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Sodium-Metal and Strontium-Metal Nanoparticles

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Nanoparticles of zerovalent sodium metal (5 ± 1 nm) and strontium metal (7 ± 3 nm) are realized in the liquid phase for the first time. The synthesis is performed in toluene with NaCp or SrI₂ and TMEDA-stabilized [LiNaph] as reducing agent (TMEDA: N,N,N',N'-tetramethylethylenediamine; [LiNaph]: lithium naphthalenide). Both nanoparticles are crystalline and highly reactive as exemplarily indicated by the formation of phase-pure alkalides.

The knowledge about metal nanoparticles is the smaller the more basic the respective metal. In particular, this relates to the great number of base metals with electrochemical potentials (E^0) of -1.0 to -2.5 V (taking E^0 of the respective bulk metal as an indicator).¹ The limited knowledge about base-metal nanoparticles can be illustrated by the annual number of publications of selected metal nanoparticles.² Thus, more than 5,000 publications addressed the synthesis of Au(0) nanoparticles ($E^0(\text{bulk-Au}) = +1.5$ V)¹ in 2025,² about 500 publications were related to the synthesis of Co(0) nanoparticles ($E^0(\text{bulk-Co}) = -0.3$ V),^{1,2} whereas no report in 2025 addressed the synthesis of, for instance, Ti(0) nanoparticles ($E^0(\text{bulk-Ti}) = -1.9$ V).¹ Current knowledge can be correlated to the synthesis of the base-metal nanoparticles, which is the more demanding the higher their reactivity and the smaller their size.³

Base-metal nanoparticles have generally attracted our interest. In recent years, we have developed various methods to prepare base-metal nanoparticles, including microemulsions with liquid ammonia, sodium-driven reduction in liquid ammonia, lithium/sodium pyridinyl-driven reduction in pyridine, or the lithium/sodium naphthalide-driven ([LiNaph], [NaNaph]) reduction in THF.⁴ In addition to the development of syntheses strategies, base-metal nanoparticles turned out to be useful starting materials for redox reactions with small molecules (O₂, S₈, NH₃, ROH, etc.), sterically demanding O–H/N–H-acidic alcohols/amines or cyclic ethers to obtain new coordination compounds, metal-metal bonding, or cluster compounds.^{4d,5} Particularly, the reduction of metal halides by [LiNaph] in THF turned out to be a versatile approach. Thus, we could

realize all group 3-to-group 6 transition metals, all lanthanide metals (La to Lu), Mg and Al with a size of 1–5 nm.^{4d} These metals – as bulk metals – have electrochemical potentials in the range of -1.0 to -2.5 V.¹ Sodium and strontium, however, exhibit even more negative electrochemical potentials of -2.7 V and -2.9 V.¹ These highly negative values add further challenges to the synthesis of nanoparticles. First, THF is no longer stable as a solvent. Thus, reductive polymerization of THF is preferred over the reduction of Na⁺ or Sr²⁺ in THF.⁶ Such behaviour and reaction were already described for the reaction of THF and alkali metals.⁷ Moreover, the reduction potential of [LiNaph] in THF was reported with about -2.3 V, so that the reducibility of Na⁺/Sr²⁺ with [LiNaph] is questionable anyway.⁸

Aiming at Na(0) and Sr(0) nanoparticles, a modification of our previous synthesis strategy using [LiNaph]-driven reduction in THF was necessary as otherwise THF is either reductively polymerized⁶ or naphthalides such as [Sr(naph)(thf)₂], containing Sr²⁺ and [naph]²⁻, are formed.^{5d} Instead of THF, therefore, we have selected toluene as the solvent, which – due to the absence of any heteroatom – can be expected to be less coordinative and with even higher redox stability. As another difficulty, however, all starting materials [LiNaph], NaCp, and SrI₂ are insoluble in toluene (*SI: Figures S1,S2*). From the synthesis of Al(0) nanoparticles,⁹ we learned that [LiNaph] becomes soluble in toluene upon coordination to N,N,N',N'-tetramethylethylenediamine (TMEDA) (*Figure 1; SI: Figure S3*). The NaCp and SrI₂, however, remain insoluble in toluene even in the presence of TMEDA.

Due to the aforementioned restrictions regarding the solubility of NaCp and SrI₂ in toluene, both were here added as pure powders. Despite these – at first sight – non-optimal conditions for nanoparticle preparation, the reduction and nucleation of Na(0) and Sr(0) nanoparticles were nevertheless successful. On the one hand, the dissolution of NaCp and SrI₂ is very slow, whereas the reduction by [LiNaph] after dissolution is very fast (*Figure 1; SI: Figure S3*). After reduction, moreover, sodium and strontium metal are both highly insoluble in toluene. These effects – fast reduction and low solubility of the as-prepared metals – cause a high supersaturation, promoting the formation of small-sized particles.¹⁰ Successful reduction and nucleation of Na(0) and Sr(0) nanoparticles are indicated by the formation of greyish to black suspensions with slightly reddish (Na) to bluish (Sr) appearance (*Figure 1; SI: Figure S3*). Whereas the

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* Electronic Supplementary Information (ESI) available: analytical equipment and additional analytic data on the Na(0) and Sr(0) nanoparticles.



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dissolution and reduction of NaCp took some minutes, the dissolution of SrI_2 is very slow and takes about two weeks to be completed. Finally, it must be noticed that the as-prepared Sr(0) nanoparticles were very small (1-2 nm) and difficult to separate via centrifugation. Here, certain heating (100 °C) was applied to slightly increase the particle size (7 ± 3 nm). Such heating was not necessary for the Na(0) nanoparticles, which were prepared at room temperature only.

Subsequent to synthesis, the as-prepared Na(0) and Sr(0) nanoparticles were purified by centrifugation and repeated redispersion/centrifugation in/from TMEDA and toluene to remove remaining naphthalene, TMEDA and LiCp or LiI. Finally, the Na(0)/Sr(0) nanoparticles were either dried in vacuum (10^{-3} mbar) at room temperature to obtain powder samples (Figure 1; *SI: Figure S3*), or they were redispersed in toluene or TMEDA to obtain suspensions. It should be noticed that handling of the Na(0) and Sr(0) nanoparticles requires specific attention as they are highly pyrophoric and even show explosion when in contact to water or other oxidizing agents (*SI: Figures S4,S5*).

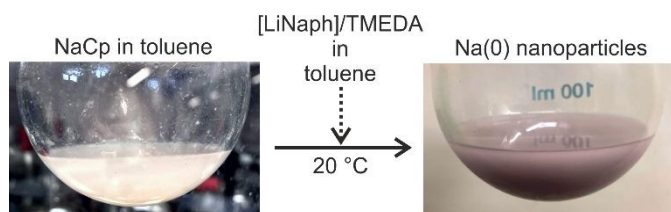


Fig. 1 Scheme illustrating the liquid-phase synthesis of Na(0) with photos of NaCp precursor in toluene and the as-prepared Na(0) nanoparticles in toluene (see Supporting Information for synthesis of Sr(0) nanoparticles: Figure S3).

Transmission electron microscopy (TEM) is most relevant for characterization and to determine size, shape, composition, and crystallinity of the as-prepared Na(0) and Sr(0) nanoparticles. However, TEM analysis turned out to be time consuming and challenging due to the high reactivity of the metal nanoparticles. First of all, contamination with oxygen – predominately due to traces of water molecules remaining adsorbed on all kinds of surfaces – is a challenge. Although suitable transfer modules were used, the few nanoparticles deposited on a TEM grid are reactive enough to attract all traces of moisture. To this concern, pre-treatment of TEM grids by heating in vacuum and fast transfer from a glove-box into the TEM device (< 30 min) are important. Moreover, the Na(0) and Sr(0) nanoparticles can react with the Lacey-carbon film of the TEM grid under high-energy electron bombardment, which requires precise examination and high experience for TEM characterization.

The as-prepared Na(0) and Sr(0) nanoparticles exhibit a uniform spherical shape with a size in the 5 nm-range and a low degree of agglomeration (Figure 2a,3a; *SI: Figures S6,S7*). Statistical evaluation of about 100 metal nanoparticles on TEM images resulted in a mean particle diameter of 5 ± 1 nm for the Na(0) nanoparticles (Figure 2b) and 7 ± 3 nm for Sr(0) nanoparticles (Figure 3b). High-resolution (HR)TEM images confirm the presence and crystallinity of the as-prepared Na(0) and Sr(0) nanoparticles, showing lattice fringes extending through the whole particle (Figures 2c,3c; *SI: Figures S6,S7*). For the Na(0) nanoparticles, the observed average lattice-fringe distance of 2.9 ± 0.1 Å agrees with body-centred cubic (bcc) bulk sodium ($d_{110} = 3.03$ Å).¹¹ The two-dimensional Fourier transformation (FT) pattern of the nanoparticle on the HRTEM image

(Figure 2c) is in agreement with the calculated diffraction pattern of

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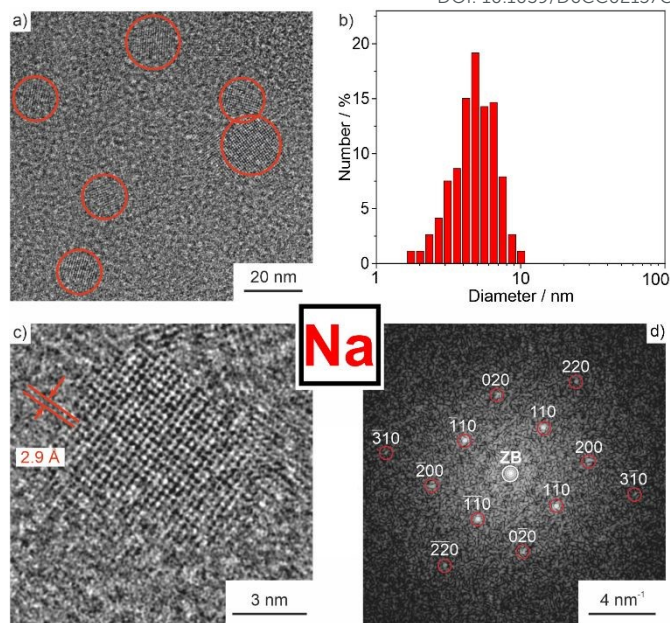


Fig. 2 Electron microscopy of the as-prepared Na(0) nanoparticles: (a) HRTEM overview image with single nanoparticles (indicated by red dots), (b) size distribution (according to a statistical evaluation of > 100 nanoparticles on HRTEM images), (c) HRTEM image of a single nanoparticle with lattice fringes, (d) FT pattern of single nanoparticle shown in (c) agrees with the calculated diffraction pattern of bcc-bulk Na (red circles and Miller indices) in the [001] zone axis (zero-order beam, ZB, marked by white circle).

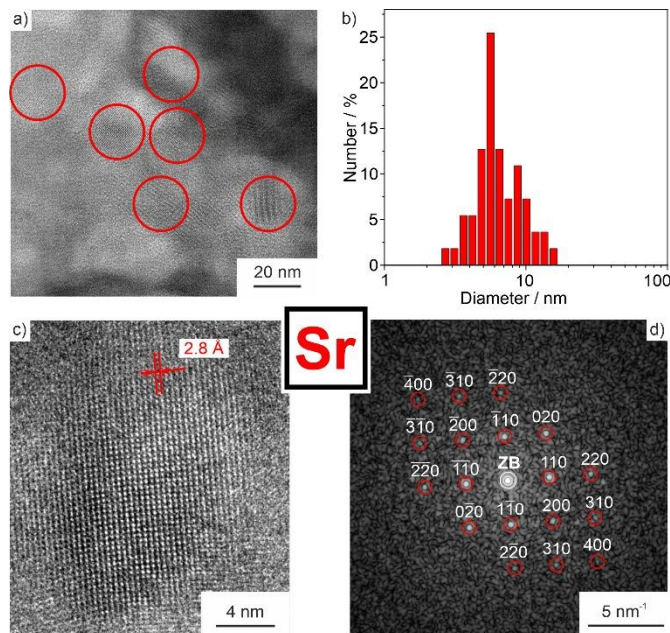


Fig. 3 Electron microscopy of the as-prepared Sr(0) nanoparticles: (a) HRTEM overview image with single nanoparticles (indicated by red dots), (b) size distribution (according to a statistical evaluation of > 60 nanoparticles on HRTEM images), (c) HRTEM image of a single nanoparticle with lattice fringes, (d) FT pattern of single nanoparticle shown in (c) agrees with the calculated diffraction pattern of bulk cubic γ -Sr (red circles and Miller indices) in the [001] zone axis (zero-order beam, ZB, marked by white circle).



bcc-bulk sodium within the [001]-zone axis (Figure 2d).¹¹ In the case of Sr(0) nanoparticles, the observed average lattice-fringe distance of $2.8 \pm 0.1 \text{ \AA}$ corresponds well to the (110) lattice-plane distances ($d_{110} = 2.91 \text{ \AA}$) of bulk cubic γ -Sr (Figure 3c).¹² Again, the 2D-Fourier transform of a single nanoparticle is in good agreement with the calculated diffraction pattern of bulk cubic γ -Sr (space group: *Im-3m*; lattice parameter: $a = 4.87 \text{ \AA}$) in the [001]-zone axis (Figure 3d).¹² Finally, it should be noticed that the HRTEM images did not show any oxide species.

The crystallinity of the Na(0) and Sr(0) nanoparticles was also examined by X-ray powder diffraction (XRD) (Figure 4). Here, it needs to be noticed that the metal nanoparticles need to be mortared and diluted with dried quartz powder to enable a filling of glass capillaries (0.3 mm in diameter) for XRD analysis. After mortaring, especially the low-melting sodium ($T_{\text{melt}}: 98 \text{ }^\circ\text{C}$) is bulk-like and well-crystallized whereas only broad reflexes hint to the presence of the high-melting strontium ($T_{\text{melt}}: 777 \text{ }^\circ\text{C}$). Due to small crystallite sizes and high X-ray absorption of Sr(0), the scattering intensity is low here. Although Na(0) loses its nanoscaled size due to mortaring, XRD is nevertheless interesting as only Bragg peaks of the pure metal are observed (Figure 4). Impurity phases (e.g., halides, oxides, hydroxides, carbonates) are not observed. Fourier-transform infrared (FT-IR) spectra, recorded by attenuated total reflection (ATR) to examine the particle surface, show only weak vibrations of toluene or naphthalene ($1575, 1238, 734 \text{ cm}^{-1}$) for both the Na(0) and the Sr(0) nanoparticles (Figure 5). Interestingly, vibrations of the most strongly coordinating chelating ligand TMEDA are not observed.

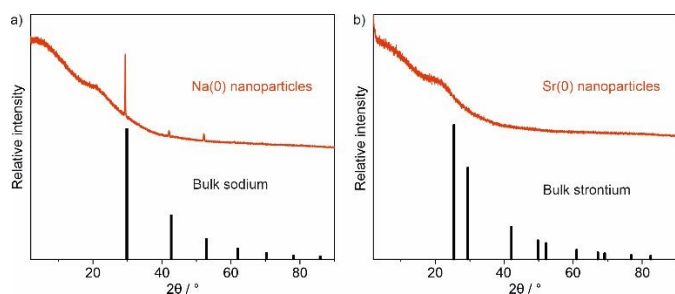


Fig. 4 XRD of (a) Na(0) and (b) Sr(0) nanoparticles with bulk sodium (ICDD-No. 01-071-4606) and bulk strontium (ICDD-No. 01-089-4045) as references. It must be noticed that filling of glass capillaries ($\varnothing: 0.3 \text{ mm}$) required dilution with dried quartz powder and mortaring, which causes fusing of the low-melting Na(0) nanoparticles to bulk-like metal.

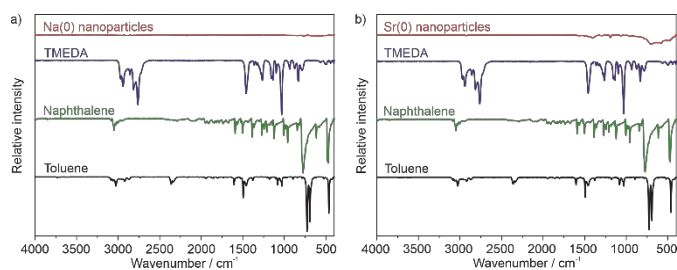


Fig. 5 FT-IR spectra of the as-prepared (a) Na(0) and (b) Sr(0) nanoparticles with spectra of TMEDA, naphthalene, and toluene as references. All spectra were recorded with ATR mode.

Elemental analysis (C/H/N) analysis of the Na(0) nanoparticles resulted in a surface conditioning containing 0.6 wt-% N, 26.0 wt-%

C, and 2.5 wt-% H. In the case of the Sr(0) nanoparticles, 1.9 wt-% N, 40.9 wt-% C, and 3.9 wt-% H were detected. Hereof, the N content can be attributed to TMEDA. Moreover, the C : H ratio of 10 : 1 for both metals points to a surface functionalization predominately of toluene (10 : 1) than of naphthalene (15 : 1) in the range of a monolayer. In addition to C/H/N analysis, the thermal properties of the Na(0) and Sr(0) nanoparticles were evaluated by thermogravimetry (TG). For Na(0), first, an increase of mass (62%) is observed (150–400 $^\circ\text{C}$), followed by a mass loss (32%, 500–1000 $^\circ\text{C}$) (Figure 6a), which can be related to the formation of sodium hyperoxide (NaO_2) (calcd. 59%) followed by the formation of sodium oxide (Na_2O) (calcd. 30%), which, as a strong base, at this temperature reacts with the corundum crucible. In the case of the Sr(0) nanoparticles, certain fluctuation of the mass is observed up to 400 $^\circ\text{C}$, which can be attributed to opposite processes of reaction with O_2 and CO_2 and release of the surface-adhered molecules (Figure 6b). Above 750 $^\circ\text{C}$, the release of CO_2 (38%) of the intermediately formed SrCO_3 occurred (calcd. 30%). Here, the thermal remnant was identified by XRD as a mixture of SrO and $\text{Sr}_3\text{Al}_2\text{O}_6$,¹³ whereof the latter results from the reaction of SrO with the corundum crucible at high temperature (SI: Figure S8).

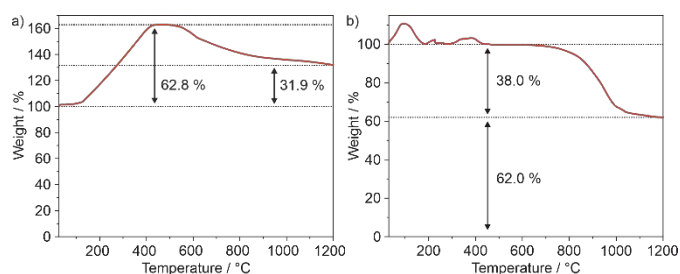


Fig. 6 TG of (a) Na(0) and (b) Sr(0) nanoparticles in air (heating rate: 5 K/min; crucible material: corundum) (for XRD analysis of the thermal remnant see SI: Figure S8).

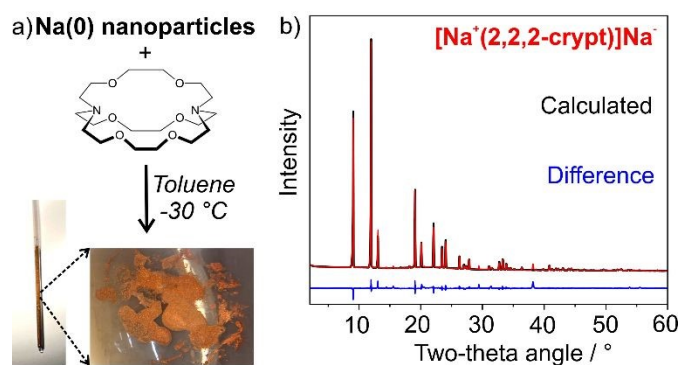


Fig. 7 Synthesis of alkalis with Na(0) nanoparticles: (a) scheme of reaction with photo of as-prepared bronze-coloured crystals of $[\text{Na}(2,2,2\text{-cryptand})]^+\text{Na}^-$; (b) XRD of $[\text{Na}(2,2,2\text{-cryptand})]^+\text{Na}^-$ (red) with Rietveld refinement (calcd. XRD: black; difference: blue).

To probe the reactivity of the metal nanoparticles and to verify their suitability for chemical syntheses, we have examined the potential formation of alkalis.¹⁴ Thus, the as-prepared Na(0) nanoparticles were dispersed in toluene and reacted with 2,2,2-cryptand in solution. At $-30 \text{ }^\circ\text{C}$ over 48 h, great amounts of bronze-coloured crystals were obtained (Figure 7a). XRD with Rietveld refinement indicates these crystals as a pure alkali with the



composition [Na(2,2,2-cryptand)]⁺Na⁻ (Figure 7b). Such alkalides were yet only prepared by dissolution of the alkali metal and cryptand in liquid ammonia as initially introduced by James L. Dye.^{14,15} Recently, [Na(2,2,2-cryptand)]⁺Na⁻ was also reported to be available from reactive ball milling.¹⁶ The formation of alkalides using metal nanoparticles as starting material is demonstrated for the first time, and the possibility to perform the reaction in toluene may further expand the repertoire for chemical syntheses.

In conclusion, zerovalent sodium and strontium metal nanoparticles are realized in the liquid phase as nanoparticles for the first time. Due to their high reactivity, the synthesis was performed in toluene with TMEDA-stabilized [LiNaph] as reducing agent as well as NaCp or SrI₂ as starting material (TMEDA: N,N,N',N'-tetramethylethylenediamine; [LiNaph]: lithium naphthalenide). This results in colloiddally stable suspensions of Na(0) and Sr(0) nanoparticles with a size of 5 ± 1 nm and 7 ± 3 nm. Both nanoparticles are crystalline, showing the structural features of body-centred cubic sodium and cubic γ-strontium. In air, the Na(0) nanoparticles react with O₂ to the hyperoxide NaO₂ (≥ 150 °C) as an intermediate and thereafter to the oxide Na₂O (≥ 500 °C), whereas the Sr(0) nanoparticles react to SrCO₃, which decomposes to SrO (≥ 750 °C). The reactivity of the metal nanoparticles is further emphasized by direct formation of the pure alkalide [Na(2,2,2-cryptand)]⁺Na⁻ upon reaction of Na(0) nanoparticles with 2,2,2-cryptand in toluene. Despite the first liquid-phase synthesis of Na(0) and Sr(0) nanoparticles, they can also offer the option for a use as reactive starting materials in non-polar solvents.

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Data Availability Statement

Additional data regarding experiments and methods can be obtained from the Supporting Information and on request from the authors.

Conflicts of interest

The authors declare no conflicts of interest.

Notes and references

#Safety advice: The as-prepared Na(0) and Sr(0) nanoparticles are highly reactive. They show instantaneous combustion when in contact to air or other oxidizing agents as well as explosion when in contact to water. The reactivity of the nanoparticles is comparable to the heavy bulk alkali metals rubidium and cesium.

Experimental: All experiments and purification were performed with inert gas (argon), using standard Schlenk techniques or glove boxes (MBraun, Germany). This also included all centrifugation and washing procedures. Toluene (Tol, 99 %, Merck, Germany) and tetrahydrofuran (THF, HPLC grade, Sigma-Aldrich, Germany) were distilled over potassium with benzophenone as indicator. N,N,N',N'-Tetramethylethylenediamine (TMEDA, 99 %, Sigma-Aldrich, Germany) was distilled over Na. Lithium (99 %, Sigma-Aldrich), naphthalene (99 %, Sigma-Aldrich), SrI₂ (99 %, Fisher Scientific, Germany), and 222-cryptand (Kryptofix 222, C₁₈H₃₆N₂O₆, 98 %, Sigma-Aldrich) were used as purchased. NaCp was synthesized according to the literature.¹⁷

Na(0) nanoparticles: 10 mg of Li (1.4 mmol) and 175 mg of naphthalene (1.4 mmol) were dissolved in a mixture of 500 μL of TMEDA (3.4 mmol) and 15 mL toluene. A dark violet colour indicates the dissolution of Li and formation of [LiNaph]. To complete reaction and dissolution of Li, the solution was intensely stirred for 2-3 days. Separately, 115 mg of NaCp (1.3 mmol) were suspended in 10 mL of toluene. Subsequently, the [LiNaph] solution was injected into the NaCp suspension with vigorous stirring, which results in a greyish black suspension of Na(0) nanoparticles with a slight reddish colour within about 5 min. The nanoparticles were purified by centrifugation/redispersion from/in toluene and THF. It should be noticed that the centrifugation needs to be carried out <8000×g to prevent agglomeration of the as-prepared Na(0) nanoparticles.

Sr(0) nanoparticles: 10 mg of Li (1.4 mmol) and 175 mg of naphthalene (1.4 mmol) were dissolved in a mixture of 500 μL TMEDA (3.4 mmol) and 20 mL toluene. The dark violet colour indicates the dissolution of Li and the formation of the [LiNaph]. By intensively stirring the solution for 2-3 days, a complete dissolution of Li was guaranteed. To this [LiNaph] solution, 205 mg of SrI₂ (0.6 mmol) were added as a powder. The reaction and formation of Sr(0) nanoparticles is indicated by the formation of a black suspension with a slight bluish colour. Due to the low solubility of SrI₂ in toluene, the reaction takes 1-2 weeks to be completed. Finally, the suspension of Sr(0) nanoparticles was heated to 100 °C for 6 h with reflux to support certain growth of the Sr(0) nanoparticles. Afterwards, the nanoparticles were centrifuged and dried in vacuum to obtain powder samples, or they were redispersed in THF or toluene to obtain colloiddally stable Sr(0) nanoparticles suspensions.

The alkalide [Na(2,2,2-cryptand)]⁺Na⁻ was prepared by adding 100 mg of 222 cryptand (0.3 mmol) at room temperature to a suspension of 24 mg Na(0) nanoparticles (1.0 mmol) in 5 mL of toluene. Upon addition, the colour of the suspension turned immediately to blue. Bronze-coloured small-sized crystals were obtained by storing the reaction vessel at -30 °C for 48 h. The yield of [Na(2,2,2-cryptand)]⁺Na⁻ can be assumed to about 80%. During separation via centrifugation part of the alkalide remains adhered on the centrifugation tubes.

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Data availability statement

Additional data regarding experiments and methods can be obtained from the Supporting Information and on request from the authors.

