing Intermediate. The mechanistic convergence of the investigated thioureas to DOlTU suggests that sulfur delivery in these systems is fundamentally mediated by this intermediate rather than by the original precursor directly. In the present interpretation, the later sulfur-release regime is controlled by DOlTU decomposition, whereas the earlier amine-release events represent precursor-dependent perturbations rather than intrinsic requirements for sulfide formation. This view is reinforced by complementary mechanistic studies of thiourea decomposition in oleylamine, which support convergent formation of oleyl-substituted thiourea intermediates and subsequent DOlTU-governed sulfur-release chemistry. This insight reframes the synthetic problem: instead of modifying external parameters to compensate for amine evolution, one may directly employ DOlTU to eliminate the initial decomposition step. [13]

To verify its independent behaviour, we monitored DOlTU decomposition in oleylamine across a broad m/z range. No early-stage amine evolution was detected (Figure S7), confirming that DOlTU bypasses the disruptive initial decomposition regime characteristic of conventional thioureas. We therefore synthesized DOlTU directly (confirmed by NMR, Figure S6) and evaluated its performance in SrS nanocrystal synthesis under identical reaction conditions.

In contrast to thiourea and N,N'-dimethylthiourea, DOlTU produces a continuous and temperature-dependent growth profile (Figure 2). At 200 °C, nanoparticles with diameters of 6–9 nm are obtained. Increasing the temperature to 225 °C yields particles of 20–30 nm, while at 250 °C well-defined octahedral crystals with sizes of 50–90 nm are formed. No dissolution events or discontinuities are observed across this temperature range.

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By eliminating early-stage amine evolution, DOlTU decouples sulfur supply from competing coordination equilibria. Sulfide formation proceeds without disruptive ligand perturbation, enabling steady growth and improved reproducibility. Importantly, this behaviour is not attributable merely to sulfur oxidation state, which is identical across all precursors, but to the decomposition pathway that governs temporal sulfur availability.

Implications for High-Temperature Sulfide Nanomaterial Synthesis. These results demonstrate that precursor decomposition chemistry—rather than precursor identity in a formal sense—determines reaction outcomes in high-temperature metal sulfide synthesis. Thioureas do not fail because of insufficient sulfur content, but because their decomposition sequence introduces reactive amines prior to sulfur delivery.

By identifying DOlTU as the mechanistic intermediate and directly employing it as a precursor, we establish a generalizable strategy: rational design of sulfur sources that decouple ligand evolution from sulfide release. Such control is essential for achieving predictable nucleation kinetics, steady growth profiles, and reproducible nanomaterial properties, particularly in systems targeting optoelectronic functionality where defect density and morphology are critical.

Conclusions

In this work, we identify a fundamental limitation of thiourea and N,N'-substituted thiourea precursors in high-temperature metal sulfide nanocrystal synthesis. Above 160 °C, these precursors intrinsically release reactive amines prior to H_2S evolution, introducing competing coordination and dissolution equilibria that disrupt nucleation and growth. Using SrS as a model system, we demonstrate that peak amine evolution coincides with suppression or reversal of crystalline material formation, resulting in discontinuous and poorly reproducible growth behaviour. More explicitly, the temperature window identified by QMS as dominated by precursor-derived amine evolution corresponds to the synthesis window in which crystalline SrS formation is interrupted, whereas direct use of DOlTU removes this mismatch and enables continuous growth.

Mechanistic analysis reveals that all investigated thioureas converge to N,N'-dioleylthiourea (DOlTU) as a common intermediate preceding sulfur release. This insight reframes conventional thioureas not as direct sulfur sources, but as indirect generators of DOlTU accompanied by chemically active byproducts. By directly employing DOlTU as a precursor, we decouple amine evolution from sulfur delivery and establish a continuous, temperature-dependent nanocrystal growth regime up to 250 °C.

These findings demonstrate that precursor decomposition pathways—rather than formal sulfur oxidation state or solubility alone—govern reaction outcomes in high-temperature colloidal sulfide synthesis. The temporal alignment between ligand evolution and sulfur release emerges as a decisive design parameter for achieving predictable nucleation and steady growth. More broadly, this work illustrates how mechanistic interrogation of precursor chemistry enables rational redesign strategies that improve reproducibility and control in nanomaterial synthesis.

As the development of emerging optoelectronic materials increasingly demands precise control over defect density, morphology, and phase purity, such precursor-level engineering becomes essential. Designing sulfur sources that deliver reactive species without introducing disruptive side chemistry represents a general strategy toward robust and scalable synthesis of high-performance metal sulfide nanomaterials. More broadly, this work highlights precursor decomposition pathway engineering as a powerful strategy for controlling nanocrystal nucleation environments, suggesting that rational precursor design may offer a general route toward reproducible synthesis of complex chalcogenide nanomaterials.

Author contributions

V. Mauritz: Conceptualization, Methodology, Formal analysis, Investigation, Writing - Original Draft, Visualization; **R. Crisp**: Funding acquisition, Project administration, Supervision, Writing - Review & Editing, Data Curation, Conceptualization

Conflicts of interest

There are no conflicts to declare.

Data availability

All data for this article are available from Zenodo.org at https://doi.org/10.5281/zenodo.18867880.

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