

Supplemental Information

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Materials and Methods

Materials: DNA staple strands were purchased from IDT. M13 bacteriophage scaffold DNA (p7560) was produced following a published protocol.¹ 10 nm gold nanoparticles (AuNPs) were purchased from Ted Pella, Inc.

DNA origami hexagon tile design and assembly: DNA origami hexagon tile (DHT) was designed by using caDNAno. In a typical annealing protocol, staples were mixed with single stranded DNA scaffold (p7560, 10 nM) in 5-fold molar excess in aqueous buffer composing of 5 mM Tris base, 1 mM EDTA, and 10 mM MgCl₂, with a total volume of 100 μ L. The mixed solution was slowly cooled down to room temperature (24 °C) in a PCR thermal cyclor over 18 hrs under the following protocol: 65 °C for 5 mins, 60 to 24 °C, at 30 mins/°C.

Conjugating DNA onto AuNPs: 50 mL of colloidal citrated 10nm AuNP solution was added with 15 mg of Bis(p-sulfonatophenyl) phenylphosphine dihydrate dipotassium salt (BSPP) and shook overnight. Then NaCl was added until a color change of the solution from red to blue was observed. The AuNPs were centrifuged to the bottom at 1,000 rcf for 30 min and the clear supernatant was removed with a pipette. The particles were dissolved in BSPP solution (1 mL, 2.5 mM) followed by addition of 3 mL of methanol. The particles were centrifuged again and re-suspended in 1 mL of BSPP. The concentration of the AuNPs was determined from the optical absorption at a wavelength of 520 nm using a Nanodrop spectrophotometer. For reducing the disulfide bonds of the thiolated ssDNA strands to monothiol, the modified strands were incubated with 100-fold of TCEP (Tris(carboxyethyl) phosphine hydrochloride) for at least 30 mins. AuNPs and thiolated DNA were mixed in 0.5×TBE buffer at a designated ratio (1: 100). Within a period of 24 hrs, NaCl solution was added to the solution to achieve a final concentration of 300 mM. To remove the unbound DNA strands, the mixture was spun for 30 min at 18,000 rcf, and the supernatant was removed carefully without disturbing the pellet. 4 additional washing and spinning steps (30 min, 18,000 rcf) with 100 μ L of 0.5 × TBE buffer containing 300 mM NaCl were conducted to fully removed unbound DNA.

Attaching AuNPs onto DNA origami templates: DNA origami structures were assembled first, as described above, which were then added with 5-fold (in terms of binding site, 1.5-fold for 2D lattice designs) of DNA-conjugated AuNPs, incubated for at least 2 hours at room temperature. Cluster structures were then subject to agarose gel electrophoresis and corresponding bands were extracted. Superlattice structures were directly characterized.

Gel electrophoresis: DNA origami with AuNPs forming clusters were run on 1% native agarose gel electrophoresis for 2 hours (gel prepared in 0.5 × TBE buffer supplemented with 10 mM MgCl₂ and 0.005% (v/v) EtBr) in an ice water bath. Then, the target gel bands were excised and placed into a

Freeze 'N Squeeze column (Bio-Rad Laboratories, Inc.). The gel pieces were crushed into fine pieces by a microtube pestle in the column, and the column was then centrifuged at 7000 rcf for 5 minutes. Samples that were extracted through the column were collected for subsequent experiments.

TEM imaging: 10 μ L of samples were deposited onto glow-discharged, carbon-coated copper TEM grids for 5-30 minutes inside a moisture chamber. Residual solution was dried by filter paper. The grids were then stained for 1 minute using a 1% aqueous uranyl formate solution containing 25 mM NaOH. Imaging was performed using a Hitachi-7700 microscope operated at 80 kV.

Stability study: 9-AuNP clusters were prepared as illustrated in the above protocol. 10 μ L of 10 nM nanoparticle clusters were incubated in 1 $\times$ TE/Mg²⁺(10 mM), 1 $\times$ PBS, or RPMI-1640/10% FBS at 37 $^{\circ}$ C for 0.5, 1, 2, or 4 hours. Immediately after incubation, the samples were subject to 1% native agarose gel electrophoresis running in 0.5 $\times$ TBE buffer supplemented with 10 mM MgCl₂.

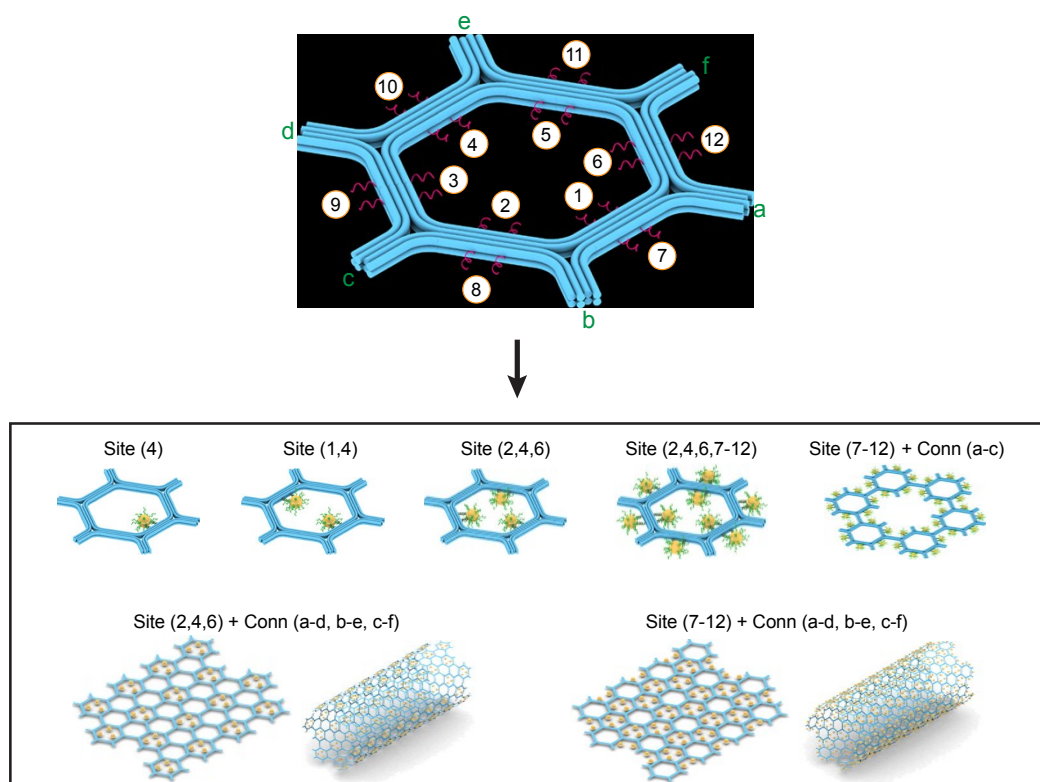
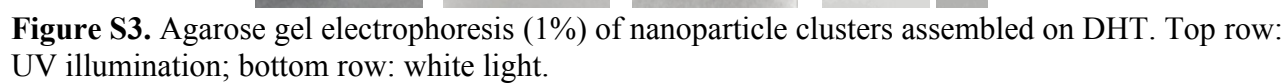
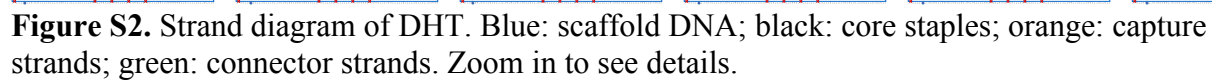


Figure S1. Summary figure. DHT-guided assembly of gold nanoparticle clusters and lattices. The use of docking handle strands and connector strands are indicated for each architecture.



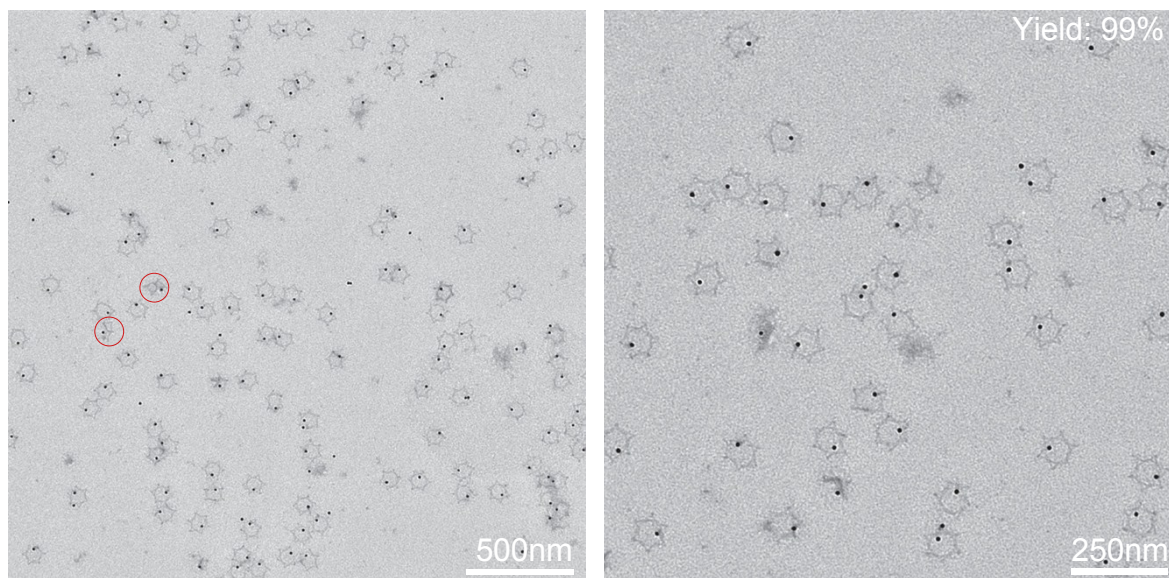


Figure S4. 1-AuNP cluster on DHT. Yield is calculated based on TEM images (N=116). Wrongly assembled clusters were marked by a red circle.

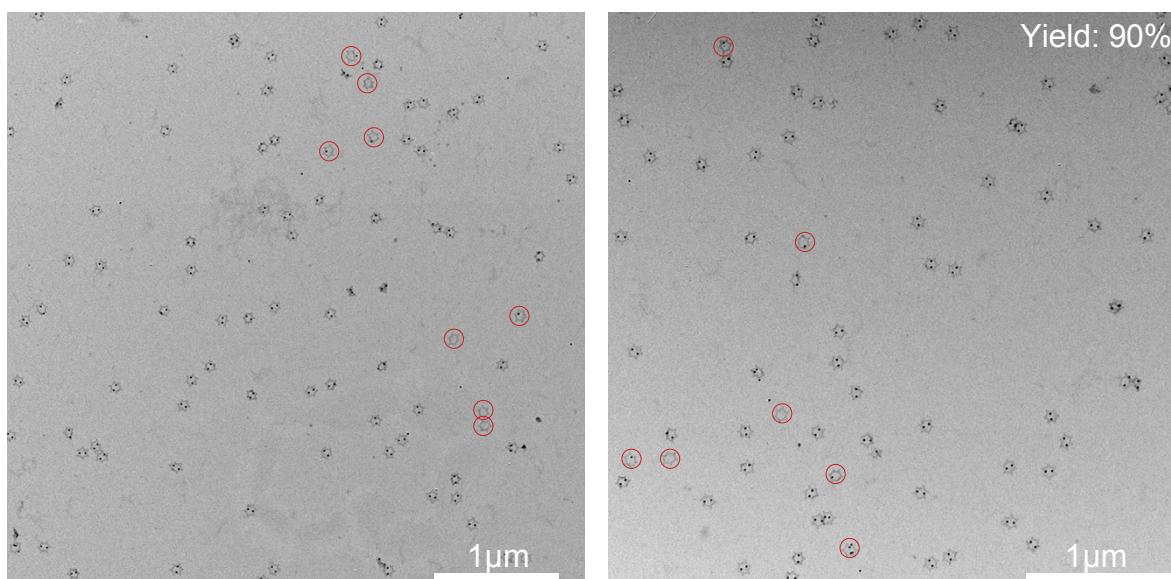


Figure S5. 2-AuNP cluster on DHT. Yield is calculated based on the images (N=205). Wrongly assembled clusters were marked by a red circle.

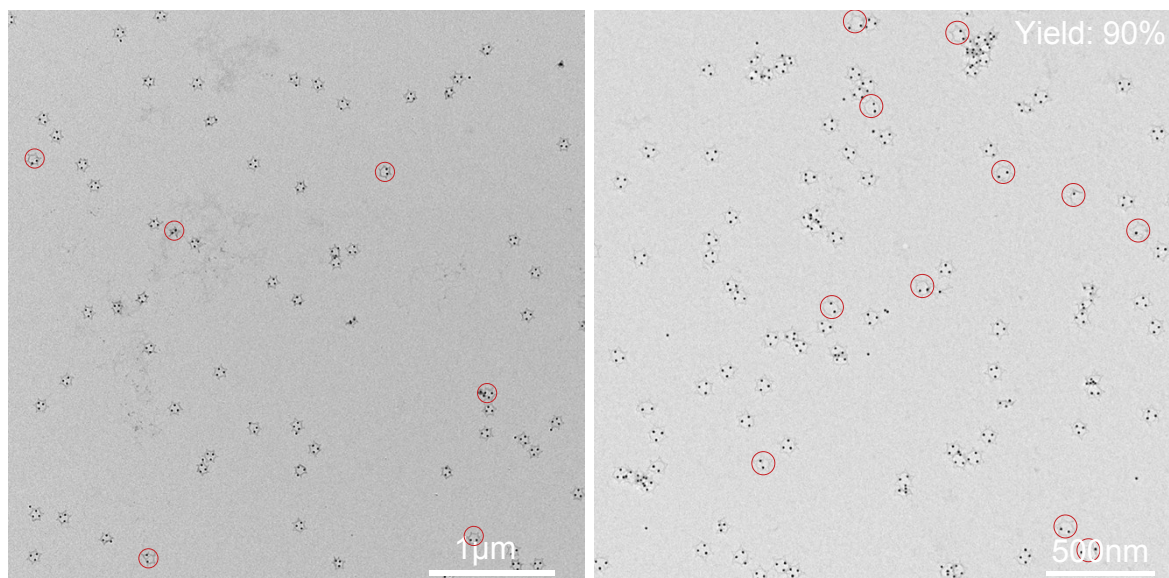


Figure S6. 3-AuNP cluster on DHT. Yield is calculated based on the images (N=155). Wrongly assembled clusters were marked by a red circle.

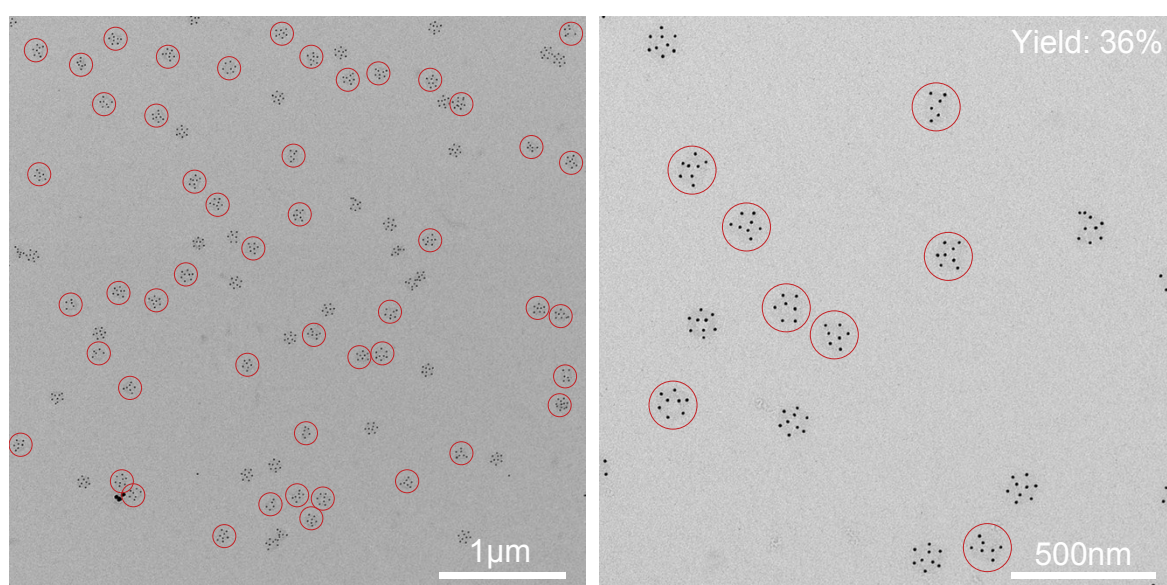


Figure S7. 9-AuNP cluster on DHT. Yield is calculated based on the images (N=87). Wrongly assembled clusters were marked by a red circle.

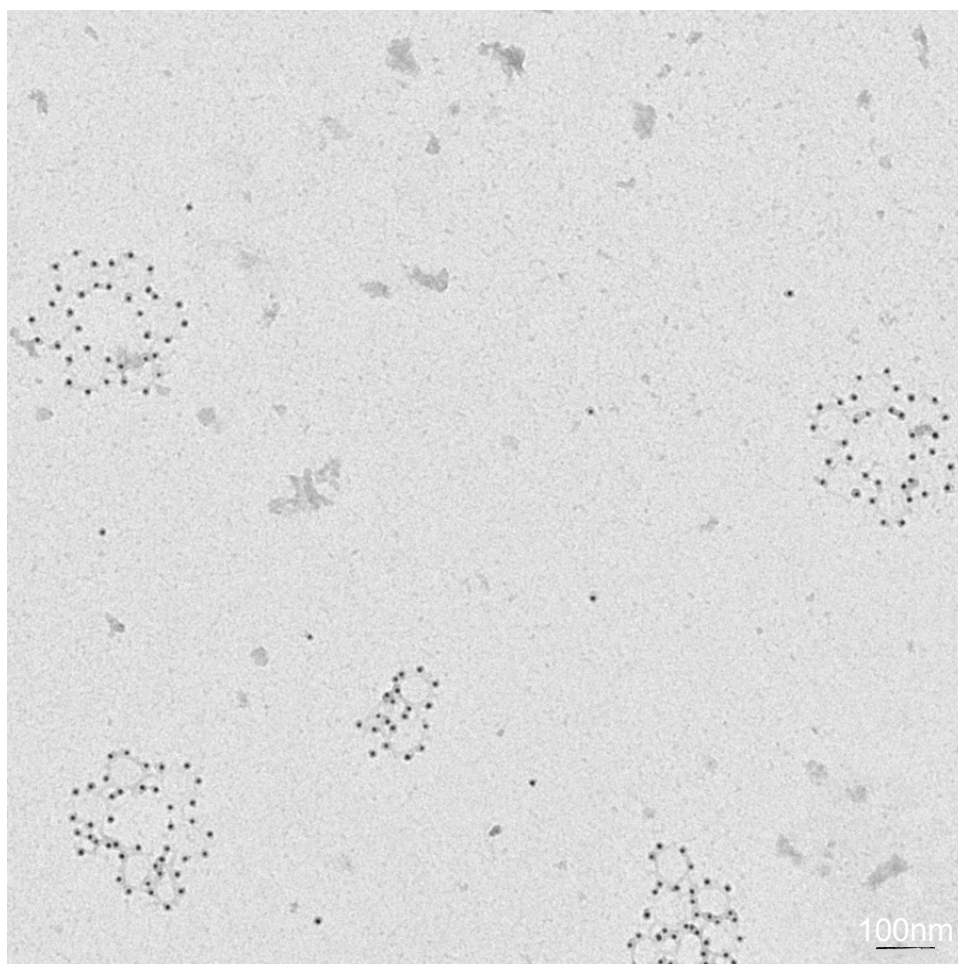


Figure S8. 36-AuNP cluster on DHT hexamer.

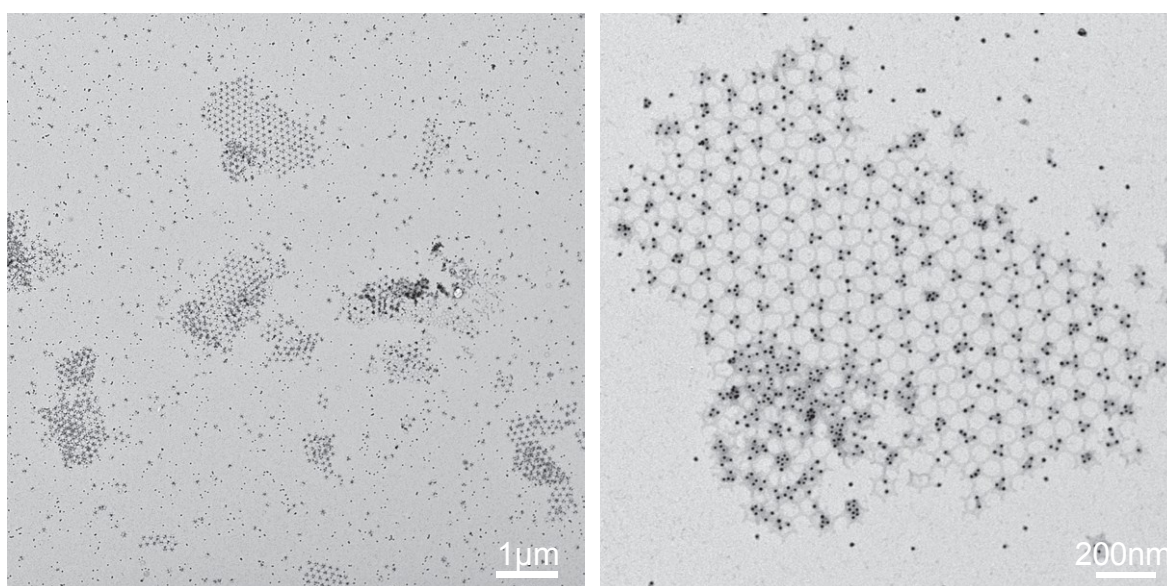


Figure S9. AuNP lattices on honeycomb array with type-1 configuration.

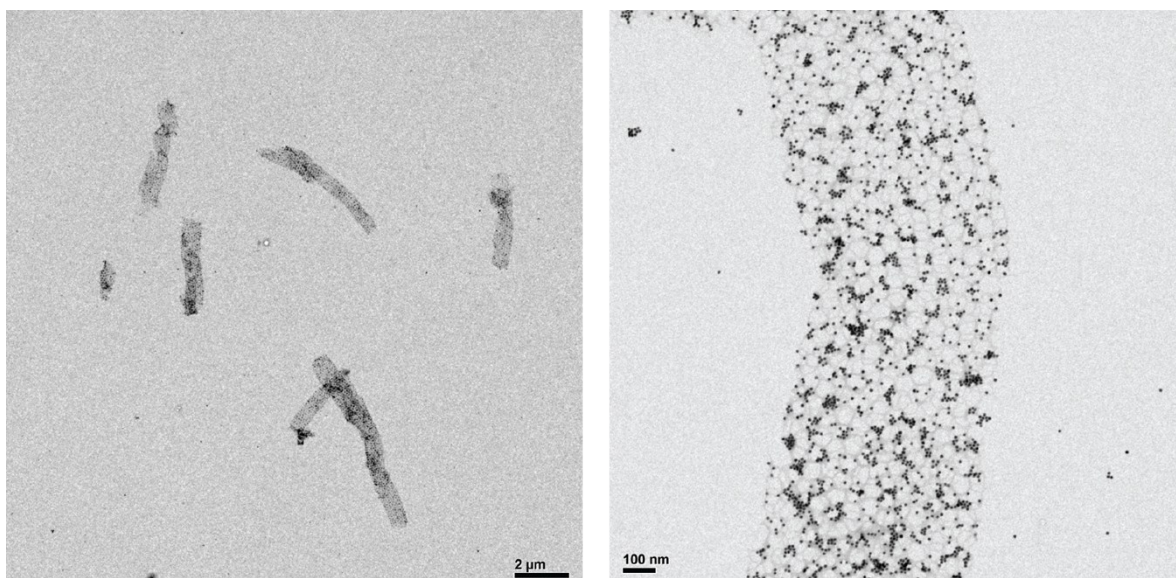


Figure S10. AuNP lattices on honeycomb tube with type-1 configuration.

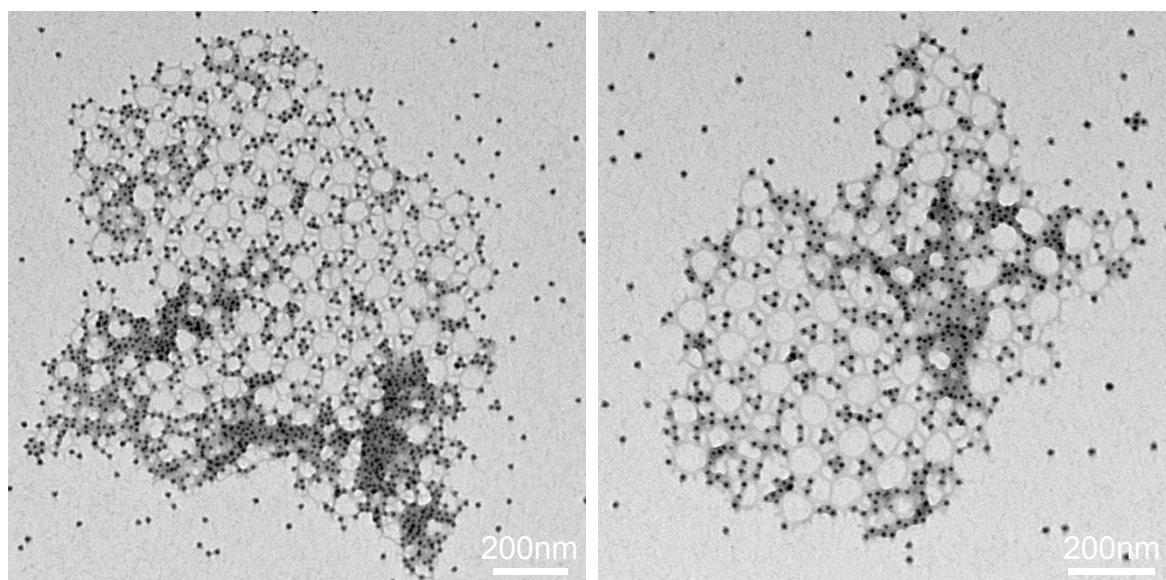


Figure S11. AuNP lattices on honeycomb array with type-2 configuration.

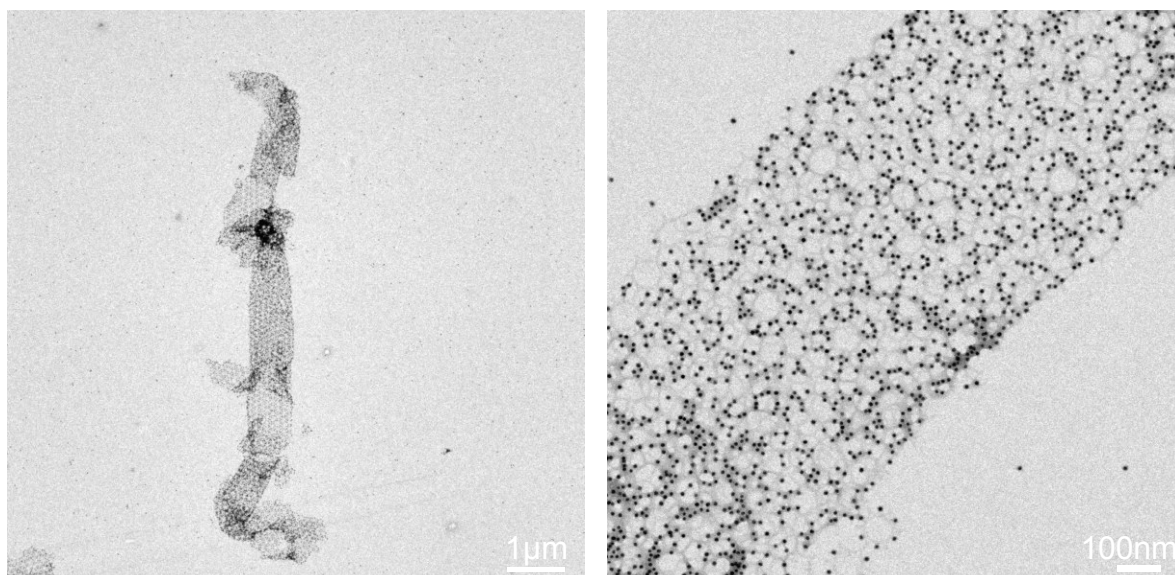


Figure S12. AuNP lattices on honeycomb tube with type-2 configuration.

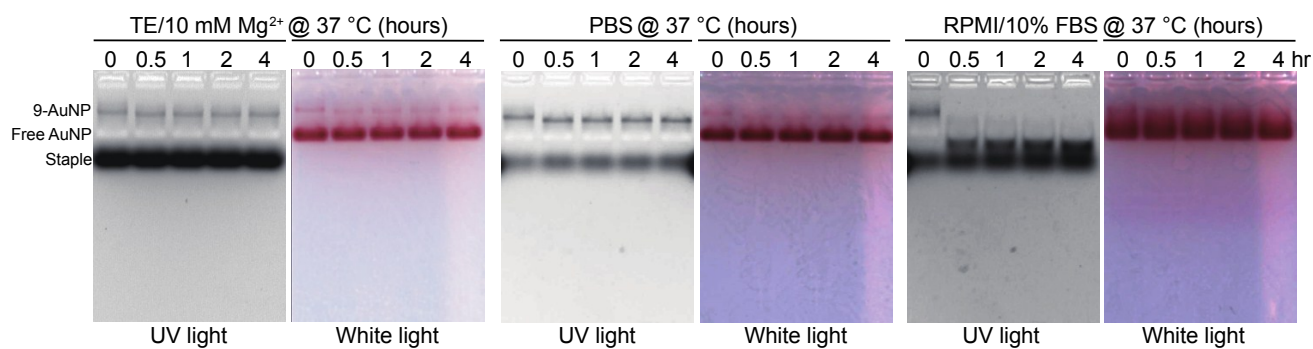


Figure S13. Stability of 9-AuNP clusters in various buffers and cell culture medium at 37 °C.

DNA strands sequences

p7560 scaffold DNA:

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Thiolated DNA strands:

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7[56]	GATAATAATTCCTGATAGCATCACCTTGCTGAAC	core
7[84]	TATTAGACTTTAAAGAAACCGTTATTAATCAGATGA	core
7[127]	AATAAGAGAATACCGGAATCATAAAATTTTCC	core
7[162]	GTAATTCCATTCCAAGCAAATTCTTACCAGTATA	core
7[190]	AAACAACATGTTAGAAACCAAGTCCTGAAGCGAACCT	core
7[233]	CCGTCACCGACAAAAGAAACGCAAAACCCCTG	core
7[268]	CAGCAAACCTTATTAGCGAAAATTCATATGGTTTA	core
7[296]	ACCATTAGCAAGGCGCGTTTAAATCAGTCAGCATTG	core
7[339]	CCAAAAAAGTAGCGTAACGATCCTCCTCAA	core

7[374]	TATCGGTCTCAGCAGCTTTCTGTATGGGATTTTG	core
7[402]	GGTGAATTTCTTGGGAGTTAACCATCGCATACACTA	core
7[445]	TAAATATTCATGAATTACGAGGCAAAATCAAC	core
7[480]	ACAGTTCGGAAGCAAACGACGATAAAAAACCAAAA	core
7[508]	TAAATCAAAAAATGCGTTTAAATTGCATCAAGTTTCA	core
7[551]	AATTCGCGTCTCAATCATATGTACTACTTTTG	core
7[586]	TCAACATGATCGCACTTTGTATAAGCAAATATTT	core
7[614]	AACCCGTCGGATTTTGAGGGGGTCACGTAGGCGATT	core
8[71]	CCTACCATAAAACAATTGAATACCAAGTTAC	core
8[106]	CTTAGAATCCTGGAACAGTACATGAGCCAGT	core
8[177]	CCGCGCCCAACGATTACAATTTTATCCTG	core
8[212]	AACAAAGTCAGAATAGCAGCCTTTAATTATCA	core
8[283]	CCTCAGAACCAGTGCCTAAAGCCAGAATGG	core
8[318]	GAGAAGGATTACAGTTAATGCCCCGAAAATCT	core
8[389]	TTCCATTAATTTGAAATGTATCATCGCCTG	core
8[424]	GTAACAAAGCTACCAGGCGCATAGAATCGTCA	core
8[495]	AGCTTAATTAATCATACATTCGCAAATGG	core
8[530]	CGGGAGAAGCCAATTAAGCAATAATCAAAAAAT	core
8[601]	TGTTGGGAACGCCAACTTTCTCAGGAGAA	core
8[636]	AATATAGGGGCACGCTCGCCCTGGTCTTTAGG	core
9[40]	ATATACAGCGCAGAGGGAAAACAA	core
9[64]	CGGGAGAAACAATAACGGATTTCGCTTAGTGGAAGGGTTAGAA	core
9[107]	TATTAATTTTACTAGAAAAAGTAAATTTAATG	core
9[146]	CCCGACTTCCAACGCTATTTATCC	core
9[170]	TTAAATCAAGATTAGTTGCTATTGGATGACGTAGGAATCATTA	core
9[213]	TTAACTGAAGACACCACGGAATCCTTATTACG	core
9[252]	ACAGGAGGAGTCTCTGTGGTAATA	core
9[276]	TTGGCCTTGATATTCACAAACAACCTGGGGCTCAGAGCCGCCAC	core
9[319]	GGCTGAGATAAAAGTTTTGTCGGAGTTTCGTCA	core
9[358]	AAACACTCGTGTCTGAATAAGGGAA	core
9[382]	TTATACCAAGCGCGAAACAAAGTATCAGTTTTTCATGAGGAAGT	core
9[425]	CATTACCCTAGTAAGAGCAACTTCATCAGTTG	core
9[464]	TTCCATATCCTGTTTAGTAGTAGC	core
9[488]	CTGCGAACGAGTAGATTTAGTTTGTAACCTGCGGATGGCTTAG	core
9[531]	CCCTGTAACCCGGTTGATAATTGCCTGAGAGT	core
9[570]	AAGTTGGGTGGATGTTTGTCAACC	core
9[594]	AGTCACGACGTTGTAAAACGACGCGGGGCTCAGGCTGCGCAAC	core
9[637]	CTCGTCGGGTGCCACGCTGAGAAAAATACCGA	core
10[55]	AAAATCGTAACAGTACTTTGCACGTGAGCGGAAT	core
10[95]	TGAGTGAATAACCTGATTGCTTTTCATTTG	core
10[161]	AATCTTAGCGGGAGGTAAATCAGATTGGGTGTC	core
10[201]	CATAAAAAACAGTTGCACCCAGCTTTTGTGTT	core
10[267]	AAAGCGCTTGAGGCAGCAGAGCCACATTTTCGGT	core
10[307]	AACCTATTATTATAAATCCTCATTTGAGTA	core
10[373]	ATAAATTATCTTTGACATACGTAATTTTTCGGG	core

10[413]	CATCAAGAGTAACAACGGAGATTGAGGACA	core
10[479]	TCAATAAAACAGTTGAAATGCTGTATTCAAAGCG	core
10[519]	AAAGCTAAATCGACCATTAGATACAGGCAA	core
10[585]	GCCAGGGTAACGCCAGGTGCGGGCCCAGTATCGG	core
10[625]	TACCGACAGTGGCCAGTGCCAAGATAACCC	core
11[48]	AATTAATTACATTTTCAAAATTACTTTTACAT	core
11[80]	AATTACCTTTTTTAATTGAAAACATGCTTCTGTGAATTTA	core
11[154]	CAATCCAAATAAGAATAGCAAGCTTTGAAGCC	core
11[186]	TAACGTCAAAAATGAAAGGGTAATAGCGCATTAAAGCCCA	core
11[260]	AGTTTTAACGGGGTCGCCACCCTGTCAGACGA	core
11[292]	ACAGTGCCCGTATAAAGGATTAGCAAACATGAGGGTTGAT	core
11[366]	CCGAACTGACCAACACGGGTAAACCCAGCGA	core
11[398]	GATGAACGGTGTACAGGCTCATTCTTGACAAGTAGTAAAT	core
11[472]	ATTAACATCCAATAGCTGAATATTTCCCAATT	core
11[504]	GGCAAAGAATTAGCAATTTATTTCTGTACCAAAATGCAAT	core
11[578]	TTATGACAATGTCCGGGCGATCGGGTTTCCC	core
11[610]	CGCTTCTAATCTATTTCTTGAATCCCCTGCCACCTCCTGG	core
3[88]	TAGCTTAGGATAGAACGCAAGACA AAAAAAAAAAAAAAAAAAAAA	Site1 handle
3[104]	GCTGATACCGACCGTGTGATAAATGGCGAAAAATATATTT AAAAAAAAAAAAAAAAAAAA	Site1 handle
3[194]	CTAATATCGTGTGTAGAAAGGAA AAAAAAAAAAAAAAAAAAAAA	Site2 handle
3[210]	AACCCTAGCAAACGTAGAAAATACTAAATCAAAATACCCA AAAAAAAAAAAAAAAAAAAA	Site2 handle
3[300]	TTGCTCAGTTTGCCCGAGAGCCAC AAAAAAAAAAAAAAAAAAAAA	Site3 handle
3[316]	GGATAAACTACAACGCCTGTAGCACTGAGAGAGCCCAATA AAAAAAAAAAAAAAAAAAAA	Site3 handle
3[406]	TAAGGCTTGGTGGTTTGGGAAGAA AAAAAAAAAAAAAAAAAAAAA	Site4 handle
3[422]	GAGAAGAATACCACATTCAACTAACGGGGAGAAACGGAAC AAAAAAAAAAAAAAAAAAAA	Site4 handle
3[512]	AGGATAAATGCCCGCTTGATAAAT AAAAAAAAAAAAAAAAAAAAA	Site5 handle
3[528]	GAACCAACAAGAGAATCGATGAACGAGCTAACTTGAGAGA AAAAAAAAAAAAAAAAAAAA	Site5 handle
1[626]	TCATAGCTGTTTCTGTGTAATGCGCGAACTG AAAAAAAAAAAAAAAAAAAAA	Site6 handle
3[618]	CGCATTTCATTCACAATCATGG AAAAAAAAAAAAAAAAAAAAA	Site6 handle
4[145]	GAATAAACATAAAGTAACATGTAA AAAAAAAAAAAAAAAAAAAAA	Site7 handle
6[137]	TTTAGGCAGAGGCATTTCAAATCAATATATG AAAAAAAAAAAAAAAAAAAAA	Site7 handle
4[251]	CAACATATTTGAGCCAATTGACGG AAAAAAAAAAAAAAAAAAAAA	Site8 handle
6[243]	AAATTATTCATTAAAGGTGACAGAGAGAATAA AAAAAAAAAAAAAAAAAAAAA	Site8 handle
4[357]	CTCATAGTGCTCCAAAAATTGCGA AAAAAAAAAAAAAAAAAAAAA	Site9 handle
6[349]	ATAATAATTTTTTCACGTTCTGCCTATTTCTCGG AAAAAAAAAAAAAAAAAAAAA	Site9 handle
4[463]	GCCAAAAGTGAATCCCTAGACTGG AAAAAAAAAAAAAAAAAAAAA	Site10 handle
6[455]	ATAGCGTCCAATACTGCGGGCTGGCTGACCTT AAAAAAAAAAAAAAAAAAAAA	Site10 handle
4[569]	TAGCATGTGGCCTTCCCTCATT TT AAAAAAAAAAAAAAAAAAAAA	Site11 handle
6[561]	TTAACCAATAGGAACGCCAAGCCTCAGAGCAT AAAAAAAAAAAAAAAAAAAAA	Site11 handle
4[39]	GTATTAACCTAATAGAAGGAATTG AAAAAAAAAAAAAAAAAAAAA	Site12 handle
6[31]	AGGAAGGTTATCTAAAATAAGTGACTCTATGA AAAAAAAAAAAAAAAAAAAAA	Site12 handle
4[109]	CATTTTGCGGAACACAAACAATTCGACATGGC	Hexamer connector
4[321]	AGCGTCAGACTGTAGCCGAAACGTCAAAAAG	Hexamer connector

5[110]	GTAACCCAGAGCCGCCGACGACAGAATCAA	Hexamer connector
5[322]	GTTTGTTTCAGGTTTAACGTTTTAAAAAGTTTGA	Hexamer connector
8[39]	AATAAAGAAACATCAACGAATTATTCATTTCATTAC	Hexamer connector
8[251]	GAGCCGCCGAGTGTACAATTTACCGTTCCAGTAAGCGT	Hexamer connector
10[17]	TGAGCCCAATGAAACCATCGATAGCAGCAAAAAGGGCGACAGTAGCACCATT	Hexamer connector
10[229]	CATACAACCTCGTATTAAATCCTTTGCCCGCCTCAATCAATAGGATTTAGAAG	Hexamer connector
11[18]	AAGATGATGAAACAAATTGCGTAGATTCTTTT	Hexamer connector
11[230]	TTTTGATGATACAGACCAGAACCACCAATTAT	Hexamer connector
4[111]	TATCATTTTGCAGAACACAAACAATTCGACC	2D connector
4[217]	CTAATTTACGAGCATGTCAGCTAATGCAGAG	2D connector
4[323]	TTTAGCGTCAGACTGTAGCCGAAACGTCAA	2D connector
4[429]	GGTCGCTGAGGCTTGCAAAACAGCTTGATAA	2D connector
4[535]	GAAAGACTTCAAATATCCAGGTCTTTACCCG	2D connector
4[641]	AACCGTGCATCTGCCAGTCTCCGTGGGAACA	2D connector
5[113]	CAAGAGGCAAAAGACCACGCATAACCGATAT	2D connector
5[219]	AGTACGGTGTCTGAAAAAGATTAAGAGGAA	2D connector
5[325]	GGGGATGTGCTGCATGGTGTAGATGGGCGCA	2D connector
5[431]	TTTCAGGTTTAACGTTTTAAAAAGTTTGAGTA	2D connector
5[537]	CGCGAGGCGTTTTACAAGAAAAATAATATCC	2D connector
5[643]	CCCAGAGCCGCCGACGACAGAATCAAGTT	2D connector
8[39]	AATAAAGAAACATCAACGAATTATTCATTTCATTACCTGA	2D connector
8[145]	CTTATCCGGCCATATTAACGAGCGCTTTCCAGAGCCTAAT	2D connector
8[251]	GAGCCGCCGAGTGTACAATTTACCGTTCCAGTAAGCGTCAT	2D connector
8[357]	GAAGGCACGTCAATCAATCCGCGACCTGCTCCATGTTACTT	2D connector
8[463]	TGTTTTAATACTAATAGCTATATTTTCATTTGGGGCGCGAG	2D connector
8[569]	ATTACGCTGCTGAATCTTCTAAGTGTTGTGAATTCATGC	2D connector
10[14]	CCCGATAGTTGCCCGACAATGACAACAGTTTCAGCGCTTGCTTTTCA	2D connector
10[120]	TTGACTATTATAGTCAGAAGCAAAGTGCAAAAGAAGTGAGAATGACCA	2D connector
10[226]	CAAACGGCGGATTGACCGTAATGGGAATATTTTGTTAGAGCGAGTAAC	2D connector
10[332]	GAACCTCGTATTAAATCCTTTGCCCGCCTCAATCAATAGGATTTAGAAG	2D connector
10[438]	TACGCGCTGTTTATCAACAATAGACAGTAGGGCTTAACGACGACAAT	2D connector
10[544]	CCCAATGAAACCATCGATAGCAGCAAAAAGGGCGACAGTAGCACCATT	2D connector
11[16]	AAGAAGATGATGAAACAAATTGCGTAGATT	2D connector
11[122]	CAGTTACAAAATAAACAGTATTCTAAGAACC	2D connector
11[228]	GGCTTTTGATGATACAGACCAGAACCACCAG	2D connector
11[334]	GGAACGAGGCGCAGACGCAACCTAAAACGAA	2D connector
11[440]	AAAGGTGGCATCAATTCATATGCAACTAAAT	2D connector
11[546]	GACTTAAGTGTCTTAGAGCTGGCGAAAGGC	2D connector
4[112]	TTATCATTTTGCAGAACACAAACAATTCGACC	Tube connector
4[218]	CCTAATTTACGAGCATGTCAGCTAATGCAGAG	Tube connector
4[324]	CTTAGCGTCAGACTGTAGCCGAAACGTCAA	Tube connector
4[430]	CGGTCGCTGAGGCTTGCAAAACAGCTTGATAA	Tube connector
4[536]	CGAAAGACTTCAAATATCCAGGTCTTTACCCG	Tube connector

4[642]	TAACCGTGCATCTGCCAGTCTCCGTGGGAACA	Tube connector
5[113]	CAAGAGGCAAAAAGACCACGCATAACCGATATA	Tube connector
5[219]	AGTACGGTGTCTGGAAAAAGATTAAGAGGAAG	Tube connector
5[325]	GGGGATGTGCTGCATGGTGTAGATGGGCGCAT	Tube connector
5[431]	TTTCAGGTTTAACGTTTTAAAAAGTTTGAGTAA	Tube connector
5[537]	CGCGAGGCGTTTTACAAGAAAAATAATATCCC	Tube connector
5[643]	CCCAGAGCCGCCGACGACAGAATCAAGTTT	Tube connector
8[39]	AATAAAGAAACATCAACGAATTATTCATTTCATTACCTGAG	Tube connector
8[145]	CTTATCCGGCCATATTAACGAGCGTCTTTCAGAGCCTAATT	Tube connector
8[251]	GAGCCGCCGAGTGTACAATTTACCGTTCCAGTAAGCGTCATA	Tube connector
8[357]	GAAGGCACGTCAATCAATCCGCGACCTGCTCCATGTTACTTA	Tube connector
8[463]	TGTTTTAATACTAATAGCTATATTTTCATTTGGGGCGCGAGC	Tube connector
8[569]	ATTACGCCTGCTGAATCTTCTAAGTGGTTGTGAATTCATGCG	Tube connector
10[14]	CCCGATAGTTGCGCCGACAATGACAACAGTTTCAGCGCTTGCTTTCGA	Tube connector
10[120]	TTGACTATTATAGTCAGAAGCAAAGTGCAAAAGAAGTGAGAATGACCA	Tube connector
10[226]	CAAACGGCGGATTGACCGTAATGGGAATATTTTGTTAGAGCGAGTAAC	Tube connector
10[332]	GAACCTCGTATTAAATCCTTTGCCCGCCTCAATCAATAGGATTTAGAAG	Tube connector
10[438]	TACGCGCTGTTTATCAACAATAGACAGTAGGGCTTAACGACGACAAT	Tube connector
10[544]	CCCAATGAAACCATCGATAGCAGCAAAAAGGGCGACAGTAGCACCATT	Tube connector
11[15]	AAAGAAGATGATGAAACAAATTGCGTAGATTT	Tube connector
11[121]	CCAGTTACAAAATAAACAGTATTCTAAGAACC	Tube connector
11[227]	TGGCTTTTGATGATACAGACCAGAACCACCAG	Tube connector
11[333]	CGGAACGAGGCGCAGACGCAACCTAAAACGAA	Tube connector
11[439]	AAAAGGTGGCATCAATTCATATGCAACTAAAT	Tube connector
11[545]	CGACTTAAGTGCCTTAGAGCTGGCGAAAGGC	Tube connector

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