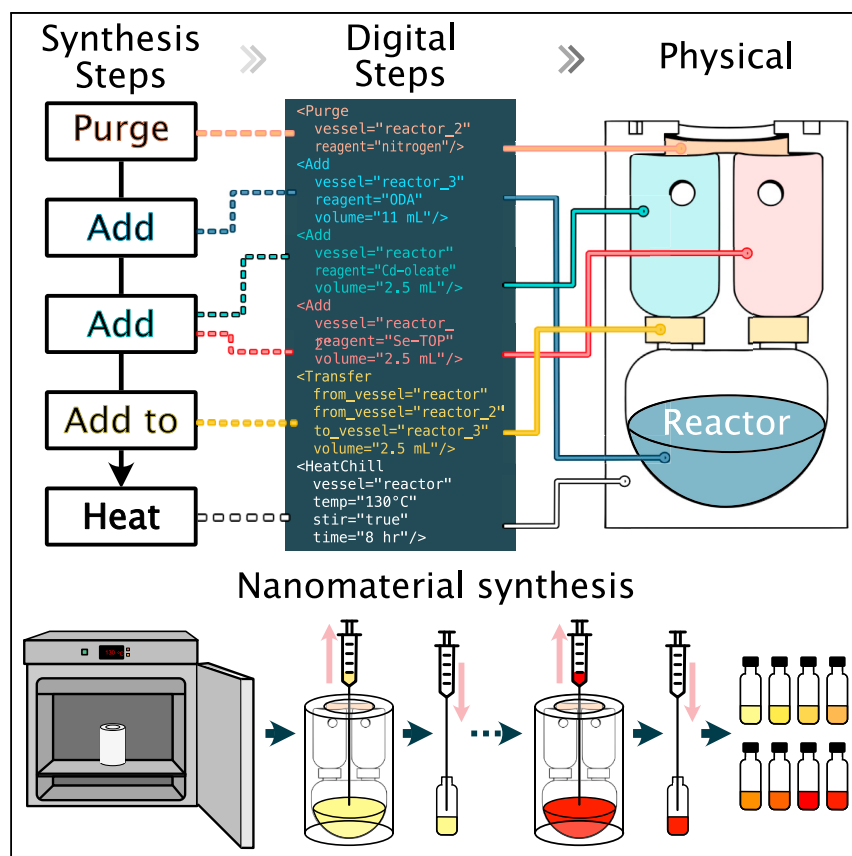


## Article

## Digitizing protocols into single reactors for the one-pot synthesis of nanomaterials



Our method encodes synthetic procedures into a one-pot synthesis reactor based on the chemical description language  $\chi$ DL. We demonstrate the automation of complex processes for the synthesis of eight time-resolved CdSe quantum dots, CdSe@ZnSe core-shell nanoparticles, and Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles through one-time, dropwise, and sequential addition, respectively. This approach enables simple, reliable, and reproducible nanoparticle synthesis, making it accessible to researchers from various fields on demand.

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**Highlights**

One-pot one-time, dropwise, and sequential addition syntheses of nanomaterials

Encodes synthetic procedures into reactor using chemical description language  $\chi$ DL

Synthesis of CdSe QDs, CdSe@ZnSe core-shell NPs, and Pt-Fe<sub>3</sub>O<sub>4</sub> Janus NPs

Simple, reproducible, and accessible reactor for automated synthesis of nanomaterials

**Demonstrate**

Proof-of-concept of performance with intended application/response

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## Article

## Digitizing protocols into single reactors for the one-pot synthesis of nanomaterials

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## SUMMARY

Nanomaterials exhibit unique properties that are tunable depending on their morphology and composition. However, widely used methods can fail due to incomplete or assumed knowledge of the synthesis protocol. A digital synthesis method could unambiguously capture the synthesis process, including required hardware, reagent inputs, and process description, thereby increasing accessibility and improving reproducibility. Our method encodes synthetic procedures into single reactors by imprinting synthetic parameters to the reactor's morphology, which are described using a chemical description language,  $\chi$ DL. This approach is consolidated into three single reactors that automate complex processes such as one-time, drop-wise, and sequential addition for the synthesis of eight time-resolved CdSe quantum dots, CdSe@ZnSe core-shell nanoparticles, and Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles, respectively. This method of translating synthetic parameters into physical instances of a reactor enables the automation of nanomaterial synthesis in a simple, reliable, and reproducible manner, making nanomaterials accessible to researchers from various fields on demand.

## INTRODUCTION

Nanomaterials, commonly synthesized as a colloidal dispersion, have been utilized in a variety of electro-, opto-magneto-, mechano-, thermal, or chemo-devices,<sup>1,2</sup> demonstrating a wide range of composition,<sup>3</sup> morphology,<sup>4</sup> particle size,<sup>5</sup> and complexity.<sup>6</sup> Despite the broad availability of these synthetic protocols, well-known methods are prone to failure due to missing or assumed details (e.g., pH, temperature, order, the rate of addition, etc.), as these can dramatically affect the final composition and properties of the nanomaterials.<sup>7–9</sup> One approach to address this is digitizing the synthesis by designing and implementing automated workflows and platforms. However, this requires researchers to acquire new skills, which is time intensive and expensive and requires extensive training. Digitizing chemical procedures, on the other hand, allows us to identify ambiguous or missing parameters and replace them with well-defined variables (e.g., room temperature, stirring rapidly, etc.).<sup>10–12</sup>

3D printing has emerged as a promising and accessible technology for prototyping chemical reactors,<sup>13</sup> which have been used to materialize synthetic procedures and operations into reactor properties. These reactors have demonstrated the ability to (1) control outcomes based solely on the reactor's morphology,<sup>13</sup> (2) prototype reactors for multi-step batch organic or inorganic reactions,<sup>14–17</sup> and (3) discover new metal-organic frameworks (MOFs).<sup>18,19</sup> In the latter example, the precursors were compartmentalized and mixed at high temperatures upon manually flipping

## PROGRESS AND POTENTIAL

Nanomaterials exhibit unique properties that are tunable based on morphology and composition. However, commonly used synthesis procedures may lack complete or accurate knowledge, leading to a reproducibility crisis. To address this, we have developed a new approach that encodes synthesis procedures into a single reactor. Our proof-of-concept automation, which does not rely on electronic components, is demonstrated in the one-pot synthesis of quantum dots, core-shell nanoparticles, and Janus nanoparticles. As materials science continues to advance, we anticipate that reactor capabilities can be further expanded by using cost-efficient and environmentally friendly functional materials. This approach offers a simple, reliable, and reproducible method for nanoparticle synthesis, which can be easily adapted to be used by researchers in different fields.



the reactor in an oven, leading to the discovery of new reactivity. Our hypothesis is that by implementing digitalized chemical procedures and synthetic parameters of nanomaterial synthesis into the physical geometry of self-contained reactors, a one-pot reactor can be produced, increasing accessibility and reproducibility of these materials.

Herein, we developed programmable one-pot reactors by translating syntheses protocols into physical descriptions of sealed reactors. The one-pot reactors were manufactured by combining polymeric materials with different thermal properties to separate precursor solutions into different compartments to prevent early or unwanted reactions. The combination of polymers that can endure desired temperatures (e.g., polypropylene [PP] and polyether-ether-ketone [PEEK]) in composite reactors can be continually optimized for digitizing nanomaterials synthesis. This can be achieved by using high-temperature materials (i.e., PEEK) for the reactor body and low-temperature materials (i.e., seals made of polycaprolactone [PCL]) to separate the compartments. The precursor solutions can be pre-loaded into the reactor, which is then placed in a pre-heated oven where the seals break at a programmed temperature to initiate the reaction. This programmed compartmentalization has been incorporated into three reactor designs that can automate complex procedures such as one-time, dropwise, and sequential addition reactions. The utility of this new approach was demonstrated through the synthesis of time-resolved CdSe quantum dots, CdSe@ZnSe core-shell nanocrystals, and Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles. This general method of digitizing traditional nanomaterials synthesis procedures into an enclosed reactor design can unlock chemistry across various research fields and provide inexperienced researchers with safe, low-cost, and reproducible access to on-demand nanomaterials.

## RESULTS

Highly luminescent nanocrystals, also known as quantum dots (QDs), have been widely used in various applications, from LED<sup>20</sup> and photovoltaics<sup>21</sup> to biomedical and environmental sensing,<sup>22</sup> due to their unique optical and electronic properties.<sup>23</sup> Traditionally, their syntheses have been challenging, requiring high temperatures (200°C–360°C), toxic reagents, air-sensitive reagents, and complex protocols. To speed up the reactor design iteration process and overcome the limitations of materials properties in the current manufacturing process, we implemented a lower-temperature (i.e., 130°C) synthesis of CdSe QDs.<sup>24</sup> We first identified the basic synthetic steps for CdSe QDs and designed a reactor that meets those requirements. Synthetic procedures can be described in various ways depending on the laboratory setup and/or researchers. To precisely encode the chemical reaction protocols, we recently developed a new approach using a programming language known as the chemical description language ( $\chi$ DL).<sup>25–27</sup> This allows us to capture all the necessary information to run the process automatically on a Chemputer.<sup>28,29</sup> After digitizing the synthesis procedure, we used the  $\chi$ DL file to identify all the relevant parameters and processes before encoding them into the physical properties of the reactor design (Figure 1). Encoding the steps into standardized actions helps identify the minimal physical instances needed for the synthesis while also understanding the reaction process. Furthermore, by digitizing the process, we can accelerate the development of an automated way to produce single cartridges, independent of the synthesis process.

For the lower temperature synthesis of CdSe QDs, the vessel was purged and sealed with a silicone septum to allow the reaction to occur in an inert atmosphere. PP, due

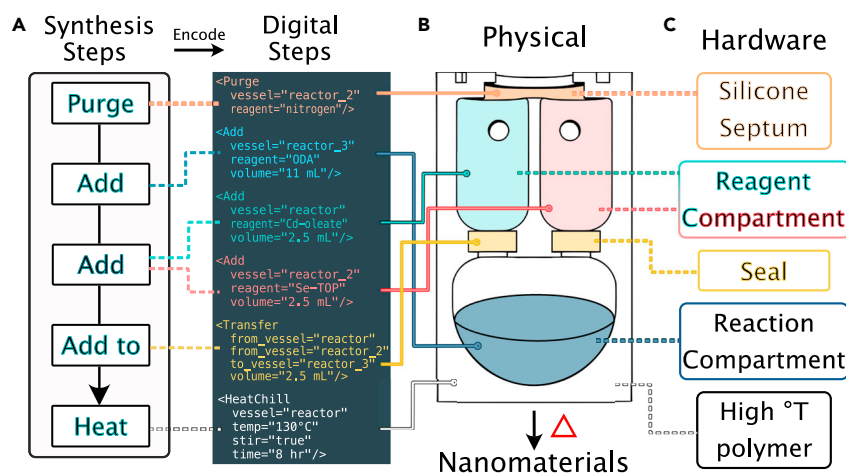
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<https://doi.org/10.1016/j.matt.2023.05.029>



**Figure 1. Digitization process for the synthesis of CdSe QDs**

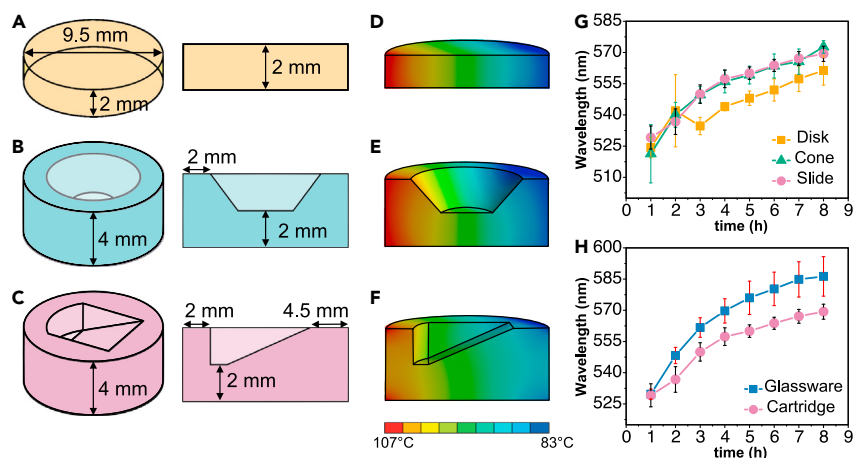
(A) From the traditional synthesis, different synthetic steps can be extracted along with the individual parameters and be converted into a digital file ( $\chi$ DL).

(B) This digital step can then be used to translate the synthesis steps and parameters into physical instances of the respective synthesis and be imprinted into a reactor design.

(C) For the lower temperature of CdSe QDs, the workflow results in a reactionware cartridge with three chambers (two reagent compartments and a reaction compartment) separated by a seal and a silicone septum to work under inert atmosphere conditions.

to its chemical compatibility and thermal stability (up to 130°C), was used as the structural material for the reactor body. During the reaction, three different precursors were mixed at high temperatures; thus, the reactor was designed to contain three chambers (two for reagents and one as the reaction compartment), all separated by a temperature-sensitive seal made of PCL, as shown in Figure 1. The custom-made seal has different thermal properties than PP (m.p. = 63°C, revealed by differential scanning calorimetry [DSC]; see Figure S1), allowing the polymer to soften and eventually fail at high temperatures. Heating the reactionware cartridge to the reaction temperature (130°C) allowed the reagents to drop into the reaction compartment and initiate the reaction.

Initially, a disk-shaped seal (Figure 2A) was designed and incorporated into the reactor body during the printing process to separate the precursor solutions. Varying the width of the seal allowed us to control the time when the solutions were mixed in the reaction compartment. The solutions were transferred into the reaction chamber after 10 (ca. 70°C), 15 (ca. 90°C), and 25 min (ca. 130°C) for the 0.5, 1, and 2 mm seals, respectively. Considering the synthesis starts at 130°C, a 2 mm thickness seal provides the appropriate timing for the reagents to be mixed. To use the programmed reactor, the solutions can be pre-loaded into separate reagent chambers through the septa under inert atmosphere (see supplemental information for preparation procedure). The loaded cartridge was placed in a pre-heated oven at 130°C, and aliquots were taken at 1 h intervals with syringes and long needles. The 2 mm disk-shaped seal resulted in irreproducible photoluminescence (PL) emissions for the first QDs formed during the first 2 h of reaction. Figure 2G (yellow square trace) shows differences as large as  $\pm 17$  nm between batches for the first and second nanocrystals (after 1 and 2 h of reaction, respectively). The differences suggest an uneven breaking of the seal, leading to a continuous addition of precursors into the reaction mixture instead of a one-time addition, generating multiple nucleation stages. To optimize the breaking of the seal, a cone-shaped seal was designed, focusing the pressure point on the middle section of the seal (Figure 2B).



**Figure 2. Effect of seal design on temperature distribution, quality, and reproducibility of CdSe QDs**

(A–C) Dimensions of the different seals: (A) disk, (B) cone, and (C) slide.

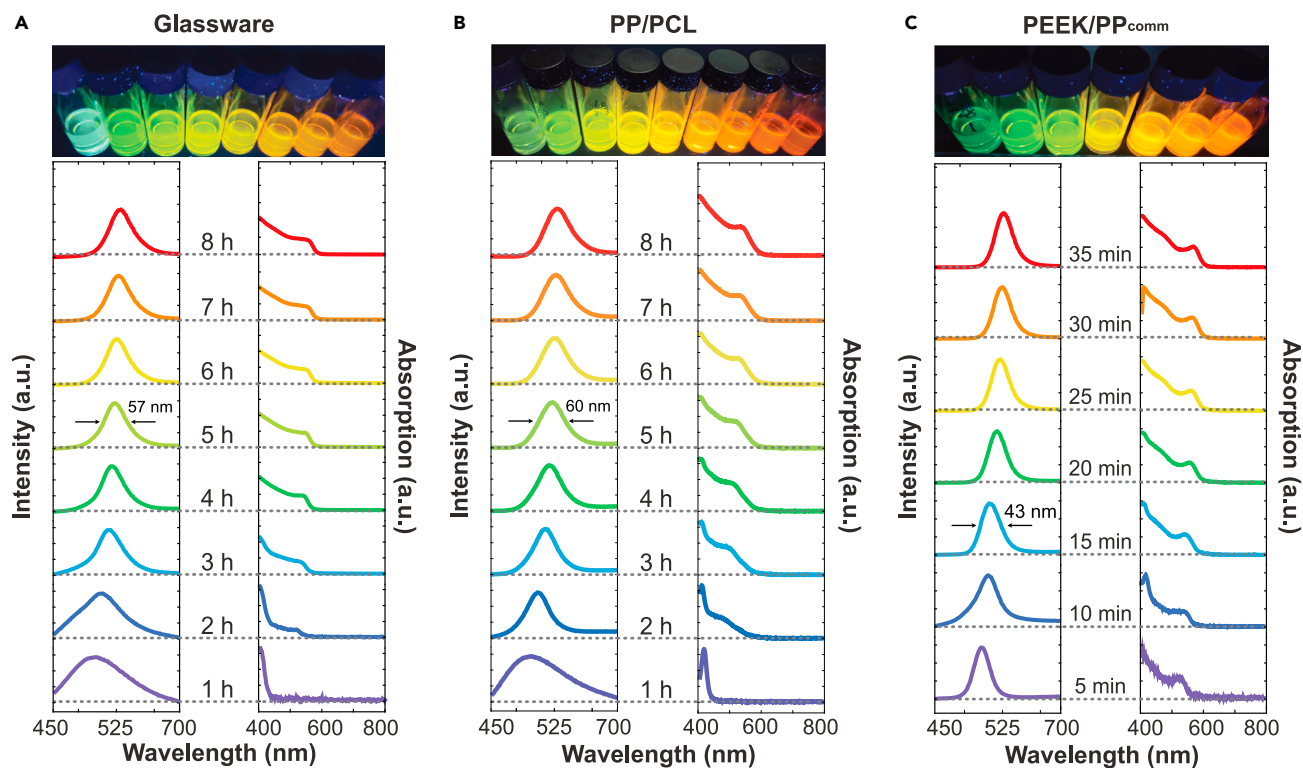
(D–F) Temperature distribution across the (D) disk, (E) cone, and (F) slide seals when the reactor body was at 130°C based on CFD simulations.

(G) Photoluminescence (PL) emission of CdSe QDs using three different seals. Disk (yellow square with yellow error bars), cone (green triangle with green error bars), and slide (pink circle with black error bars).

(H) Comparison of PL for CdSe QDs synthesized using traditional glassware methods (blue squares with red error bars) and using a pre-loaded cartridge (pink circles with black error bars). Error bars correspond to the standard deviation from 9 different experiments.

See also [Figure S7](#).

The cone-shaped design is based on a carved-in cone with a total height of 4 mm and a 2 mm bottom thickness to maintain the breaking time and temperature. This new seal resulted in more reproducible PL emissions for the second sample of QDs ( $\pm 6$  nm compared with  $\pm 17$  nm obtained with the disk-shaped design; [Figure 2G](#)). However, the first nanocrystal PL still showed significant differences between batches ( $\pm 14$  nm), most likely due to an uneven temperature distribution between the cartridge and seal batches. To analyze the temperature distribution across the reactor and seal during the reaction, computational fluid dynamics (CFD) simulations were performed on the system ([Figure S2](#)). A cross-section of the seal region at the time when the reactor body is at 130°C is shown in [Figures 2D](#) and [2E](#). For the disk- and cone-shaped seals, the highest temperature point across the membrane is located toward the outside edge (104°C), while the middle section of the seal has a  $\Delta T$  ca. 19°C. The temperature difference suggests that even if there is a pressure point in the seal design, the temperature difference is determined solely by a slow heat transfer in PP and PCL. Knowing that the highest temperature is focused on the external edge of the seal, a new shape was designed to locate the pressure point toward the outer edge (slide-shaped design; [Figures 2C](#) and [2F](#)). The slide-shaped seal has a total height of 4 mm, with a 2 mm slope of 24°, leading to a bottom thickness of 2 mm. The seals were loaded into the reactionware cartridge, with the breaking point facing toward the wall of the reactor. The new design resulted in PL properties being more reproducible across different reactor batches, minimizing error bars to ca.  $\pm 3$  nm ([Figure 2G](#)). Consistent results were obtained with three batches of triplicates (i.e., 9 replicates) compared with using glassware ([Figure 2H](#)) indicating that with a standard system reducing human input, the QDs are more reproducible.



**Figure 3. PL emission and UV absorption of CdSe QDs**

(A–C) The nanomaterials were prepared using (A) a traditional glassware synthesis method, (B) a PP/PCL reactionware cartridge, and (C) a PEEK/PP<sub>comm</sub> reactionware cartridge. Both cartridges were pre-loaded with the respective precursors.

The time-evolution profile of the CdSe nanocrystal (NC) nucleation and growth process was monitored by UV-visible (UV-vis) and fluorescence spectroscopy for each aliquot (0.5 mL) sampled from the reaction mixtures. To analyze the differences between traditional glassware synthesis and the pre-loaded cartridges, optical absorption and PL emission spectra were obtained from precursor injection to further grow at 130°C (Figures 3A and 3B). When the NCs are at the early stage of formation (<1 h of precursor injection), broadband emission dominates due to the high surface-to-volume ratio. As the NCs grow, the band edge emission becomes the prominent emission, leading to narrower peaks. The absorbance spectra typically show a peak centered at around 412 nm, indicating CdSe magic-size clusters.<sup>30</sup> The transition from clusters to NCs is evidenced by a shoulder appearance in the 450 nm region, along with a drastic change in the PL properties. During the initial growth period, only the cluster broad emission was observed (Figures 3A and 1H), while with the appearance of the absorption shoulder, the PL emission peak sharpens (Figures 3A and 3H). The optical properties of the NC continued to redshift for the remainder of the reaction time, suggesting a continuous growth of the CdSe QDs. For example, after 4 h of precursor injection in glassware, the diameter of the particles is 3 nm, while for the NCs generated with the one-pot system, the diameter is 3.4 nm.<sup>31</sup> The PL emission peak full-width-half-maximum (FWHM) for the NCs prepared in glassware samples was  $57 \pm 3$  nm, while for PP reactionware, the FWHM was  $60 \pm 3$  nm, with quantum yields of 1.4% and 3.6%, respectively.

To expand the synthesis temperature range, a different combination of 3D printing materials was used for the body and seals of the reactor. PEEK is one of the most

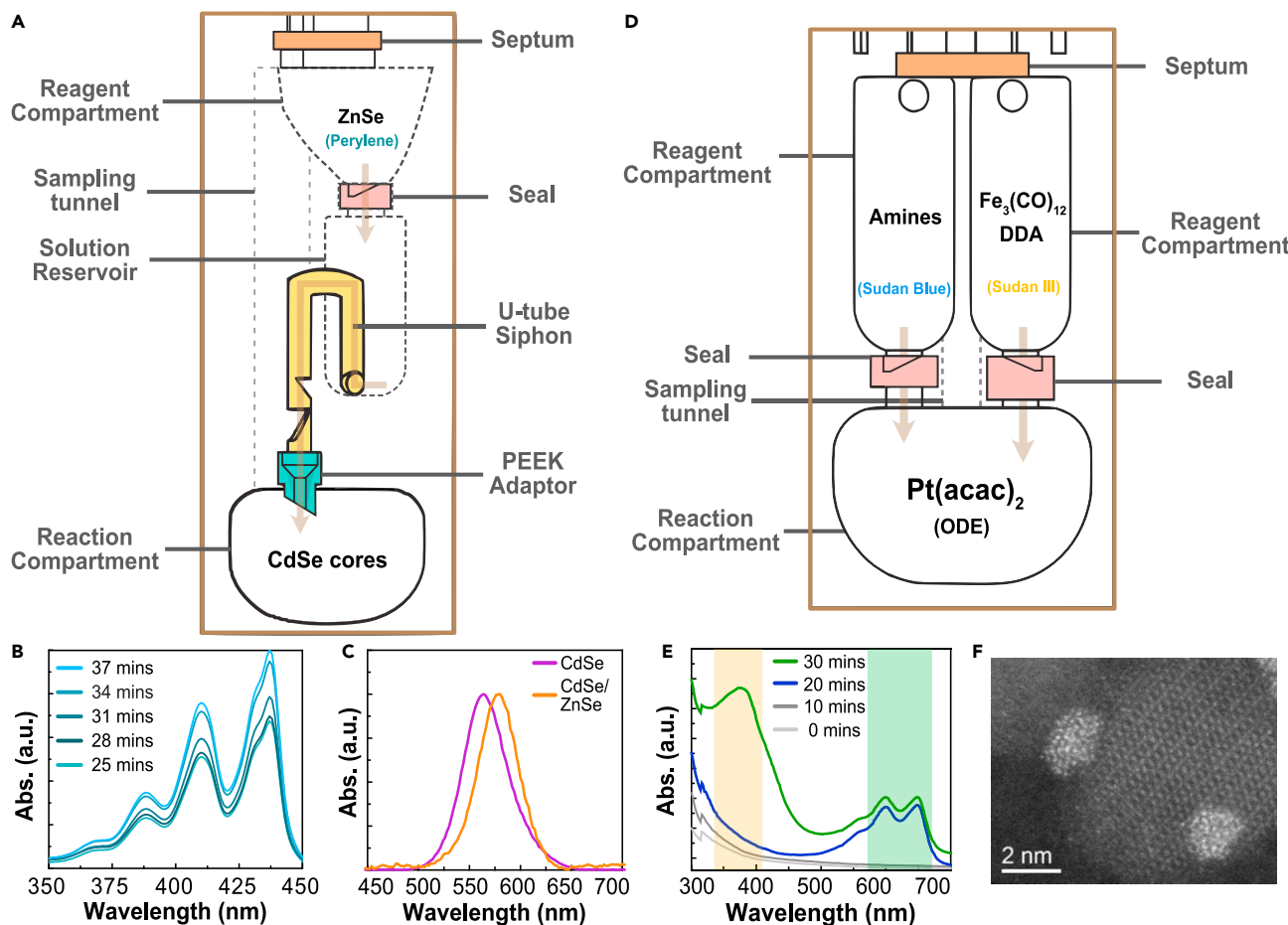
chemically and temperature-resistant polymers available, with a printing temperature of ca. 400°C and an application temperature of up to 250°C.<sup>32</sup> Considering these printing conditions, PCL is not suitable to be used as a seal due to the large difference in thermal properties ( $\Delta T_m = 340^\circ\text{C}$ ); therefore, a different material ID required for the seals. Commercially available PP (PP<sub>comm</sub>), which is sold as a copolymer with PP, has a melting point of 138°C, as revealed by DSC (Figure S1). PP<sub>comm</sub> shows a decent adhesion property and good thermal compatibility with PEEK during the printing process, sealing the reactor and preventing leaking between the different chambers. This new PEEK/PP<sub>comm</sub> (body/seal) reactor combination can perform synthesis at higher temperatures (180°C). As observed in CFD simulations, when the reactor body is at 180°C, the seal temperature is at ca. 140°C (Figure S3). The PEEK/PP<sub>comm</sub> reactor was used for the synthesis of CdSe at 180°C, with the solutions pre-loaded into the cartridge using the same techniques as in the PP/PCL reactor. The first drastic difference between the systems (PP/PCL vs. PEEK/PP<sub>comm</sub>) is the timescale. While the PP/PCL reaction requires ca. 4 h to grow to 3.2 nm, the PEEK/PP cartridge requires as little as 10 min to grow to the same size. For this reason, to monitor the growing process of the NCs, aliquots were taken in 5 min intervals (Figure 3C). At high temperatures, the reaction is more thermally driven, and thus there is no peak at ca. 412 nm, corresponding to the magic-size clusters. Figure 3C shows how the optical properties of the NCs redshifted continuously during the reaction; for example, after 15 min of growth, the PL emission FWHM is  $43 \pm 2$  nm. At an elevated temperature (180°C), the reaction requires 5 min to reach a size of 2.6 nm compared with 4 h at 130°C.

#### Dropwise addition: CdSe@ZnSe core-shell QDs

The CdSe core is usually covered by a semiconductor shell with a high-energy band gap (e.g., ZnS, ZnSe, or CdS) to improve the quantum efficiency. Introducing these shells to the surface of the NCs prevents electron-hole recombination caused by surface dangling bonds.<sup>33</sup> To add the shell to the NC, most reactions rely on a two-step synthesis, where the shells can form through the addition of precursors to a reaction mixture containing the cores. To avoid homogeneous nucleation, the shell precursors are typically added dropwise. Among the different shells used to coat the CdSe cores, ZnSe is highly desirable as it prevents interfacial misfits that can turn into traps for electrons and holes.

To imprint the synthesis of core-shell QDs into the reactor design, the base processes need to be identified (as depicted in Figure 1). First, two different solutions need to be mixed at 190°C, which requires two chambers. A critical step, the dropwise addition, must also be incorporated when adding the shell precursor. To encode this into the blueprint of the reactor, a new design was developed based on the principle of a Soxhlet extractor. In this design, the reagent compartment was connected to a solution reservoir, which was then connected to the reaction compartment via a U-tube siphon (Figures 4A and S5). A PEEK adaptor was added to the end of the siphon tubing to control the rate of addition. Considering that the reaction is carried out at 190°C, PEEK was used due to its thermal resistance, and PP was used as the material for the seal. The final design containing all these features is shown in Figure 4A.

To test the rate of addition, a perylene solution was loaded into the reactionware cartridge and was set up in a pre-heated oven at 190°C. Aliquots were sampled every 3 min, with the first sample collected 25 min after the reaction started, showing how the concentration of perylene increases over time (Figure 4B). The sampling was completed once the absorbance values plateaued, matching the concentration of the diluted stock solution (5 mM). Considering the elapsed time between samples



**Figure 4. Reactionware cartridge designs for dropwise and sequential addition synthesis**

(A) Cartridge design for the synthesis of CdSe@ZnSe nanoparticles using a dropwise addition design. The body of the reactor (brown outline) and the adaptor (green insert) were 3D printed using PEEK, while the seal (pink insert) was 3D printed with PP<sub>comm</sub>. The arrow indicates the flow of the reagent solution. See also Figure S10.

(B) UV-vis trace of a perylene solution, showing a slow addition over ca. 37 min.

(C) PL characterization of CdSe@ZnSe prepared using the pre-programmed cartridge showing a redshift in the fluorescence emission of the core-shell nanoparticles. See also Figure S18.

(D) Cartridge design for the synthesis of Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles, featuring a sequential addition controlled by the thickness of the seal (pink inserts, PP<sub>comm</sub>). The body of the cartridge was 3D printed using PEEK (brown outline). The arrows indicate the flow of reagents. See also Figure S11.

(E) UV-vis traces of Sudan solution dropping at different times in a timed/sequential fashion. See also Figure S14.

(F) HR-STEM image of the Janus Pt-Fe<sub>3</sub>O<sub>4</sub> nanoparticles prepared using the pre-programmed cartridge. See also Figure S22.

and the total volume that was added, the addition rate using a PEEK adaptor of 2 mm is ca. 0.2 mL/min. After determining the rate of addition, the CdSe QD and ZnSe solutions were loaded into the cartridge and then placed in a pre-heated oven at 190°C, and samples were taken every hour to monitor the shell growth. As shown in Figure 4C, the growth of the ZnSe shell on the surface of the CdSe core shell caused a small bathochromic shift of 25 nm in the UV and the PL spectra. The CdSe@ZnSe nanoparticles prepared with the cartridge showed a standard deviation of 1.5 nm (Table S2), showcasing the reproducibility of the system.

### Sequential addition: Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles

Seed-mediated growth is one of the most successful traditional methods used to control the shapes and properties of noble metal NCs due to its reliability and versatility.<sup>34</sup> This process involves the sequential reduction of different metal



precursors to synthesize various nanoparticle morphologies (e.g., Janus, dumbbell, core shell, etc.).<sup>35</sup> Here, we incorporated sequential growth into the reactor design for the synthesis of Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles (JNPs). Platinum NPs have shown excellent properties in energy, chemical, and electronic applications.<sup>36</sup> Controlling the size, shape, and composition of Pt-based NPs is important for achieving optimal catalytic properties.<sup>37</sup> Unlike core-shell NPs, JNPs have two different functional NPs accessible with different properties.<sup>38</sup> The sequential growth of Pt-Fe<sub>3</sub>O<sub>4</sub> JNPs includes first a reduction of the Pt precursor to form the NPs, followed by oxidation of Fe. In a traditional synthesis, a mixture of reagents is added to a round-bottom flask under argon flow and heated to reflux at 290°C. A platinum precursor solution is then injected, and after 5 min, the solution is cooled to 220°C, and Fe(CO)<sub>5</sub> is added. The solution is then refluxed at 220°C for a desired amount of time and then cooled to room temperature.<sup>39</sup> One of the main disadvantages of this procedure is the use of Fe(CO)<sub>5</sub>, which has a low decomposition temperature (ca. 103°C) and can hinder the stoichiometry of the reaction.

To alleviate the issues that arise from using Fe(CO)<sub>5</sub> and high temperatures, the synthesis temperature was lowered to 200°C, and Fe(CO)<sub>5</sub> was replaced with Fe<sub>3</sub>(CO)<sub>12</sub> stabilized with 1-dodecylamine.<sup>40</sup> To implement sequential additions in the reactor, different seal thicknesses were used (Figure 4D), allowing one solution to be confined for longer times than the other. To test this setup, seals with 2 and 4 mm seals (Figure S8) were printed and added to the reactor. Sudan solutions were loaded into the different compartments, and the module was set in a pre-heated oven at 200°C. Figure 4E shows the time evolution of the UV-vis spectra for this test setup. During the first 20 min, no trace from any of the Sudan solutions was observed. At 20 min, Sudan Blue was observed, consistent with the breakage of the thinnest seal in the reactor. The sample continued to show only the Sudan Blue trace for up to 30 min. After 30 min, breakage of the second seal was evident, as traces of Sudan III were observed.

The reactionware cartridge design for the synthesis of Pt-Fe<sub>3</sub>O<sub>4</sub> JNPs, featuring a sequential addition controlled by the thickness of the seal, consists of two reagent compartments of 5 mL to hold the amine and Fe precursors and a 20 mL reaction compartment, where the NP formation takes place. The cartridge is placed in a pre-heated oven at 200°C for 2 h, and then the solution is extracted and purified to obtain Pt-Fe<sub>3</sub>O<sub>4</sub> JNPs. High-resolution transmission electron microscopy (HR-TEM) images show that the average NPs are ca. 7 nm (Figure 4F). The crystalline structure of the JNPs was analyzed using powder X-ray diffraction (PXRD). Peaks at 40°, 46°, 68°, and 81° can be assigned to (111), (200), (220), and (311) planes of cubic phase platinum (Fm3m), respectively, while diffractions at 36°, 43°, 57°, 63°, and 74° correspond to (311), (411), (511), (440), and (642) planes of Fe<sub>3</sub>O<sub>4</sub> (Figure S20).

## DISCUSSION

We have demonstrated how synthetic procedures can be implemented into the geometry of four distinct reactors through the synthesis of ten different NPs. The method involves extracting synthetic steps and parameters and implementing them into a single reactor design for automated one-pot synthesis. This results in reactors with multiple compartments, separated by temperature-sensitive seals that can be activated (melted) at a desired temperature by controlling their material, thickness, and design. For lower-temperature reactions (<130°C), a reactor made of a PP/PCL polymer combination is used for the synthesis of continuously growing CdSe QDs. For higher temperatures (130°C < T < 220°C), a PEEK/PP<sub>comm</sub> polymer

combination is used for the synthesis of core-shell CdSe@ZnSe QDs and Pt-Fe<sub>3</sub>O<sub>4</sub> JNPs. The reactors not only involved a different set of polymeric materials but also complex addition conditions, such as one-time (CdSe), dropwise (CdSe@ZnSe), and sequential addition (Pt-Fe<sub>3</sub>O<sub>4</sub>). The reactors contain all the pre-loaded precursors and all the synthetic conditions needed (i.e., timed addition, inert atmosphere, etc.), making them ready to be used in a pre-heated oven. We have demonstrated a new, efficient, and fully automated approach to synthesize nanomaterials based on the synthesis description generated from  $\chi$ DL. This new approach of enclosing traditional nanomaterials synthesis procedures into a reactor design has the potential to revolutionize chemistry across different research fields by making nanomaterials accessible, low cost, and reproducible for inexperienced researchers. With the advances in materials science, synthesis capabilities can be further extended by reactors manufactured with novel, cost-efficient, and environmentally friendly functional materials.

## EXPERIMENTAL PROCEDURES

### Resource availability

#### Lead contact

Further information and requests for resources should be directed to the lead contact, Leroy Cronin ([lee.cronin@glasgow.ac.uk](mailto:lee.cronin@glasgow.ac.uk)).

#### Materials availability

The study did not generate new unique reagents.

#### Data and code availability

Full details to reproduce this work are available in the [supplemental information](#). This includes full experimental details, as well as the designs for all the one-pot, multi-step synthesis cartridges. No code was used during this study.

## Materials

### CdSe QDs

All chemicals were used as received. Cadmium acetate dihydrate (Cd(CH<sub>3</sub>COO)<sub>2</sub> · 2H<sub>2</sub>O; 98%), oleic acid (CH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>CH = CH(CH<sub>2</sub>)<sub>7</sub>COOH), 1-octadecene (ODE; 90%), selenium powder (Se; 99%), toluene (C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub>; 99.5%), trioctylphosphine (TOP; 90%), and octadecylamine (ODA) were obtained from Sigma-Aldrich.

### CdSe@ZnSe core shell

All chemicals were used as received. Cd(CH<sub>3</sub>COO)<sub>2</sub> · 2H<sub>2</sub>O (98%), CH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>CH = CH(CH<sub>2</sub>)<sub>7</sub>COOH, ODE (90%), Se (99%), C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub> (99.5%), TOP (90%), ODA, TOP oxide (TOPO; 99%), hexadecylamine (HDA; 98%), and zinc stearate (tech. grade) were obtained from Sigma-Aldrich.

### Pt-Fe<sub>3</sub>O<sub>4</sub> JNPs

All chemicals were used as received. Platinum(II)acetylacetonate (Pt(acac)<sub>2</sub>; 99.99%), triirondodecacarbonyl (Fe<sub>3</sub>(CO)<sub>12</sub>; contains 1%–10% methyl alcohol), 1,2-hexadecanediol (90%, tech. grade), oleylamine (70%, tech. grade), octyl ether (99%), 1-dodecylamine (DDA; 98%), Sudan Blue II, and Sudan III were obtained from Sigma-Aldrich.

### CdSe QD experiment in glassware at 130°C

To synthesize CdSe QD NPs in glassware, stock solutions (Cd-oleate, Se-TOP) were prepared under inert atmosphere. The Cd-oleate, Se-TOP solution was swiftly injected into a solution of ODA heated to 130°C, and the mixture was reacted for

8 h, sampling every hour to collect NCs of different sizes and emission properties. To convert this procedure into a one-pot synthesis system, as a standard, we performed the synthesis in an oven instead of the standard hotplate. ODA was added to the Erlenmeyer flask capped with a silicone septum and then placed in a pre-heated oven at 130°C. Once the temperature reached 130°C (after ca. 25 min), the Cd-oleate and Se-TOP were swiftly injected into the reactor. The reaction was carried out for 8 h, and 0.5 mL aliquots were taken at each hour interval.

#### *CdSe QD experiment at 180°C*

To synthesize CdSe QD NPs in glassware, stock solutions (Cd-oleate, Se-TOP) were prepared under inert atmosphere. The Cd-oleate, Se-TOP solution was swiftly injected into a solution of ODA heated to 180°C, and the mixture was reacted for 1 h, sampling every hour to collect NCs of different sizes and emission properties. To have a direct comparison with the reactionware procedure, the synthesis was performed in the oven rather than in a standard hotplate. ODA was added to the Erlenmeyer flask capped with a silicone septum and then placed in a pre-heated oven at 180°C. Once the temperature reached 180°C (after ca. 25 min), the Cd-oleate and Se-TOP were swiftly injected into the reactor. The reaction was carried out for 1 h, and 0.5 mL aliquots were collected every 5 min.

#### *CdSe QD reactionware cartridge test*

Sudan solutions were loaded into the different reagent chambers, while ODE was loaded into the reactor chamber. The cartridge was placed in a pre-heated oven at 130°C and 180°C, and 0.5 mL aliquots were sampled every 1 h or 5 min, respectively.

#### *CdSe@ZnSe experiments*

In a typical synthesis of CdSe@ZnSe, CdSe cores dispersed in heptane were added to a three-neck flask under inert atmosphere. To the QD solution, TOPO and HAD were added, and the mixture was heated to 190°C. Separately, a 2 M solution of zinc stearate in toluene was prepared and mixed with a 0.2 M solution of Se in TOP. This solution was injected (using a syringe pump) into the core-QD solution at 190°C. After the addition was completed, the NCs were annealed at 190°C for an additional 1 h. To prepare the final pre-loaded cartridge, a solution (1 mL) of the CdSe cores in heptane and a mixture of TOPO (2.25 g) and HAD (1.26 g) were added to the reaction compartment, while a 2 M solution of zinc stearate (1.25 mL) and a 2 M solution of Se-TOP (1.25 mL) were added to the reagent compartment.

#### *CdSe@ZnSe reactionware cartridge test*

0.2 mM perylene solution was added to the reaction compartment and 3 mL ODE to the reaction compartment. The pre-loaded cartridge was placed in a pre-heated oven at 190°C, 0.5 mL aliquots were sampled every 5 min, and a UV-vis spectrum was collected.

#### *Pt-Fe<sub>3</sub>O<sub>4</sub> experiments*

The first reagent compartment was loaded with a mixture of 1,2-hexadecanediol, oleylamine, and octyl ether, the second was loaded with a mixture of Fe<sub>3</sub>(CO)<sub>12</sub> and DDA, while the reaction compartment was loaded with Pt(acac)<sub>2</sub> in octyl ether. The cartridge was placed in a pre-heated oven at 200°C for 2 h and cooled to room temperature. The thickness of the shell can be controlled by the reaction time.

#### *Pt-Fe<sub>3</sub>O<sub>4</sub> reactionware cartridge test*

After printing, the cartridge was pre-loaded with three different solutions: Sudan Blue for reagent compartment 1, Sudan III for reagent compartment 2, and ODE

for the reaction compartment. The loaded cartridge was set in a pre-heated oven at 200°C, and samples from the reaction compartment were taken every 5 min after 10 min and analyzed by UV-vis spectroscopy.

### SUPPLEMENTAL INFORMATION

Supplemental information can be found online at <https://doi.org/10.1016/j.matt.2023.05.029>.

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### AUTHOR CONTRIBUTIONS

L.C. invented the concept and devised the project, with help from J.S.M. and P.J.K. J.S.M. developed the initial system design and built the first prototype together with H.W. and C.-G.L. H.W. improved the system and carried out all nanomaterials syntheses. M.M. and I.I.S. helped with the TEM characterization of the nanomaterials. H.W. and J.S.M. wrote the paper with help from P.J.K. and L.C.

### DECLARATION OF INTERESTS

The authors declare no competing interests.

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**Matter, Volume 6**

**Supplemental information**

**Digitizing protocols into single reactors  
for the one-pot synthesis of nanomaterials**

**Hsin Wang, J. Sebastián Manzano, Philip J. Kitson, Margaret Mullin, Chang-Gen Lin, Igor I. Slowing, and Leroy Cronin**

## Supplemental Experimental Procedures

### 1. Supplemental Materials

#### CdSe QD

All chemicals were used as received. Cadmium acetate dihydrate ( $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , 98%), oleic acid ( $\text{CH}_3(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{COOH}$ ), 1-octadecene (ODE, 90%), selenium powder (Se, 99%), toluene ( $\text{C}_6\text{H}_5\text{CH}_3$ , 99.5%), trioctylphosphine (TOP, 90%), octadecylamine (ODA) were obtained from Sigma-Aldrich.

#### CdSe@ZnSe core-shell

All chemicals were used as received. Cadmium acetate dihydrate ( $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , 98%), oleic acid ( $\text{CH}_3(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{COOH}$ ), 1-octadecene (ODE, 90%), selenium powder (Se, 99%), toluene ( $\text{C}_6\text{H}_5\text{CH}_3$ , 99.5%), trioctylphosphine (TOP, 90%), octadecylamine (ODA), trioctylphosphine oxide (TOPO, 99%), hexadecylamine (HDA, 98%), and zinc stearate (tech. grade) were obtained from Sigma-Aldrich.

#### Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles

All chemicals were used as received. Platinum(II)acetylacetonate ( $\text{Pt}(\text{acac})_2$ ) (99.99%), triiron dodecacarbonyl ( $\text{Fe}_3(\text{CO})_{12}$ , contains 1-10% methyl alcohol), 1,2-hexadecanediol (90%, tech. grade), oleylamine (70%, tech. grade), Octyl ether (99%), 1-dodecylamine (DDA, 98%), Sudan Blue II, and Sudan III were obtained from Sigma-Aldrich.

### 2. 3D printing information

#### Reactionware

##### *Polypropylene*

3D printing was done using an Ultimaker 2+ FDM printer. Polypropylene (PP) filament was purchased from Barnes Plastic Welding Equipment Ltd., Blackburn, UK. All prints were performed on a 12 mm PP sheet as a replacement of the glass bed provided by Ultimaker. This allowed printing without heating the printer bed and avoiding warping. The printing process was carried out at 260°C, with a speed of 15 mm/s.

##### *PEEK*

3D printing was done using an Intamsys FUNMAT HT printer. Polyether ether ketone (PEEK) filament was purchased from VisionMiner ThermaX PEEK 3DXTech. All prints were performed on a 12 mm PEEK sheet as a replacement of the glass bed provided by Intamsys. This allowed printing without heating the printer bed and avoiding warping. The printing process was carried out at 403°C, with a speed of 50 mm/s.

#### Seals

##### *PCL*

3D printing was done using an Ultimaker 3 FDM printer. Polycaprolactone (PCL) filament was purchased from Ultimaker. All prints were performed on the glass bed provided by Ultimaker at room temperature. The printing process was carried out at 140°C, with a speed of 15 mm/s.

##### *Polypropylene*

3D printing was done using an Ultimaker 3 FDM printer. Polypropylene (PP) filament was purchased from Barnes Plastic Welding Equipment Ltd., Blackburn, UK. All prints were performed on the glass bed

provided by Ultimaker at room temperature. The printing process was carried out at 260°C, with a speed of 15 mm/s.

### 3. CdSe QD synthesis

#### *Stock solutions*

#### **Cadmium oleate**

Cadmium acetate (107 mg, 0.4 mmol) was mixed with oleic acid (1.2 mL, 3.8 mmol) in 11 mL ODE. The mixture was stirred under a nitrogen atmosphere at 130°C for 1h to form cadmium oleate.

#### **Se-TOP**

Se (158 mg, 4.0 mmol) was mixed with TOP (8 mL, 18 mmol) and toluene (0.9 mL) in a round bottom flask. The flask was stirred at room temperature under nitrogen for 1 h.

#### **Octadecylamine (ODA)**

ODA (220 mg, 0.6 mmol) was dissolved in 5 mL ODE, and the mixture was degassed with nitrogen at 90°C for 1h.

#### *Glassware synthesis at 130 °C*

The flask containing the ODA solution was purged with nitrogen for 30 min and then placed in the preheated oven (130°C) for 25 min to equilibrate the temperature. Cd-oleate (2.5 mL) and Se-TOP Cd-oleate (2.5 mL) were swiftly injected to start the reaction. After the first hour of reaction, 0.5 mL aliquots were taken every hour for 8 hrs. To purify the samples, 0.5 mL of hexane and 1 mL of methanol were added, and the coloured hexane layer was extracted into a centrifuge tube and then filled with acetone and centrifuged at 14,000 rpm for 3 min. The solids were redispersed in 1.5 mL of toluene for storage.

#### *Reactionware synthesis (PP)*

The 3D-printed cartridge was purged with nitrogen for 30 min before the addition of reagents. Cd-oleate (2.5 mL) was cooled to room temperature before injecting into one of the reagent compartments, while Se-TOP (2.5 mL) was injected into the remaining *reagent compartment*. Finally, the ODA solution (5 mL) was injected into the *reaction compartment* while warm to prevent the solidification of the material. The prepared cartridge was placed into a preheated oven at 130°C. 0.5 mL aliquots were taken every hour for 8 hrs. For each sample, 0.5 mL of hexane and 1 mL of methanol were added, and the coloured hexane layer was extracted into a centrifuge tube which then was filled with acetone and centrifuged at 14,000 rpm for 3 min. The solids were redispersed in 1.5 mL of toluene for storage.

#### *Glassware synthesis at 180 °C*

The flask containing the ODA solution was purged with nitrogen for 30 min and then placed in the preheated oven (180°C) for 25 min to equilibrate the temperature. Cd-oleate (2.5 mL) and Se-TOP Cd-oleate (2.5 mL) were swiftly injected to start the reaction. After the first 25 min of reaction, 0.5 mL aliquots were taken every 5 min for 1 h. To purify the samples, 0.5 mL of hexane and 1 mL of methanol were added, and the coloured hexane layer was extracted into a centrifuge tube and then filled with acetone and centrifuged at 14,000 rpm for 3 min. The solids were redispersed in 1.5 mL of toluene for storage.

#### *Reactionware synthesis (PEEK)*

The 3D-printed cartridge was purged with nitrogen for 30 min before the addition of reagents. Cd-oleate (2.5 mL) was cooled to room temperature before injecting into one of the reagent compartments, while Se-TOP (2.5 mL) was injected into the remaining *reagent compartment*. Finally, the ODA solution (5 mL) was injected into the *reaction compartment* while warm to prevent the solidification of the material. The prepared cartridge was placed into a preheated oven at 180°C. 0.5 mL aliquots were taken every 5 min for 1 hr. For each sample, 0.5 mL of hexane and 1 mL of methanol were added, and the coloured hexane layer was extracted into a centrifuge tube which then was filled with acetone and centrifuged at 14,000 rpm for 3 min. The solids were redispersed in 1.5 mL of toluene for storage.



#### 4. CdSe@ZnSe Core-shell synthesis

##### *Stock solution-CdSe Core*

**Cadmium oleate:** Cadmium acetate (107 mg, 0.4 mmol) was mixed with oleic acid (1.2 mL, 3.8 mmol) in 11 mL ODE. The mixture was stirred under nitrogen atmosphere at 130°C for 1h to form cadmium oleate. **Se-TOP:** Se (158 mg, 4.0 mmol) was mixed with TOP (8 mL, 18 mmol) and toluene (0.9 mL) in a round bottom flask under nitrogen atmosphere. The flask was stirred at room temperature under nitrogen for 1 h. **ODA:** Octadecylamine (ODA, 220 mg, 0.6 mmol) was dissolved in 5 mL ODE, and the mixture was degassed with nitrogen at 90°C for 1h. **CdSe Core:** The flask with ODA solution was placed in the pre-heated oven (180°C) for 8 min to equilibrate the temperature. Cd-oleate (2.5 mL) and Se-TOP Cd-oleate (2.5 mL) were swiftly injected to start the reaction. After 8 min the flask was quenched with an ice bath. For the sample, 5 mL of hexane and 10 mL of methanol were added, the coloured hexane layer was extracted into a centrifuge tube which then was filled with acetone and centrifuged at 14,000 rpm for 15 min. The solids were redispersed in 2 mL of heptane. **CdSe core solution:** Trioctylphosphine (TOPO, 2.25 g, 5.8 mmol) and hexadecylamine (HAD, 1.26 g, 5.3 mmol) were mixed with CdSe in heptane (2 mL synthesis below) at 190°C for 15 min under the flow of nitrogen.

##### *Stock solution-ZnSe Shell*

**Zinc:** Zinc stearate (158 mg, 0.25 mmol) was mixed with ODE (1.5 mL). The mixture was stirred under nitrogen atmosphere at 60°C until dissolved and cooled to room temperature. **Se-TOP:** Se (19.7 mg, 0.25 mmol) was mixed with TOP (1.5 mL, 3.4 mmol) under nitrogen atmosphere. The flask was stirred at room temperature under nitrogen for 1 h. **ZnSe shell solution:** Zinc solution was combined with Se-TOP under nitrogen atmosphere.

##### *Glassware synthesis*

ZnSe shell solution was injected into CdSe core solution at the rate of 0.15 mL/min. After the addition was complete the mixture was annealed at 190°C for 1 h. CdSe@ZnSe QDs were washed with methanol several times and dried to collect as powder.

##### *Reactionware synthesis (PEEK/PP<sub>comm</sub>)*

The printed cartridge was purged with nitrogen for 30 min before addition of reagents and solvents. The oven was preheated to 190°C. CdSe core solution was injected into the bottom reactor. ZnSe shell solution was injected into the top reagent compartment. The loaded cartridge was placed in the oven for 1.5 h and quenched with an ice bath. Methanol was injected into the reaction compartment to collect the CdSe@ZnSe QD. CdSe@ZnSe QDs were washed with methanol several times and dried to collect as powder.

#### 5. Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticle synthesis

##### *Stock solutions*

**Amine solution:** 1,2-hexadecanediol (0.75 mmol, 193 mg), oleylamine (0.25 mmol, 85 µL), were added to octyl ether (2.5 mL) and heated to 200°C under nitrogen for 15 min. **Pt solution:** Platinum(II)acetylacetonate Pt(acac)<sub>2</sub> (0.25 mmol, 98.5 mg) was dispersed in octyl ether (5 mL) under nitrogen. **Fe solution:** Fe<sub>3</sub>(CO)<sub>12</sub> (0.25 mmol, 0.167 g) was mixed with DDA (7.5 mmol, 1.39 g) 1:30 mol ratio.

##### *Glassware synthesis*

Amine solution (2.5 mL) was heated at 200°C under nitrogen for 15 min. Pt solution (5 mL) was swiftly injected into the amine solution flask, the colour will turn black indicating the spontaneous formation of nanoparticles. After 20 min, the Fe solution was added to the reaction flask, and it was heated for 1 hr. The solution was cooled to room temperature and washed with ethanol (40 mL) twice. The black precipitant was dispersed in hexane (5 mL) in the presence of oleic acid and oleylamine and precipitated out by adding ethanol (20 mL). The final product was dispersed in hexane.

### Reactionware synthesis (PEEK)

Pt(acac)<sub>2</sub> (0.25 mmol, 100 mg) was added to the reaction compartment during printing. The printed cartridge was purged with nitrogen for 30 min before the addition of reagents and solvents. Octyl ether (2 mL) was added into the *reaction compartment* containing Pt(acac)<sub>2</sub>. Amine solution (3 mL) was added to one of the *reagent compartments* while warm to prevent solidification. Finally, Fe solution (1.5 mL) was injected into the remaining *reagent compartment* at room temperature. The prepared cartridge was placed in a pre-heated oven at 200°C for 2 h. The cartridge was taken out of the oven and was cool to room temperature. The recovered solution was washed with ethanol three times. The final product was dispersed in hexane with a small amount of oleic acid.

## 6. Cartridge tests

### *One time addition*

ODE (3 mL) was added to the *reaction compartment*. Sudan Blue II (2.5 mL, 0.2 mM in ODE) was added to one *reagent compartment*, while Sudan III (2.5 mL, 0.2 mM in ODE) was added to the remaining *reagent compartment*. The prepared cartridge was placed in a preheated oven at 130°C. 0.2 mL aliquot was collected every 10 mins.

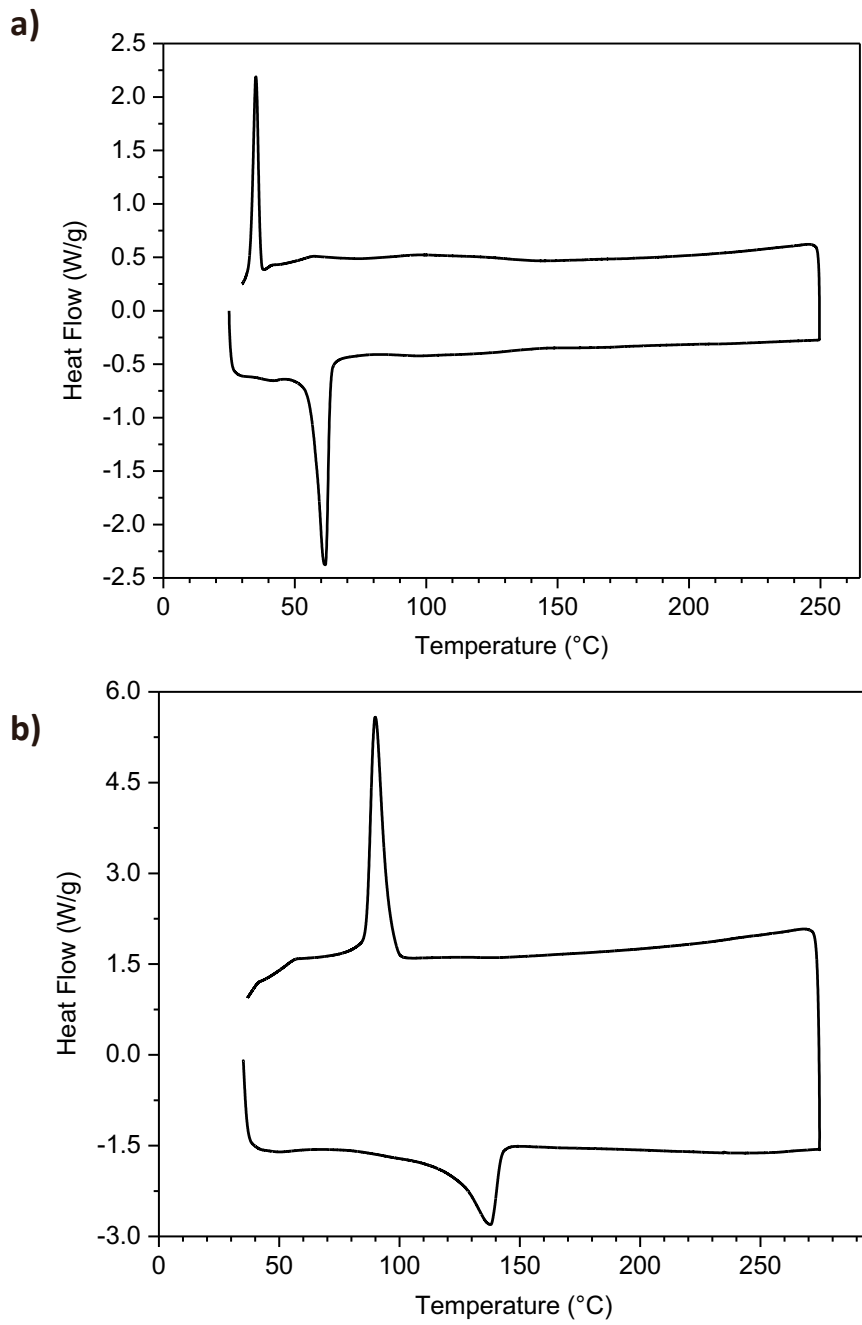
### *Dropwise addition*

To determine the rate of addition of the system shown in **Figure S10** ODE (3 mL) was added to the *reaction compartment* and a perylene solution (2.5 mL, 0.2 mM in ODE) was added to the *reagent compartment*. The prepared cartridge was placed in a pre-heated oven at 190°C. 0.2 mL aliquot was collected every 3 mins. The UV-Vis spectra show a steady increment in the perylene concentration in the *reaction compartment*, with an estimated rate of addition of 0.2 mL/min.

### *Sequential addition*

ODE (3 mL) was added to the *reaction compartment*. Sudan Blue II (2.5 mL, 0.2 mM in ODE) was added to one *reagent compartment*, while Sudan III (2.5 mL, 0.2 mM in ODE) was added to the remaining *reagent compartment*. The prepared cartridge was placed in a preheated oven at 200°C. 0.2 mL aliquot was collected every 10 mins.

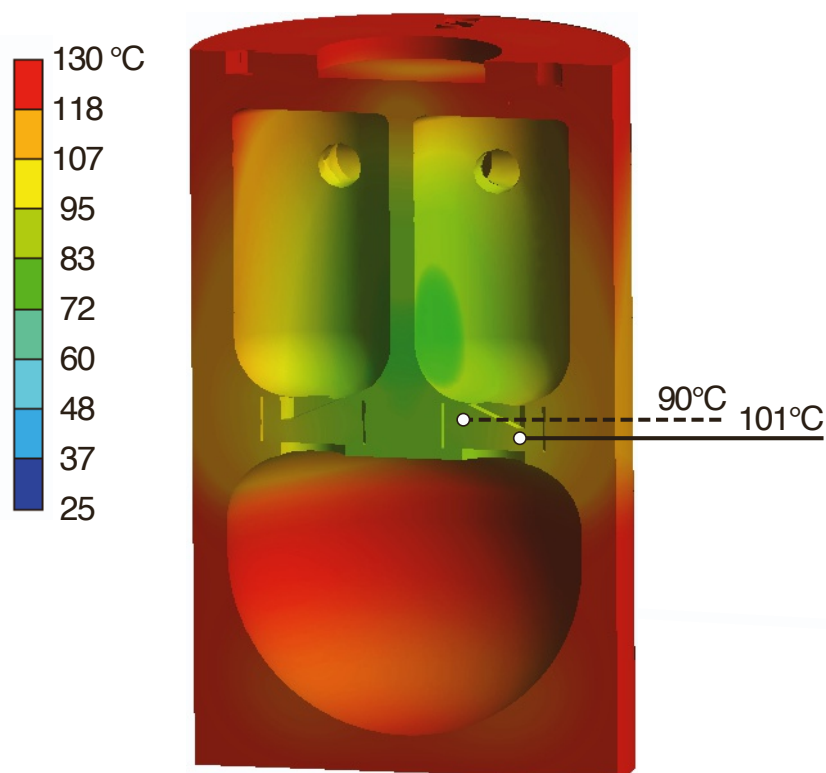
Supplemental Items



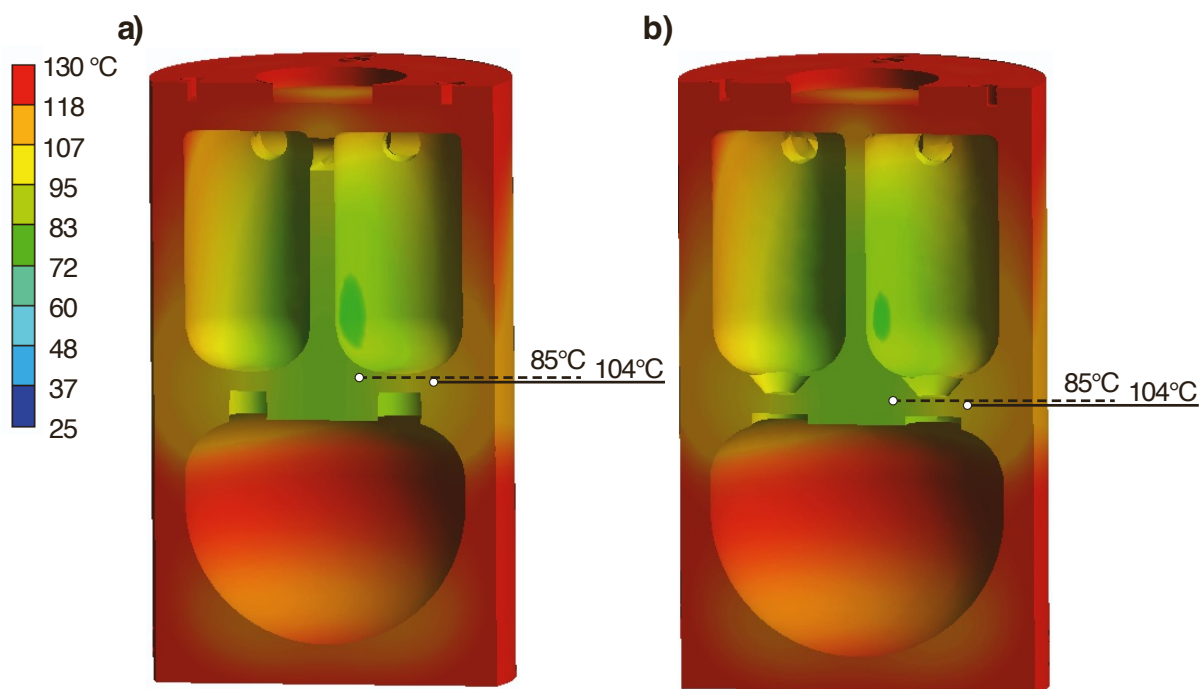
**Figure S1. DSC of a) PCL with a crystallizing temperature of 38°C and melting point of 63°C and b) PP with a crystallizing temperature of 90°C and melting point of 138°C**

## 7. Computational Fluid Dynamic (CFD) simulations

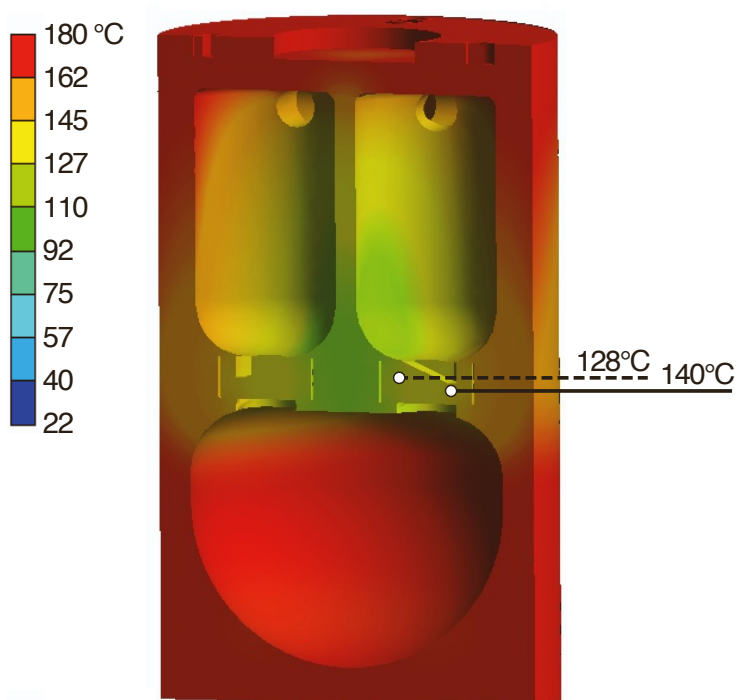
CFD simulations were performed using the Ansys software suite. The models considered all the parts of the designs including the silicone septum, seals, and body of the reactor. Convection heating with a power of 20 was used to surround the cartridge. The outside temperature was set to the respective temperatures (130°C for CdSe, 190°C for Pt-Fe<sub>3</sub>O<sub>4</sub>, and 200°C for CdSe@ZnSe), while the material of the reactor and the seals were specified for the respective polymer. The simulation ran for 18 mins, where the cartridge reached the desired temperature and cross-section analysis was performed on each cartridge to check the temperature distribution across the entire system.



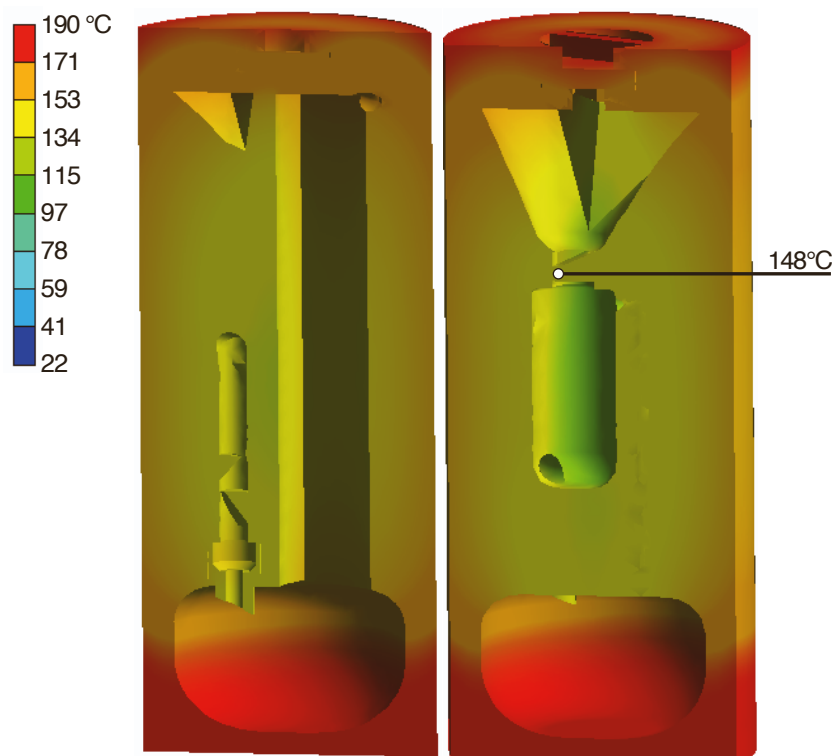
**Figure S2. CFD simulation of One-time addition- CdSe cartridge (PP/PCL).** Cartridge containing slide-seal at 18 min in a 130°C oven. Some parts of the cartridge show 130°C, while the seals are at ca. 30°C lower and have a temperature difference of ca. 11°C across them.



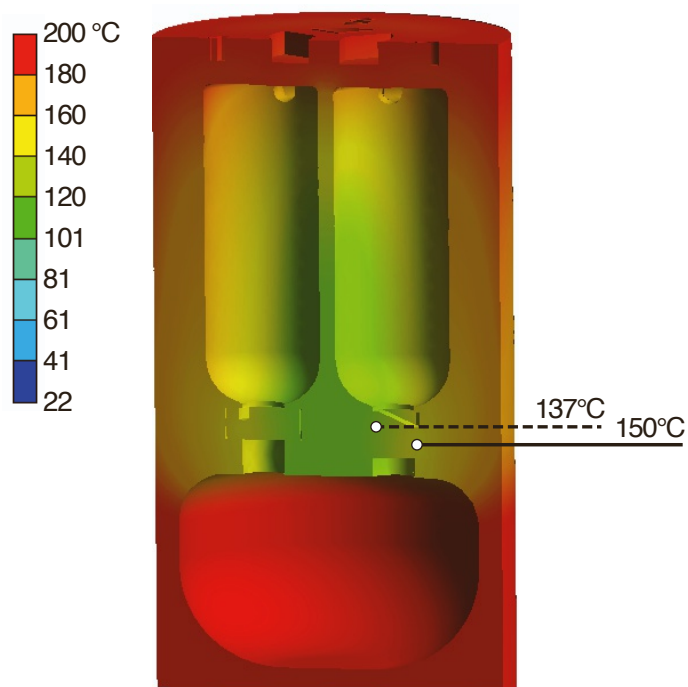
**Figure S3. CFD simulation of One-time addition- CdSe cartridge (PP/PCL).** Cartridge containing a) disk b) cone seal at 18 min in a 130°C oven. The temperature profile indicates that, while the reactor body is at 130°C, the seals are at ca. 40°C lower and have a temperature difference of ca. 15°C across them.



**Figure S4. CFD simulation of One-time addition- CdSe cartridge (PEEK/PP).** Cartridge containing slide-seal at 23 min in a 180°C oven. The temperature profile indicates that, while the reactor body is at 180°C, the seals are at ca. 40°C lower and have a temperature difference of ca. 12°C across them.

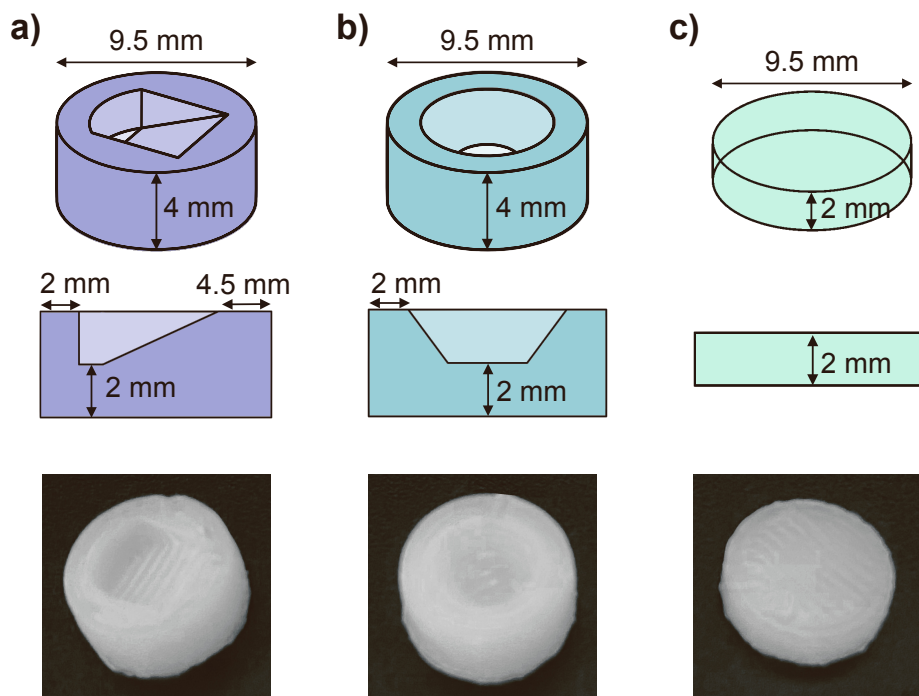


**Figure S5. CFD simulation of Dropwise addition- CdSe@ZnSe cartridge (PEEK/PP).** Cartridge containing slide-seal at 35 min in a 200°C oven. The temperature profile indicates the breaking point in the seal is at 148°C.

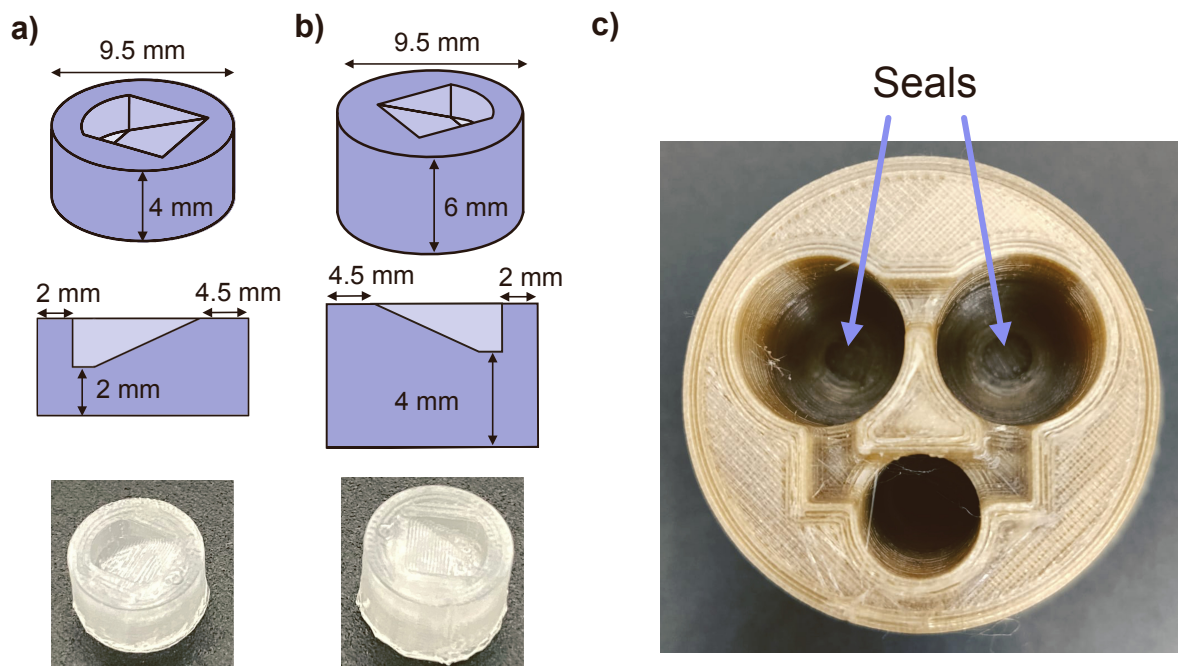


**Figure S6. CFD simulation of Sequential addition- Pt-Fe<sub>3</sub>O<sub>4</sub> cartridge (PEEK/PP<sub>comm</sub>).** Cartridge containing slide-seal at 26 min in a 200°C oven. The temperature profile indicates the breaking point in the seal is at 150°C, while it has a  $\Delta T$  of ca. 13°C across the slide-seal.

## 8. Seal designs



**Figure S7. Seal designs, parameters, and real 3D printed seals.** a) slide with a diameter of 9.5 mm and depth of 2mm b) cone with a diameter of 9.5 mm and depth of 2mm c) disk with diameter of 9.5 mm and thickness of 2mm

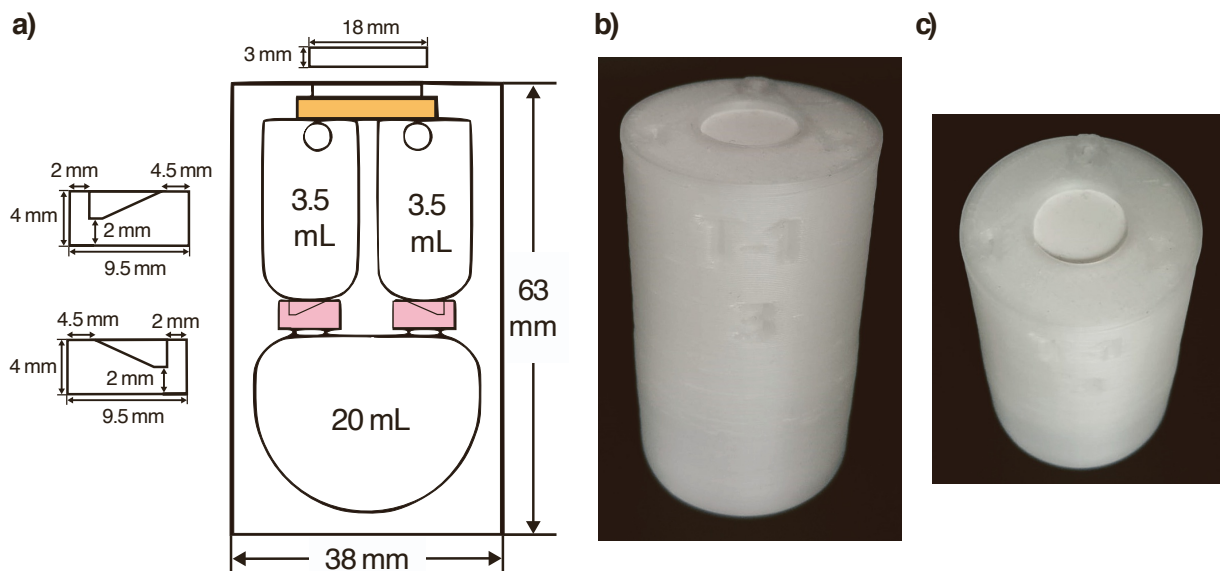


**Figure S8. PP<sub>comm</sub> seal designs and parameters.** a) slide with a diameter of 9.5 mm and thickness of 2mm b) slide with a diameter of 9.5 mm and thickness of 4mm c) position of seals inside a Pt-Fe<sub>3</sub>O<sub>4</sub> cartridge with the pressure point pointing outward

## 9. Cartridge designs

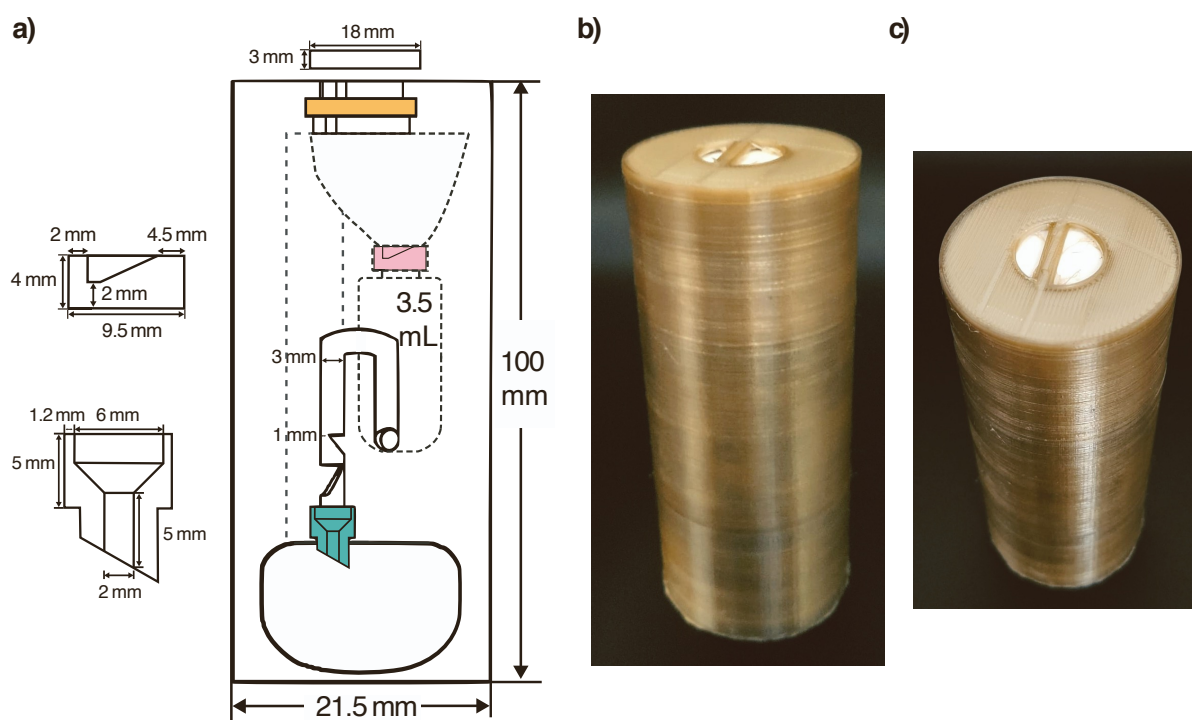
All the cartridges were designed using OnShape™.

*One-time addition- CdSe QD*



**Figure S9. CdSe QD cartridge dimensions.** The cartridge is composed of two 3.5 mL reactor chambers, a 20 mL reactor chamber, two slide-seals of 2 mm of thickness, and a silicone septum with a thickness of 3 mm and a diameter of 18 mm.

*Dropwise addition- CdSe@ZnSe QD*

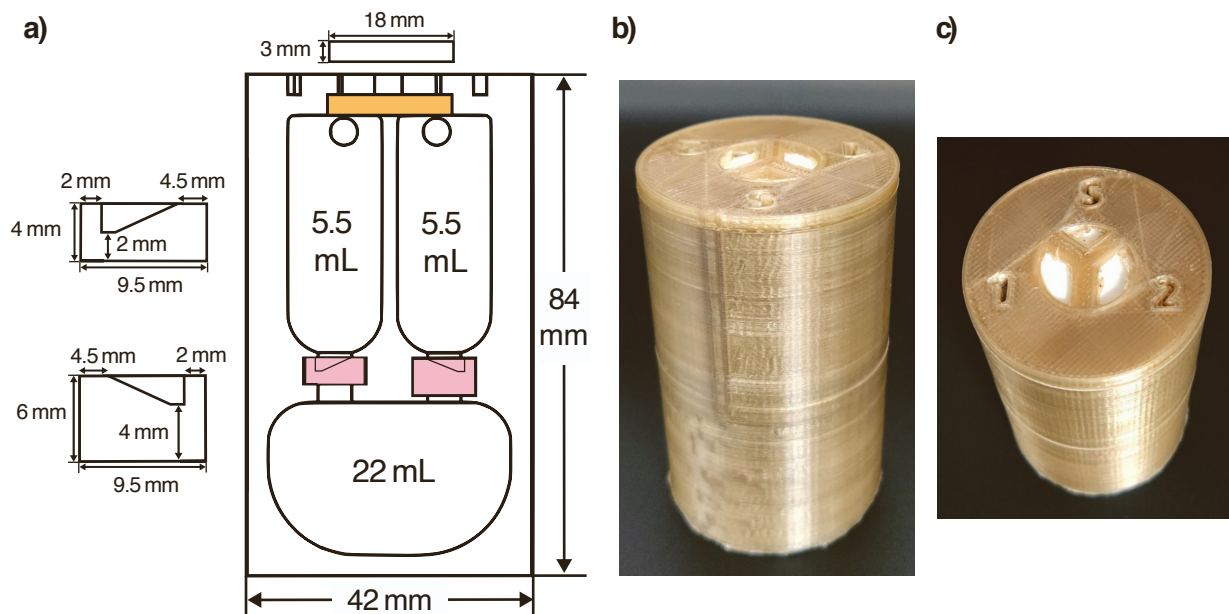


**Figure S10. CdSe@ZnSe core-shell QD synthesis cartridge.** The cartridge is composed of two 3.5 mL reagent chambers, an 18 mL reactor chamber, one slide-seal with 2 mm thickness, one



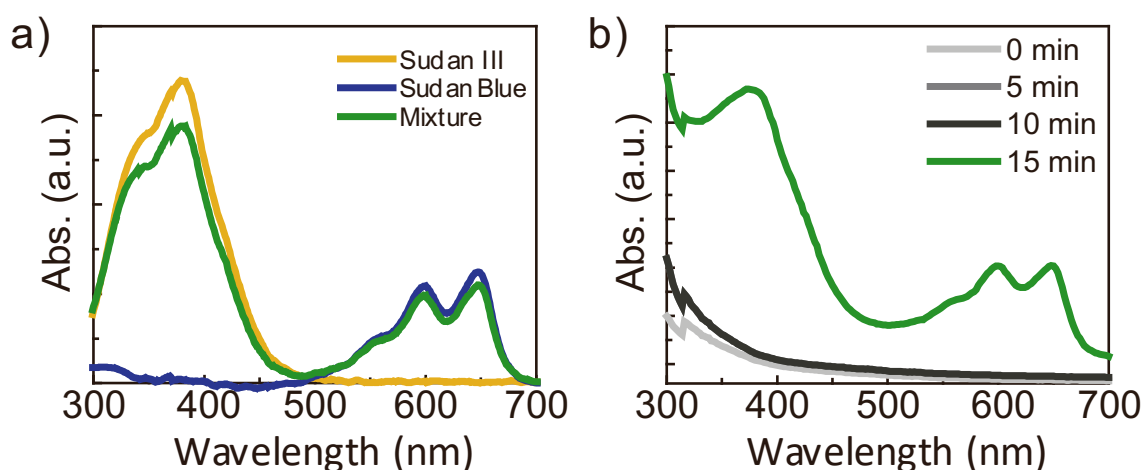
dropwise adaptor with a final opening of 2 mm, and a silicone septum with a thickness of 3 mm and a diameter of 18 mm.

Sequential addition-  $Pt-Fe_3O_4$  Janus nanoparticles

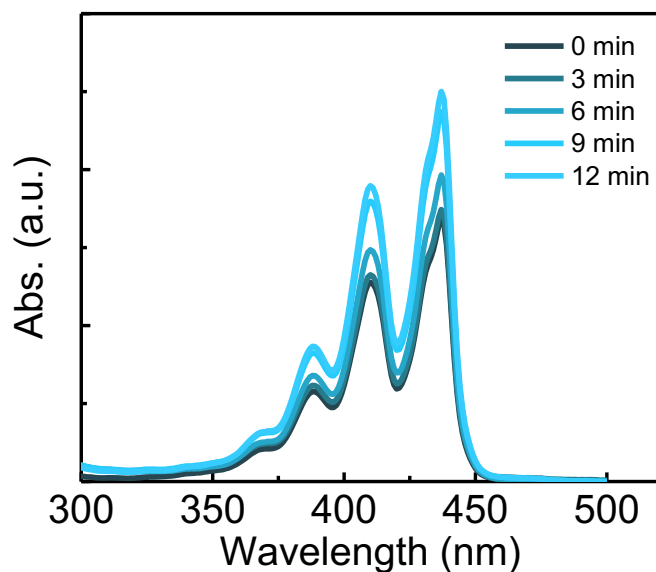


**Figure S11.  $Pt-Fe_3O_4$  Janus nanoparticle synthesis cartridge.** The cartridge is composed of two 5.5 mL reagent chambers, a 22 mL reactor chamber, two slide seals with different thicknesses (2 mm that breaks at minute 25 at 200°C, and a 4 mm that breaks at minute 30 at 200°C), and a silicone septum with 3 mm thickness and 18 mm of diameter. The final cartridge has a diameter of 42 mm and a height of 73 mm.

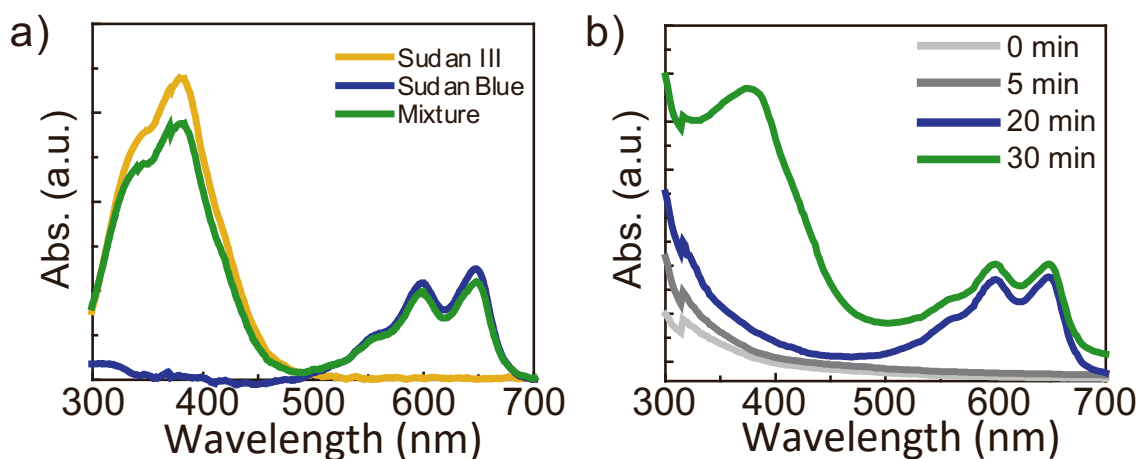
## 10. Cartridge tests



**Figure S12. UV-Vis absorption spectra of One-time addition cartridge test.** a) Sudan III (yellow), Sudan Blue II (blue), and a mixture of Sudan III and Sudan Blue II (green), and b) sampling from reaction compartment intervals showing that both solutions drop simultaneously after 15 mins in a preheated oven at 130°C

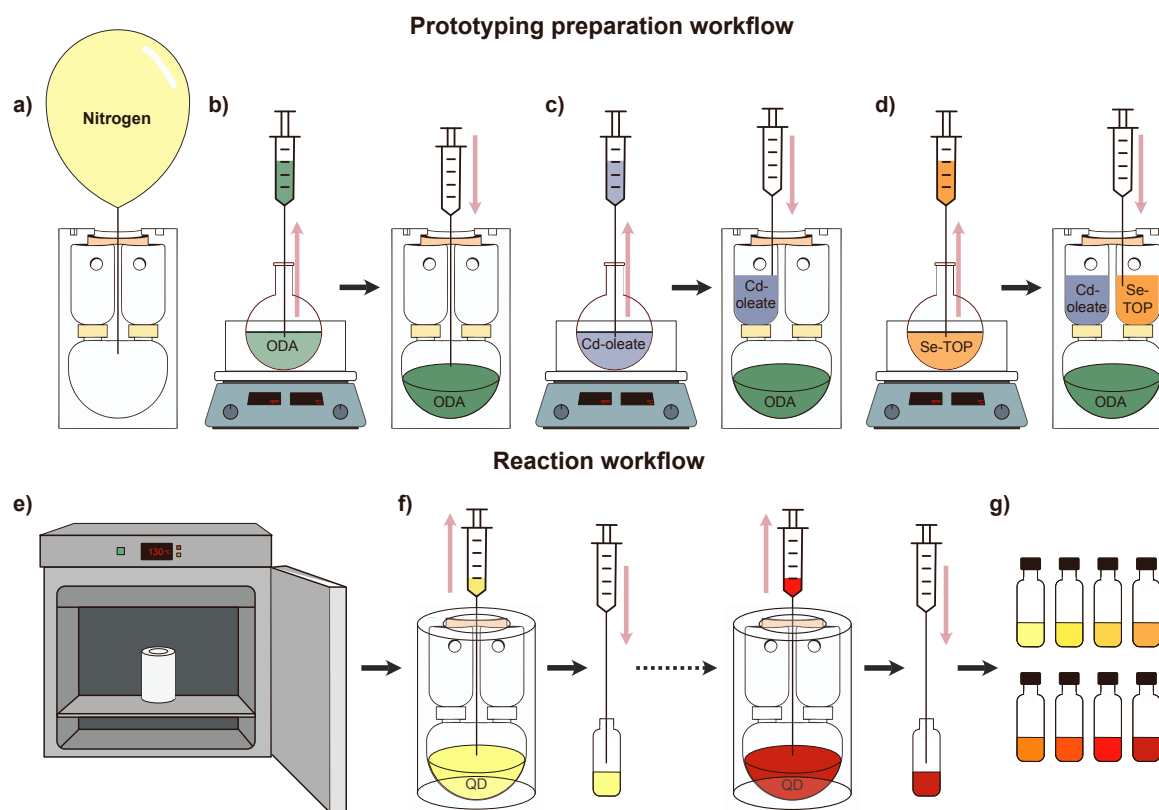


**Figure S13. Absorption spectra of the programmed dropwise addition.** Spectra of perylene to octadecene using the cartridge design shown in Figure S10. UV-Vis spectra were taken every 3 mins after 25 min, to determine the increment in perylene concentration.



**Figure S14. UV-Vis absorption spectra of sequential addition cartridge test.** a) Sudan III (yellow), Sudan Blue II (blue), and mixture of Sudan III and Sudan Blue II (green), and b) the aliquots samples in 10 mins intervals from the reaction compartment showing that at 20 mins only Sudan blue is present, while at 30 mins, Sudan III was added.

## 11. Reaction set-up



**Figure S15. Workflow of cartridge preparation and reaction.** Prototyping preparation workflow- a) the cartridge is purged with nitrogen for 1h before loading reagents b) Octadecylamine was prepared in a round bottom flask and then transferred to the reaction compartment with a long needle and syringe c) Cd-oleate was prepared in a round bottom flask and then transferred to one of the reagent compartments with a long needle and syringe at room temperature d) Se-TOP was prepared in a round bottom flask and then transferred to one of the reagent compartments with a long needle and syringe at room temperature. Reaction workflow e) The prepared cartridge was placed in the preheated oven at the desired temperature. f) Aliquots were taken every while (depending on the reaction) g) 8 samples were collected for purification.

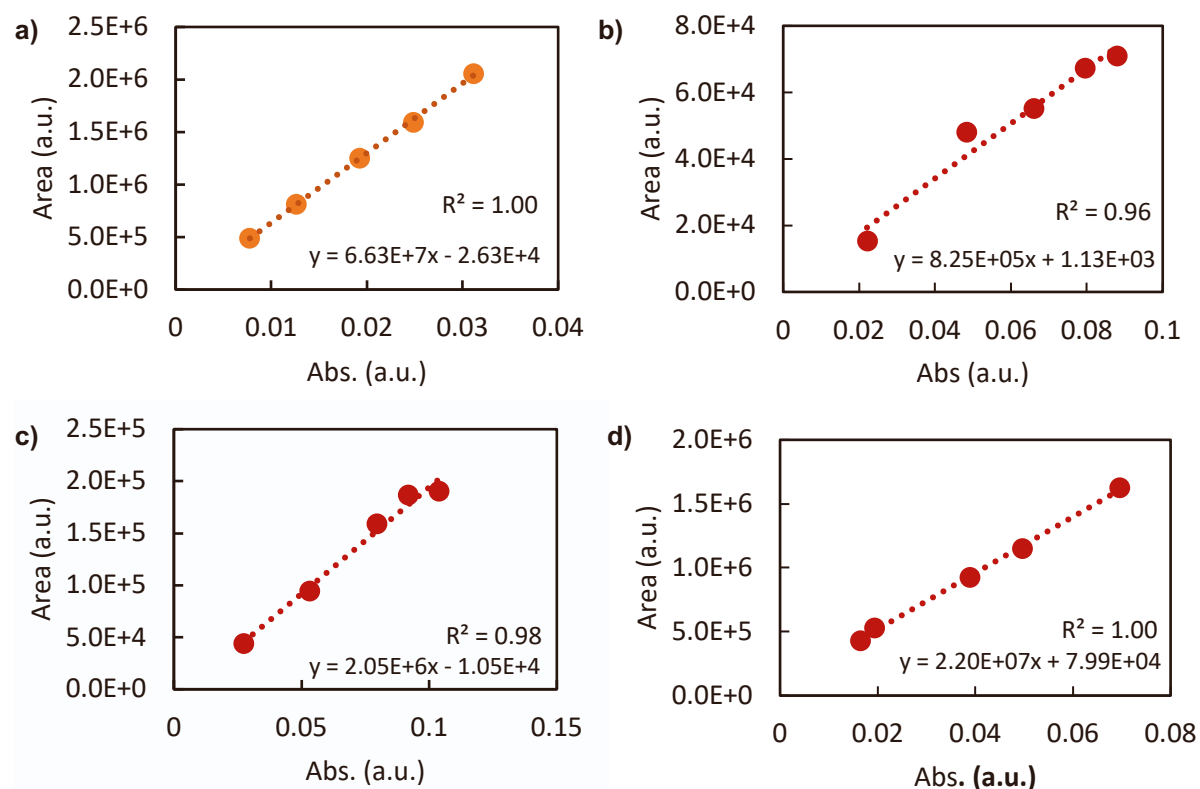
## 12. Characterisations

### CdSe Quantum Yield Calculation

$$\Phi_{QD} = \Phi_{st} \left( \frac{m_{QD}}{m_{st}} \right) \left( \frac{\eta_{QD}^2}{\eta_{st}^2} \right) \quad (1)$$

- $\Phi$  = Quantum yield
- $m$  = Slope of the plot of integrated fluorescence intensity against absorbance
- $\eta$  = Refractive index of the solvent
- Subscript "QD" and "st" refer to the quantum dot prepared and the standard (Rhodamine 6G)
- The reported quantum yield of Rhodamine 6G is 0.95. The Refractive index for quantum dots in toluene is 1.496 and rhodamine in ethanol is 1.36.

- Table S1 shows the calculated quantum yield for the quantum dot in each cartridge and Glassware control.



**Figure S16. Slope fit for the QY calculation (peak area vs. absorbance).** a) Rhodamine 6G reference b) Glassware at 130°C c) PP cartridge at 130 °C d) PEEK cartridge 180°C.

**Table S1. Reported and experimental quantum yield calculated for the CdSe QD nanoparticles prepared using different methods. The values are based on equation 1.**

<b>Sample</b>	<b>Quantum yield (%)</b>
<b>Reported 130 °C</b>	3 %
<b>Glassware at 130 °C</b>	1.4 %
<b>PP at 130 °C</b>	3.6 %
<b>PEEK at 180 °C</b>	25 %

#### *CdSe QD size calculation*

The concentration of cleaned CdSe quantum dot cores is determined through the use of the absorbance. From the optical absorbance curve, we are able to calculate the size of the CdSe cores using the following equation:

$$D(\text{nm}) = (1.6122 \times 10^{-9})\lambda^4 - (2.6575 \times 10^{-6})\lambda^3 + (1.6242 \times 10^{-3})\lambda^2 - (0.4277)\lambda + 41.57 \quad (2)$$

Where  $\lambda$  is the wavelength of the first excitonic absorption peak of the corresponding sample

#### *CdSe QD absorbance and fluorescence*

UV-vis absorption spectra were measured on JASCO V-670 UV-Vis spectrometer. All measurements were performed at room temperature with the plate-reader. 0.2 mL of each solution were placed in every other compartment and row. The QDs were excited at 405 nm.

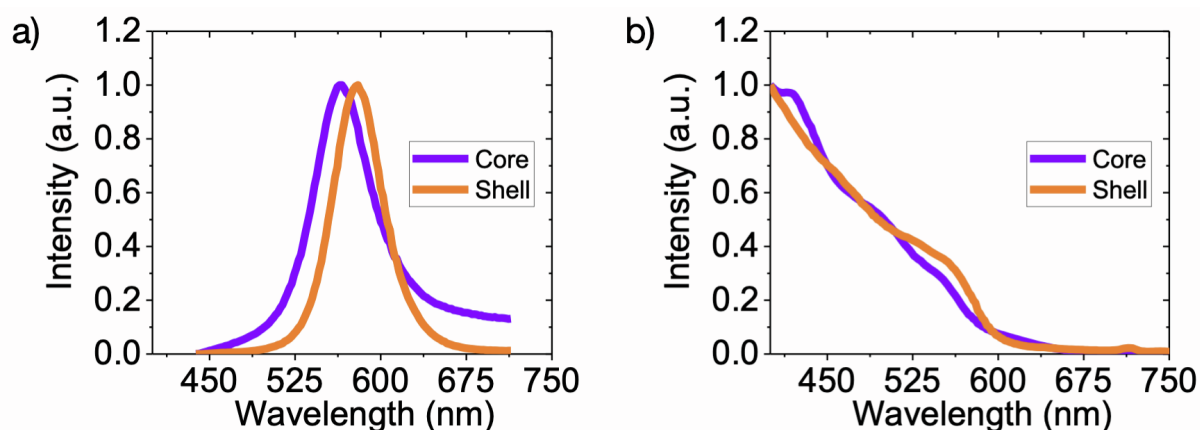


Figure S17. Fluorescence and UV-vis for CdSe@ZnSe core-shell nanoparticles prepared in glassware at 190°C. a) A red shift in the photoluminescence (PL) peak of 14 nm is observed, demonstrating the formation of the shell structure.

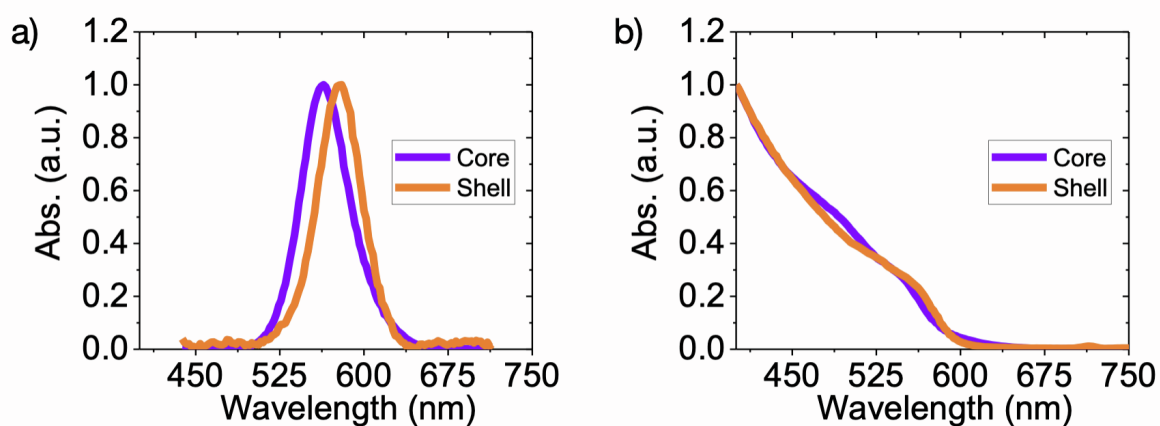
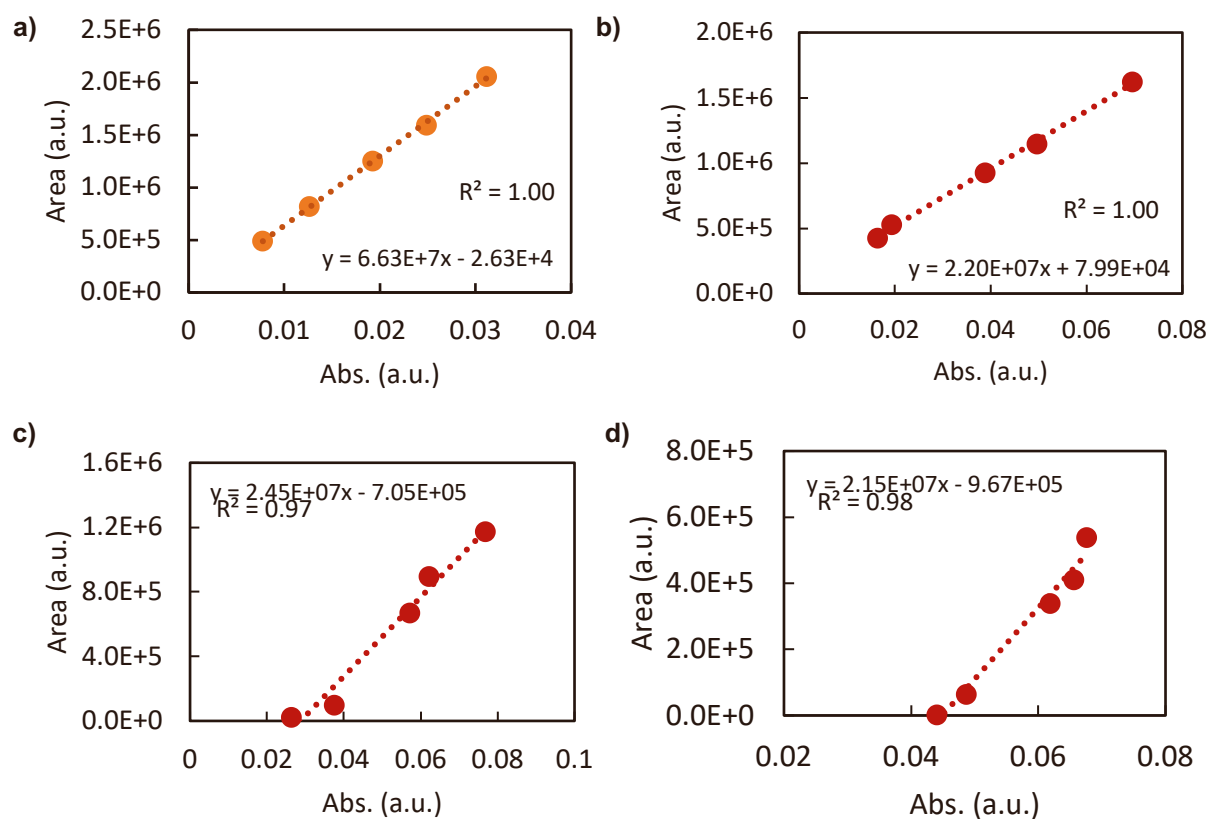


Figure S18. Fluorescence and UV-vis for CdSe@ZnSe core-shell nanoparticles prepared using a preloaded reactor at 190°C. a) A red shift in the photoluminescence (PL) peak of 14 nm is observed, demonstrating the formation of the shell structure.

Table S2. Wavelength comparison of three runs of the CdSe@ZnSe QD nanoparticles prepared using the one-pot reactor.

Number of Runs	CdSe core (nm)	CdSe@ZnSe (nm)
1	556	569
2	556	572
3	556	570



**Figure S19. Slope fit for the QY calculation (peak area vs. absorbance).** a) Rhodamine 6G reference b) Glassware core at 180°C c) PEEK cartridge core shell at 190 °C d) Glassware core shell 190°C.

**Table S3. Experimental quantum yield calculated for the CdSe@ZnSe QD nanoparticles prepared using different methods.** The values are based on equation 1

Sample	Quantum yield (%)
Glassware core at 190 °C	28 %
PEEK CS at 190 °C	36 %
Glassware CS at 190 °C	32 %



Powder X-ray Diffraction (PXRD)

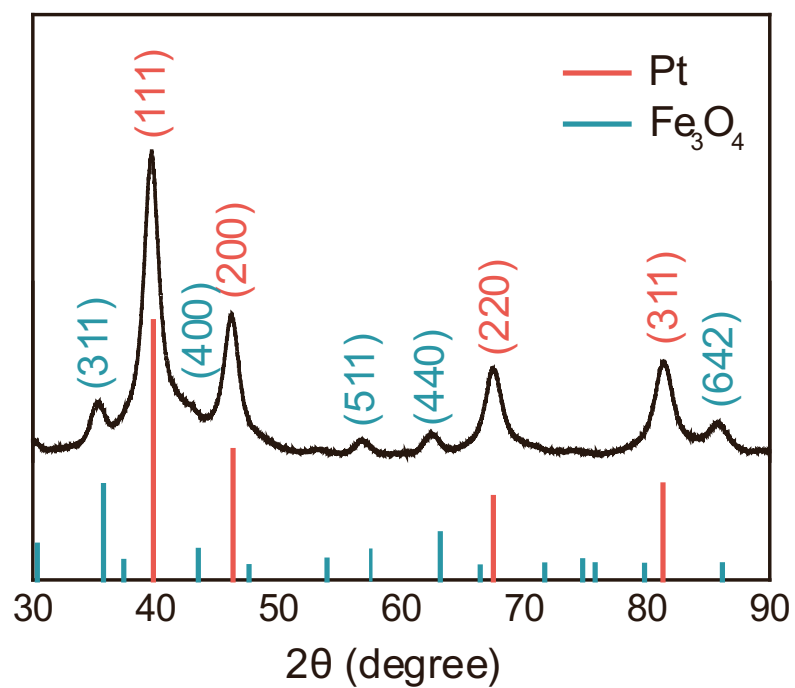


Figure S20. Powder X-ray Diffraction (PXRD) pattern of the Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles. Pt reference (red trace), Fe<sub>3</sub>O<sub>4</sub> reference (teal trace), and the Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles (black trace).

Transmission Electron Microscopy (TEM)

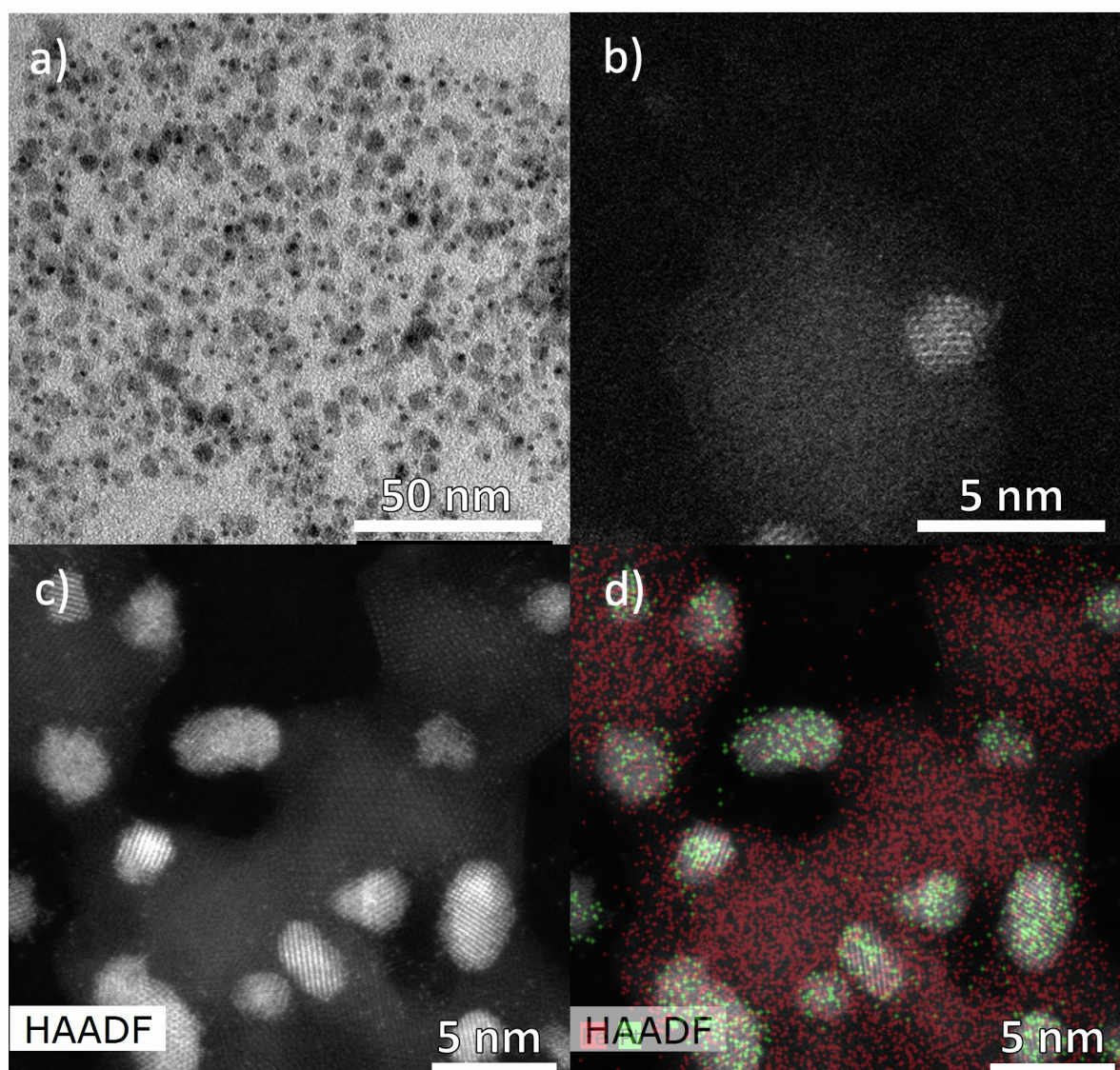
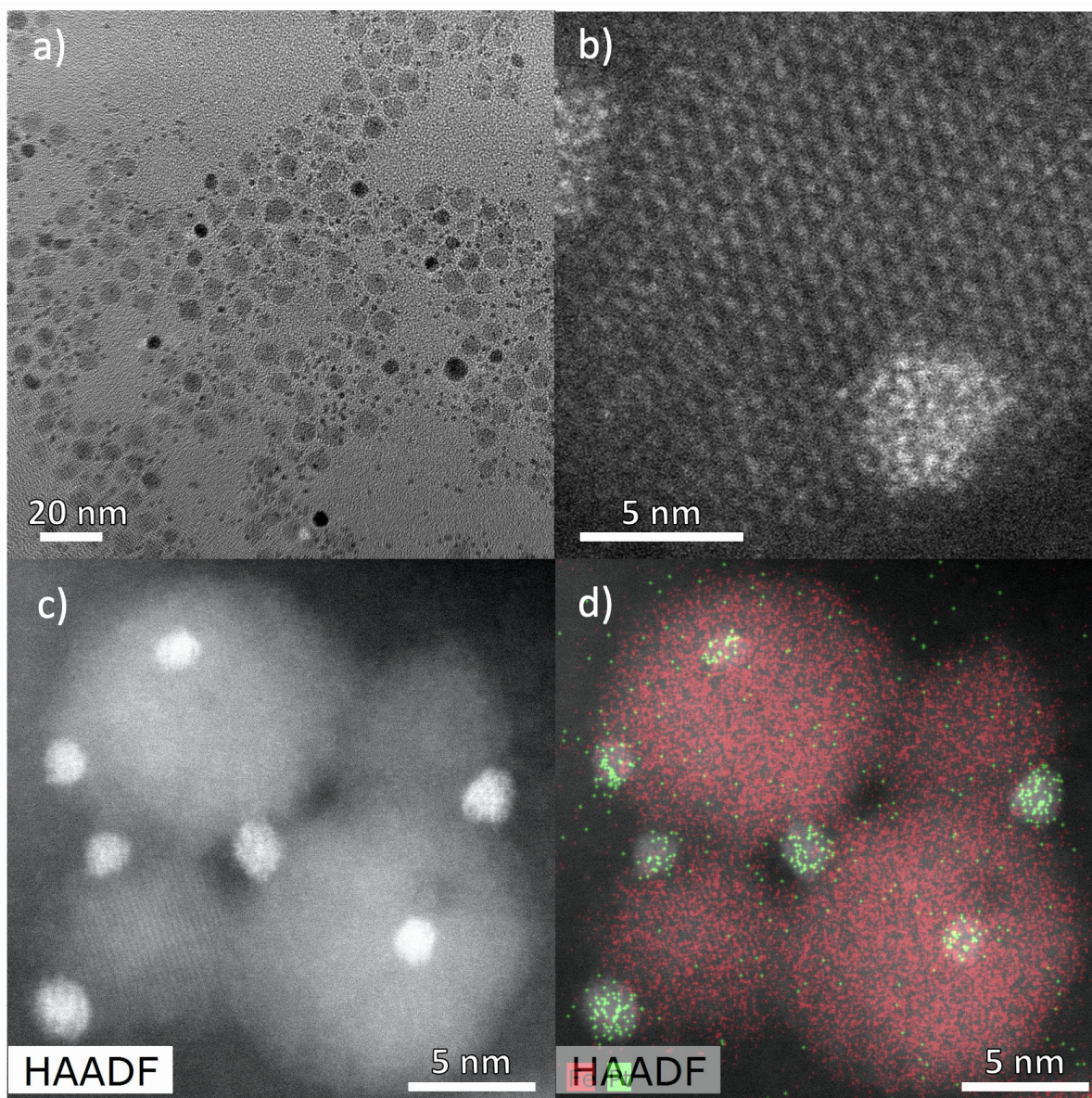


Figure S21. TEM images of Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles prepared using the traditional glassware synthesis. a) Representative TEM images, b) HR-TEM, c) STEM, and d) EDS elemental mapping, showing the Pt (green), and Fe (red)



**Figure S22.** TEM images of Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles prepared using the automated encoded reactor. a) Representative TEM images, b) HR-TEM, c) STEM, and d) EDS elemental mapping, showing the Pt (green), and Fe (red).

### 13. Digital steps from xDL

It is important to normalize the synthetic procedure to improve the reliability and reproducibility of chemical synthesis. To normalize and identify the synthetic procedures we generated the digital steps with the chemical programming language (xDL).

#### CdSe QD

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    <Component
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      type="reactor" />
    <Component
      id="reactor"
      type="reactor" />
    <Component
      id="sample_vial"
      type="reactor" />
  </Hardware>

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      role="reagent" />
    <Reagent
      name="ODE"
      role="reagent" />
    <Reagent
      name="TOP"
      role="reagent" />
    <Reagent
      name="cadmium acetate"
      role="reagent" />
    <Reagent
      name="Se"
      role="reagent" />
    <Reagent
      name="toluene"
      role="reagent" />
  </Reagents>

  <Procedure>
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    />
    <AddSolid
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      reagent="Cd acetate"
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      vessel="compartment_1"
      reagent="oleic acid"
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      vessel="compartment_1"
      reagent="ODE"
      volume="11 mL" />
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      temp="130°C"
      stir="true"
      time="1 hr" />
    <Purge
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    />
    <AddSolid
      vessel="compartment_2"
      reagent="Se"
      mass="0.158 g"
      stir="True" />
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      reagent="toluene"
      volume="0.9 mL"
      stir="True" />
    <Add
      vessel="compartment_2"
      reagent="TOP"
      volume="8 mL"
      stir="True" />
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      time="30 min" />
    <Purge
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    />
    <AddSolid
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      reagent="ODA"
      mass="0.220 g"
      stir="True" />
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      reagent="ODE"
      volume="5 mL"
      stir="True" />
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      temp="130°C"
      stir="true"
      time="30 min" />
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      to_vessel="reactor"
      volume="2.5 mL" />
    <Transfer
      from_vessel="compartment_2"
      to_vessel="reactor"
  </Procedure>
```

```
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  stir="true"  
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<Transfer  
  from_vessel="reactor"
```

```
    to_vessel="sample_vial"  
    volume="0.5 mL" />  
<HeatChill  
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  stir="true" />  
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</Synthesis>
```

## CdSe@ZnSe QD

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    <Component
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      type="reactor" />
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      id="compartment_1"
      type="reactor" />
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      role="reagent" />
    <Reagent
      name="HAD"
      role="reagent" />
    <Reagent
      name="CdSe_core"
      role="reagent" />
    <Reagent
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    <Reagent
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      role="reagent" />
    <Reagent
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      role="reagent" />
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      reagent="TOPO"
      mass="2.25 g" />
    <AddSolid
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      reagent="HAD"
      mass="1.26 g" />
    <Add
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      reagent="CdSe_core"
      volume="2 mL"
      stir="true" />
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      stir="true"
      time="15 min" />
    <Purge
      vessel="reactor_2"
      reagent="argon" />
    <AddSolid
      vessel="reactor_2"
      reagent="Zn"
      mass="0.158 g"
      stir="True" />
    <Add
      vessel="reactor_2"
      reagent="ODE"
      volume="1.5 mL"
      stir="True" />
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      temp="60°C"
      stir="true"
      time="30 min" />
    <HeatChill
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      temp="25°C"
      stir="true" />
    <Purge
      vessel="reactor_3"
      reagent="argon" />
    <AddSolid
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      reagent="Se"
      mass="0.0197 g"
      stir="True" />
    <Add
      vessel="reactor_3"
      reagent="TOP"
      volume="1.5 mL"
      stir="True" />
    <Stir
      vessel="reactor_2"
      time="1 h" />
    <Transfer
      from_vessel="reactor_2"
      to_vessel="compartment" />
    <Transfer
      from_vessel="reactor_3"
      to_vessel="compartment" />
    <Stir
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      time="30 min" />
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      to_vessel="reactor"
      time="4 min" />
```

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  temp="190°C"  
  stir="true"  
  time="1 hr" />  
</HeatChill
```

```
  vessel="reactor"  
  temp="25°C"  
  stir="true" />  
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</Synthesis>
```

## Pt-Fe<sub>3</sub>O<sub>4</sub> Janus nanoparticles

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    <Component
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      type="reactor" />
    <Component
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      type="reactor" />
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  <Reagents>
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    <Reagent
      name="OLA"
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    <Reagent
      name="octyl ether"
      role="reagent" />
    <Reagent
      name="Pt acac"
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    <Reagent
      name="Fe"
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    <Reagent
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  <Procedure>
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      reagent="argon" />
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      reagent="HDD"
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    <Add
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      reagent="octyl ether"
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      stir="true" />
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      reagent="OLA"
      volume="0.086 mL"
      stir="true" />
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      temp="200°C"
      stir="true"
      time="15 min" />
    <Purge
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      reagent="argon"/>
    <AddSolid
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      reagent="Pt acac"
      mass="0.0985 g" />
    <Add
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      reagent="octyl ether"
      volume="5 mL"
      stir="true" />
    <Purge
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      reagent="argon" />
    <AddSolid
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      reagent="Fe"
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      stir="True" />
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      reagent="DDA"
      mass="1.39 g"
      stir="True" />
    <HeatChill
      vessel="compartment_2"
      temp="40°C"
      stir="true"
      time="15 min"/>
    <Transfer
      from_vessel="compartment_1"
      to_vessel="reactor"
      volume="5 mL" />
    <HeatChill
      vessel="reactor"
      temp="200°C"
      stir="true"
      time="20 min"/>
    <Transfer
      from_vessel="compartment_2"
      to_vessel="reactor" />
    <HeatChill
      vessel="reactor"
      temp="200°C"
      stir="true"
      time="1 hr"/>
    <HeatChill
      vessel="reactor"
      temp="25°C"
      stir="true" />
  </Procedure>
</Synthesis>
```