Nanocrystals for the fabrication of new functional mesostructured materials

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Outline

Introduction

Chemical synthesis of nanoparticles

Self-organization of nanoparticles at 2D and 3D

Towards new properties:

Conclusions

Introduction

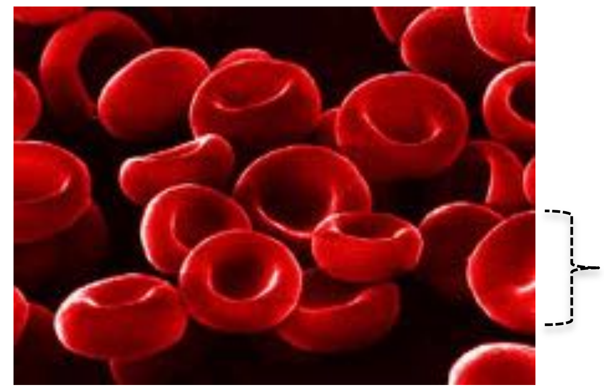
Nanoparticles?

Nanoparticles are particles (typically crystals of inorganic elements) for which the largest characteristic dimension is around 1-100 nm ($1nm=10^{-9}$ m= 0,000000001 m)

Nanocrystals correspond to well crystallized Nanoparticles

~ 100 µm=0,1 nm

Human hair

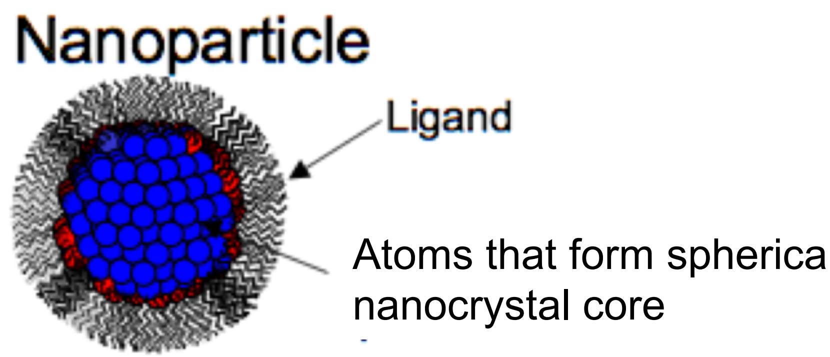


10 µm

Red blood cell

They are sticky little things that adhere to anything (including each other)

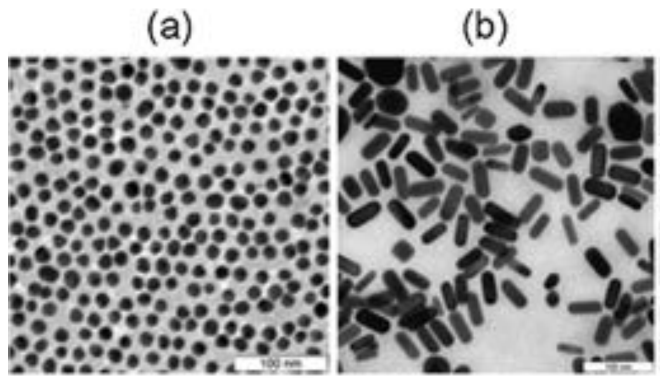
Remedy: coat them with ligands



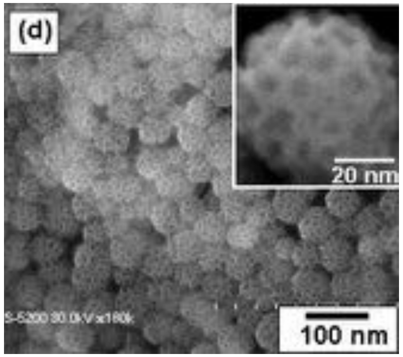
Classification of nanoparticles?

Nanoparticles can be classified into different classes based on their properties, shapes or sizes. The different groups include fullerenes, metal NPs, ceramic NPs, and polymeric NPs..

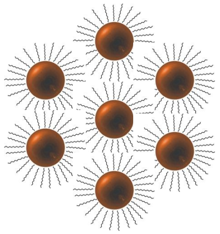
Fullerene= NPs



Au NPs



Mesoporous silica NPs



Self-organization of nanoparticles ?

NP organization correspond to a process where components of the system acquire non-random spatial distribution with respect to each other and the boundaries of the system (N.A. Kotov)

The NP organization can be due to direct specific interaction, collective effects, and/or occur indirectly through their environment

Due to the proliferation of nanoparticle synthesis techniques, the study and design of nanoparticle self-assembly has become widespread

Kotov, N.A. Europhysics Letters. **119** (6): 66008.

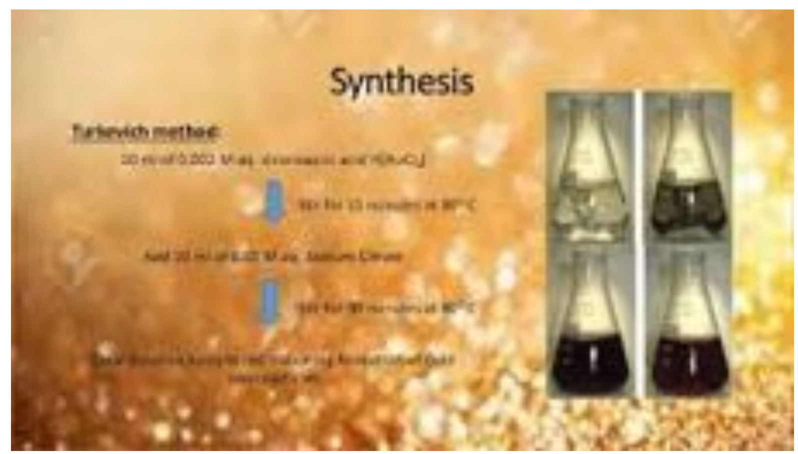
Chemical synthesis of Nanoparticles

Nanoparticles synthesis

Faraday solutions exposed in his laboratory

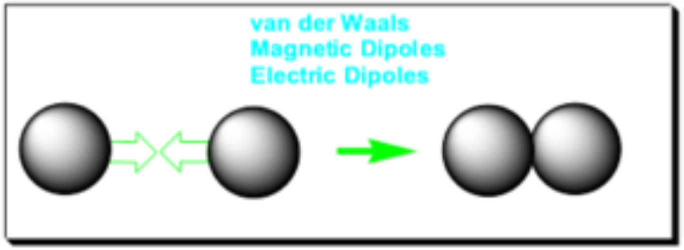
1857 **first controlled synthesis** of gold colloids from the reduction of $AuCl_4^-$ by phosphorous solubilized in carbon disulfide (CS_2) by Faraday
The color of the colloidal solution is appeared to be related to the particle size.

1951 Turkevitch introduces the most popular synthesis of Au NPs by using citrate reduction of Au(III) from $HAuCl_4$ salt in water.

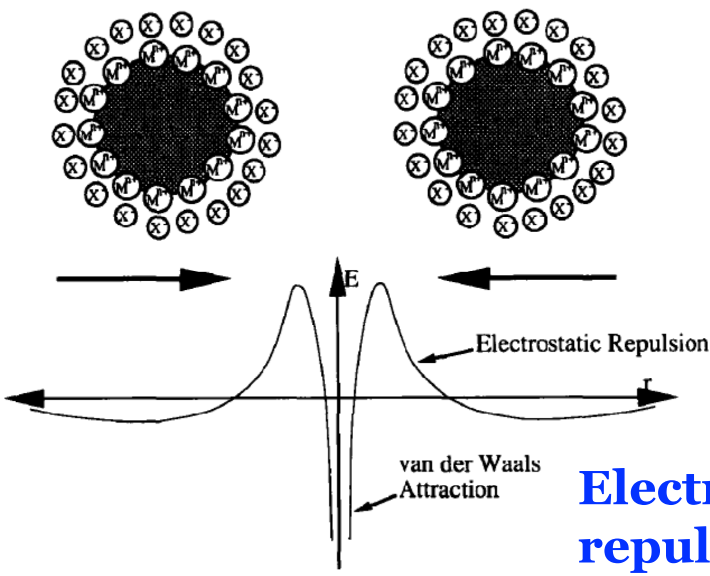


M. Faraday, Philos.trans.R.Soc.London, 1857, 147, 145

Nanoparticle stabilization



- by **placing them in an inert environment** → an inorganic matrix or → polymer
- by **adding surface-protecting reagents** → organic ligands – figure 9 → inorganic capping materials – figure 8



Electrostatic repulsion

Figure 8. Electrostatic stabilization of metal colloid particles. Attractive van der Waals forces are outweighed by repulsive electrostatic forces between adsorbed ions and associated counterions at moderate interparticle separation [Gunter Schmid - **Clusters and Colloids,** VCH, 1994]

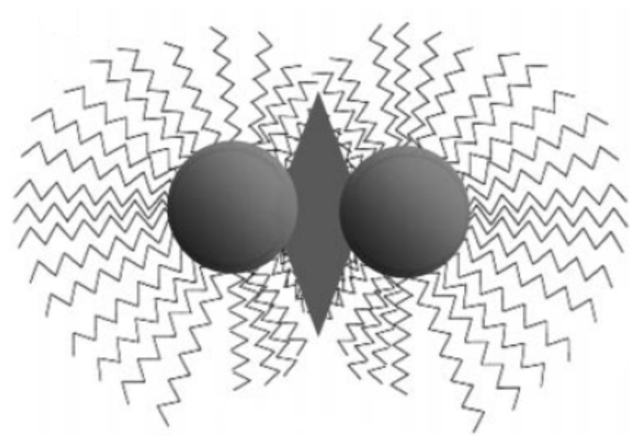
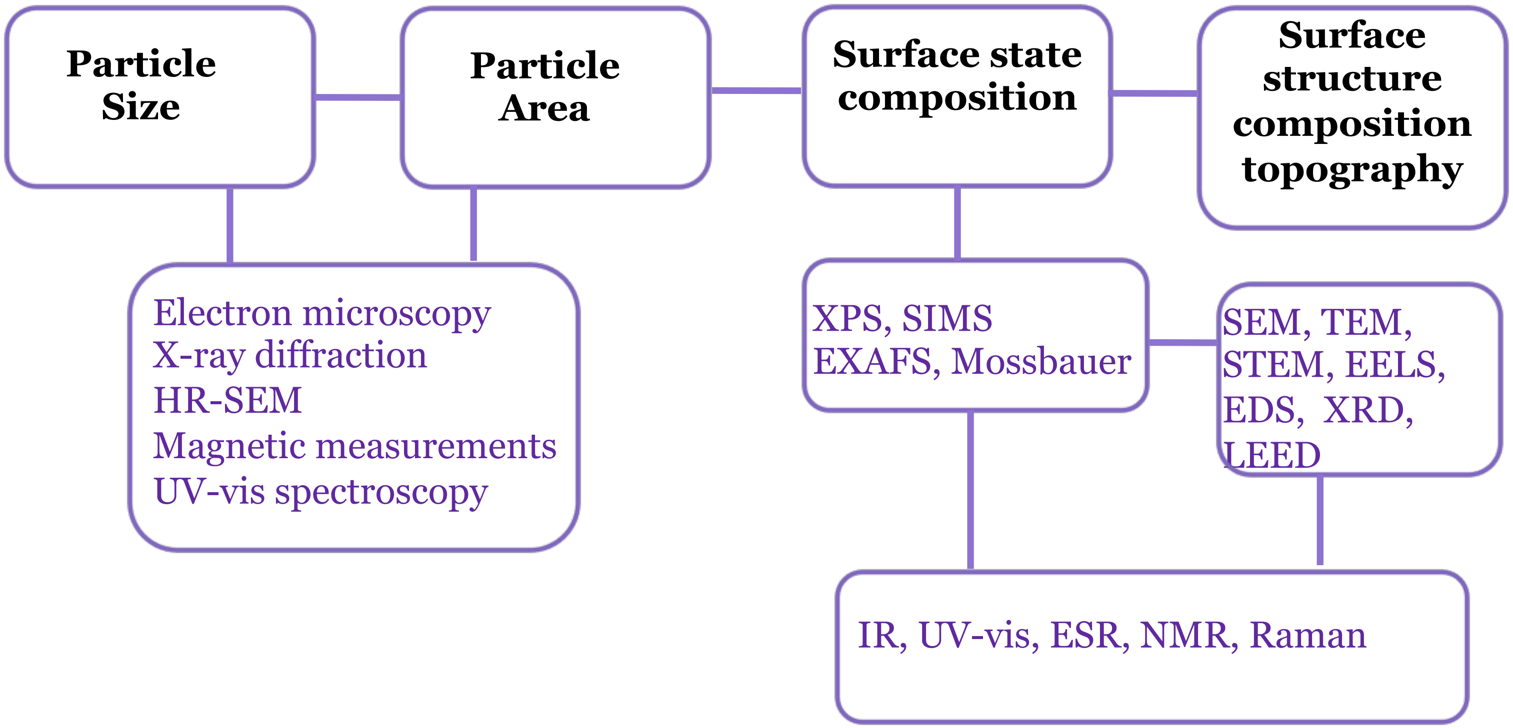


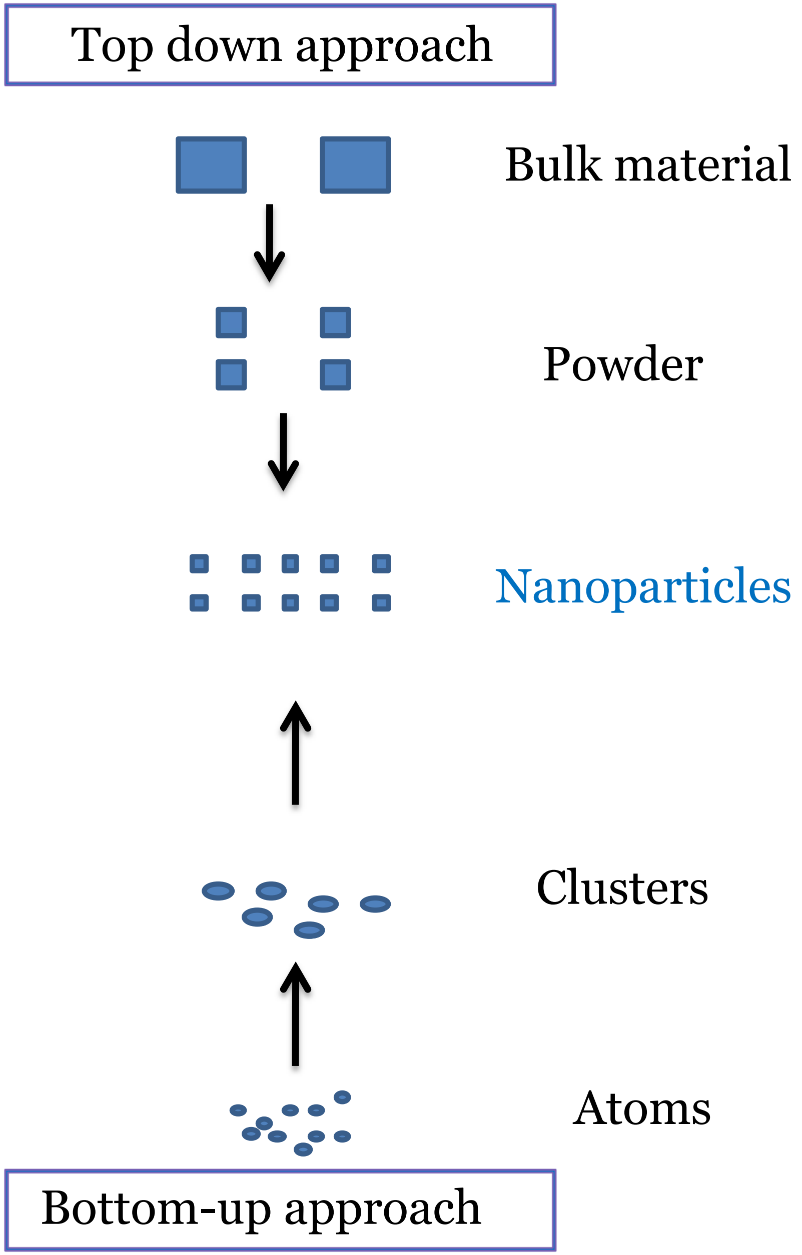
Figure 9 . Steric stabilization of nanostructured metal colloids [Helmut Boennemann – Eur.J.Inorg.Chem.2001]

Steric repulsion 16

Nanoparticle Characterization



Nanoparticle synthesis:



Nanoparticle synthesis:

Bottom-up approach

Physical methods:
High production of nanoparticles
drastic experimental conditions, ultra-high vacuum
Limited concerning the control of NP size and shape

Chemical methods: well control of the size, shape and composition of the nanoparticles
A large number of parameters can be adjusted (Temperature, Concentration and nature of reactants etc)

Formation Mechanism of metallic nanoparticles in solution: Lamer Diagram

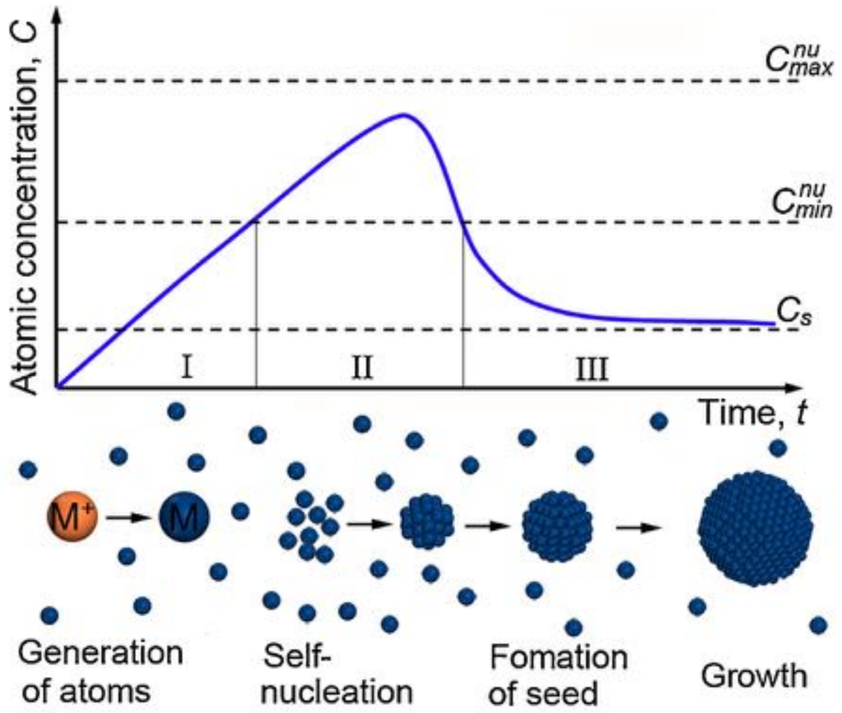
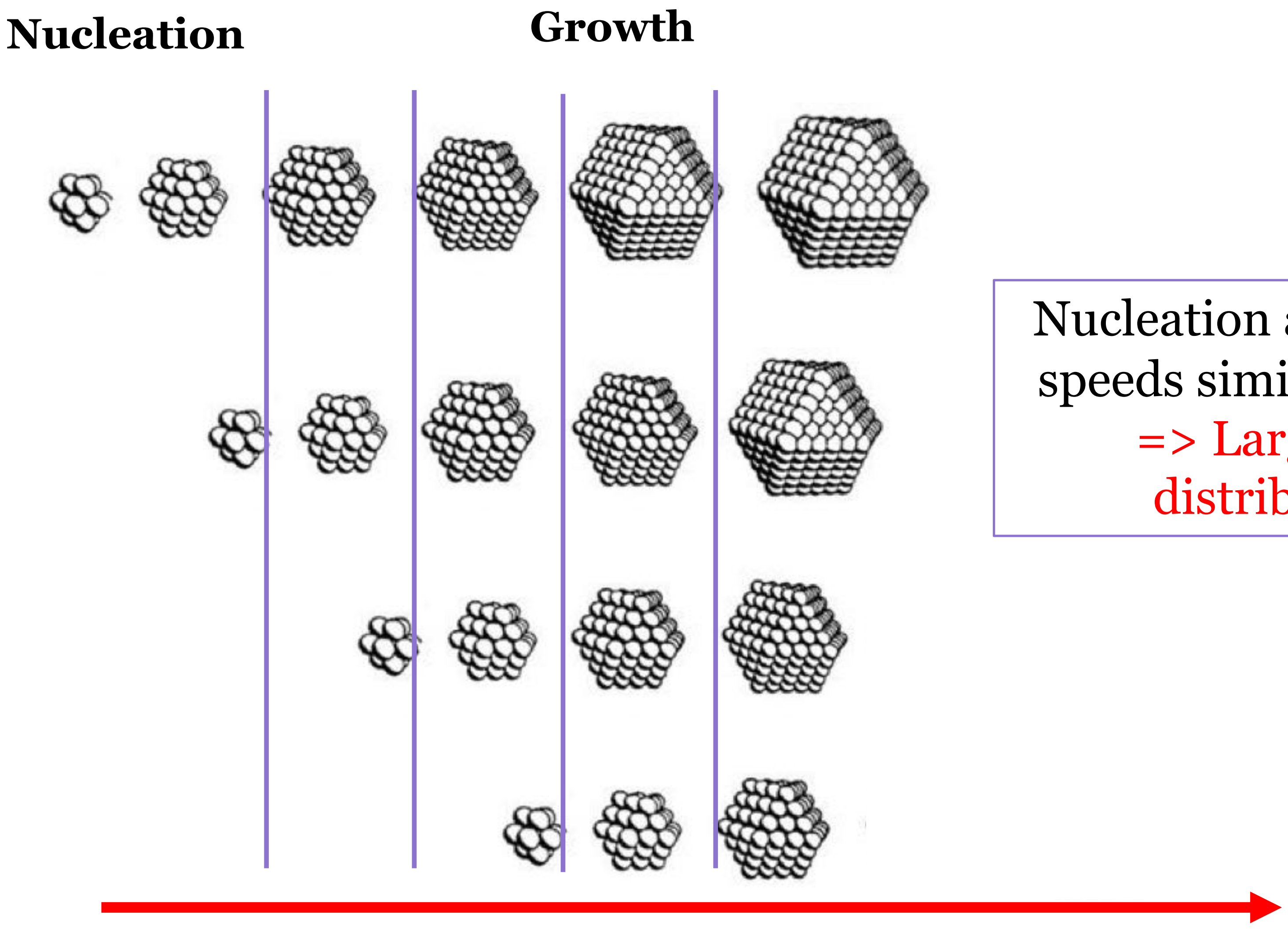


Figure 1 LaMer curve describing three stages of metal nanocrystal formation in solution system. Stage I: atom producing, stage II: nucleation, and stage III: seed formation and growth.

Preparing monodisperse NPs require a single temporally short nucleation event followed by slower growth on the existing nuclei

Schematic illustration of the nucleation and growth process of nanocrystals in solution

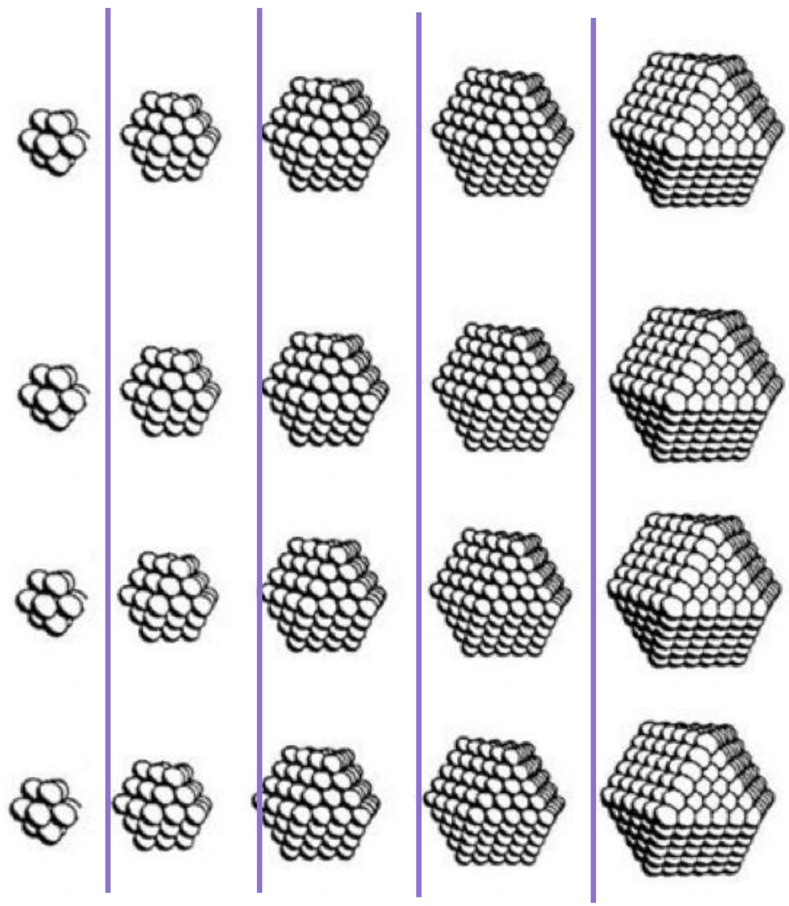
Chemical synthesis of nanoparticles



Nucleation and growth speeds similar: $V_n \approx V_g$
=> Large size distribution

Chemical synthesis of nanoparticles

Nucleation **Growth**



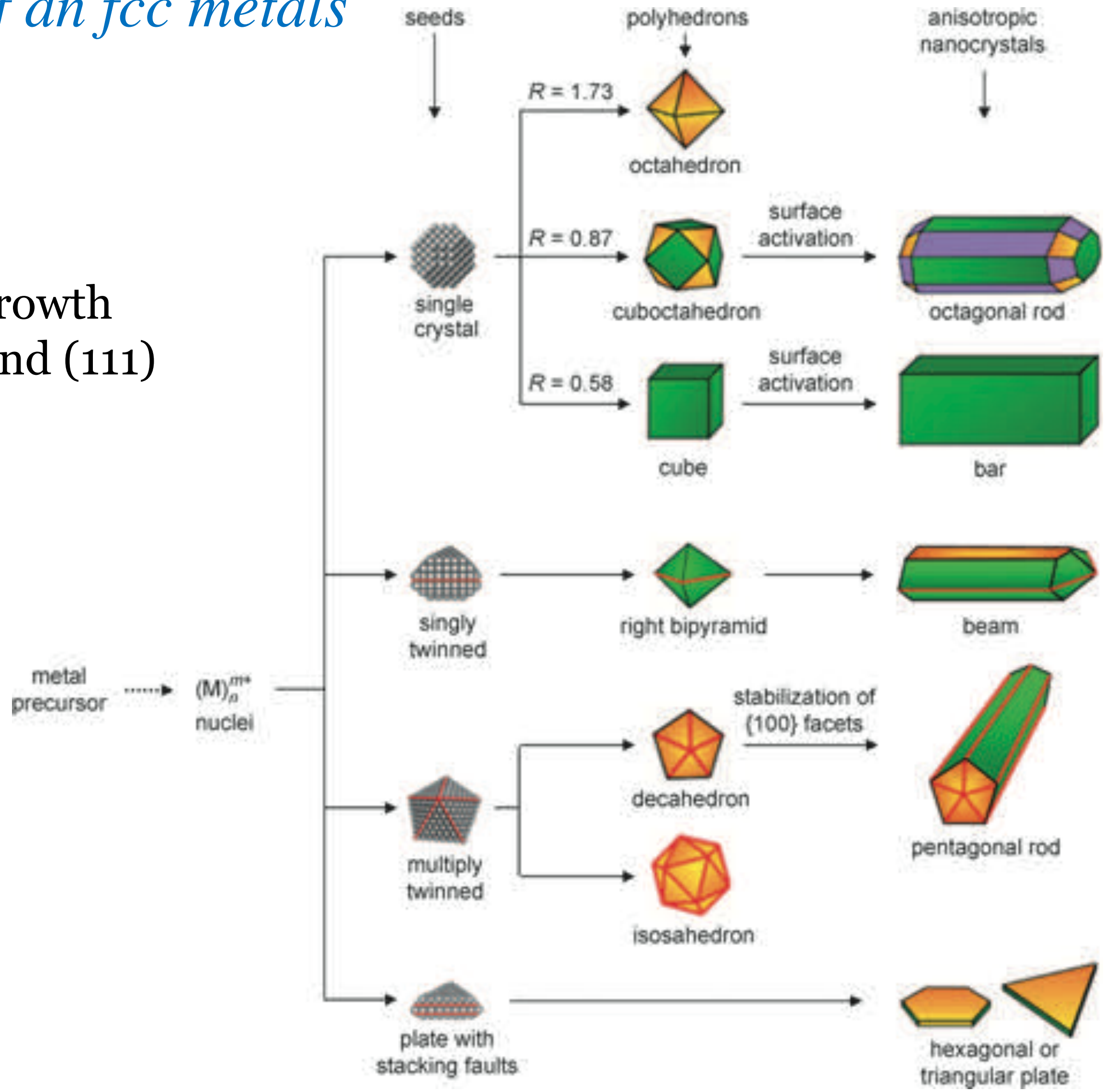
$V_n \gg > V_g$

=> Narrow size distribution

Time

Correlation between different types of seeds and the final nanocrystals shape of an fcc metals

R=ratio between the growth rates along the (100) and (111) directions



Main Chemical Methods for NPs synthesis

Microemulsion technique

NPs: Au, Ag, Cu, Ni, Pt, Ag2S, CuS, CdS etc

Liquid-liquid phase transfer

NPs: Au, Ag, Pt, Pd, CoPt…

Metallic/ organometallic salt reduction

NPs: Au, Ag, Cu

Sol-gel synthesis

NPs : ZnO, SnO2, Fe_2O3, TIO_2, SIO_2…

Polyol synthesis

NPs: Cu, Ag, Pt,Pd, CoPt, ZnO, Cu_2O…

Chemical vapor synthesis

NPs: CoO, SiO2, ZnO, Fe2O3…

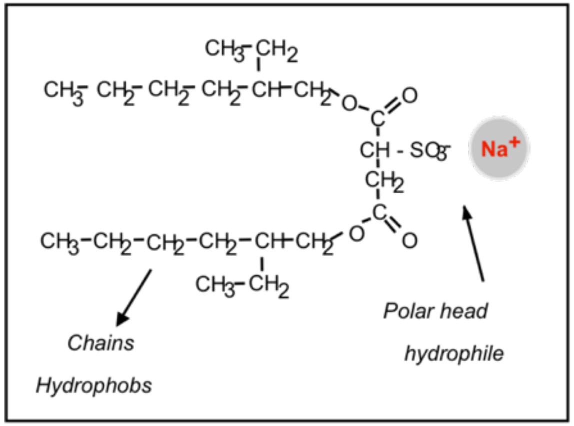
Organometallic decomposition

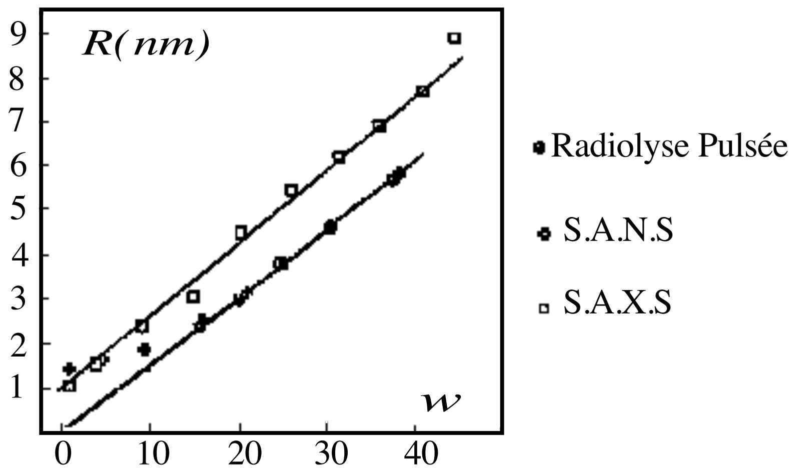
NPs: Co, Pt, Ag…

Microemulsion technique

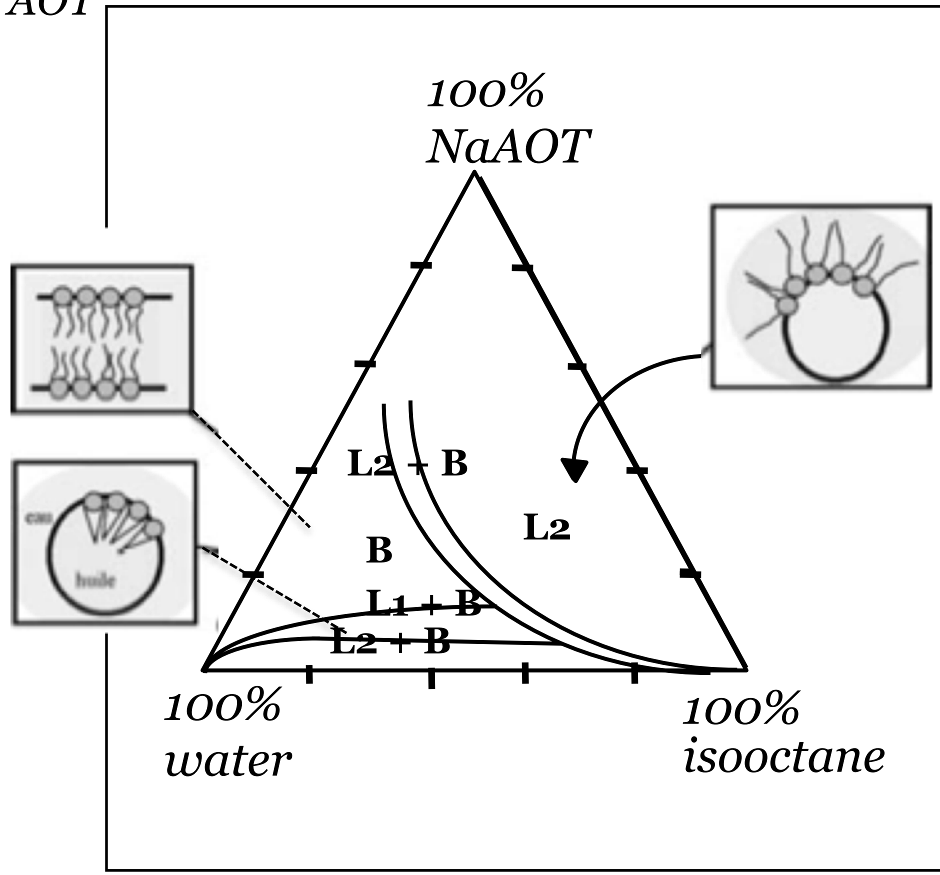
Molecular structure of Na AOT

di (ethyl hexyl) sulfosuccinate of sodium, Na AOT





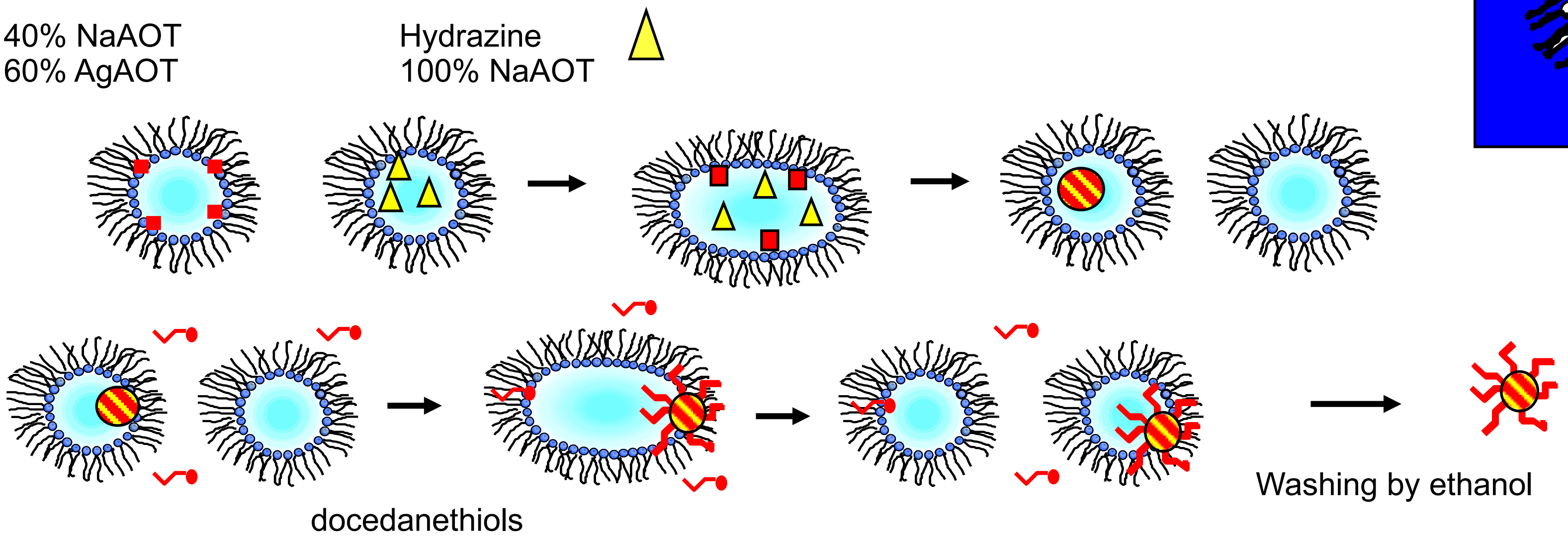
Phase diagram of the system AOT/water/isooctane



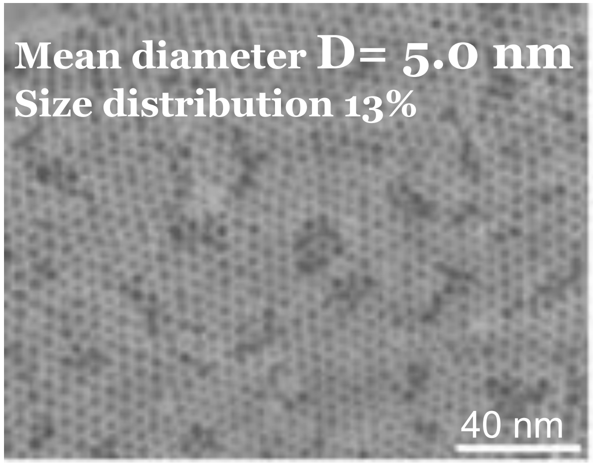
Water content : **W = [H_2O]/[AOT]**

$$R\ (nm) = 0{,}15\ W$$

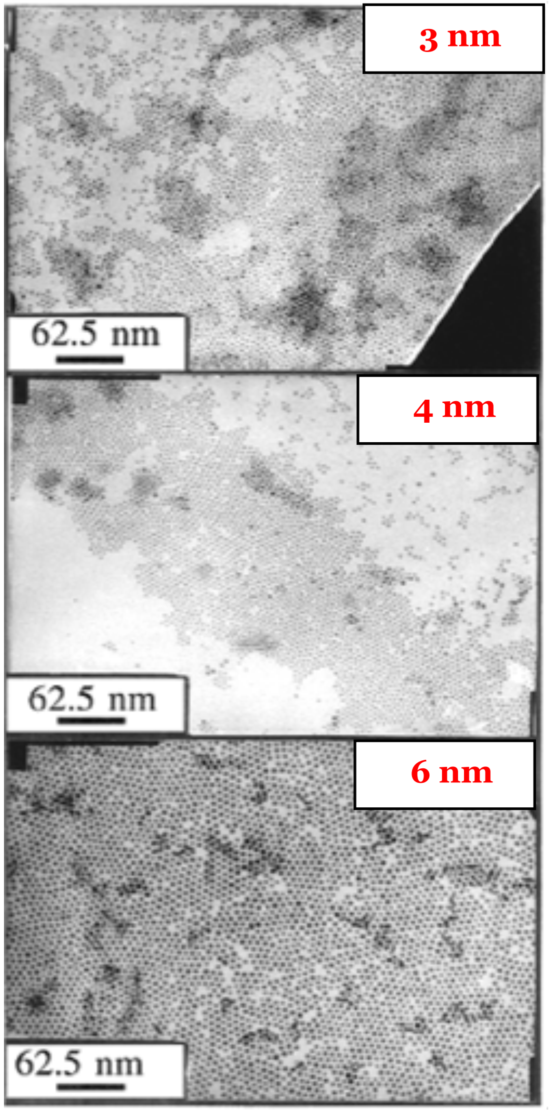
Synthesis of silver nanocrystals in reverse micelles



- ✿ After adsorption of the ligands the nanocrystals can be collected as powder or dispersed in organic solvent
- ✿These hybrid organic/inorganic system are stables in time.

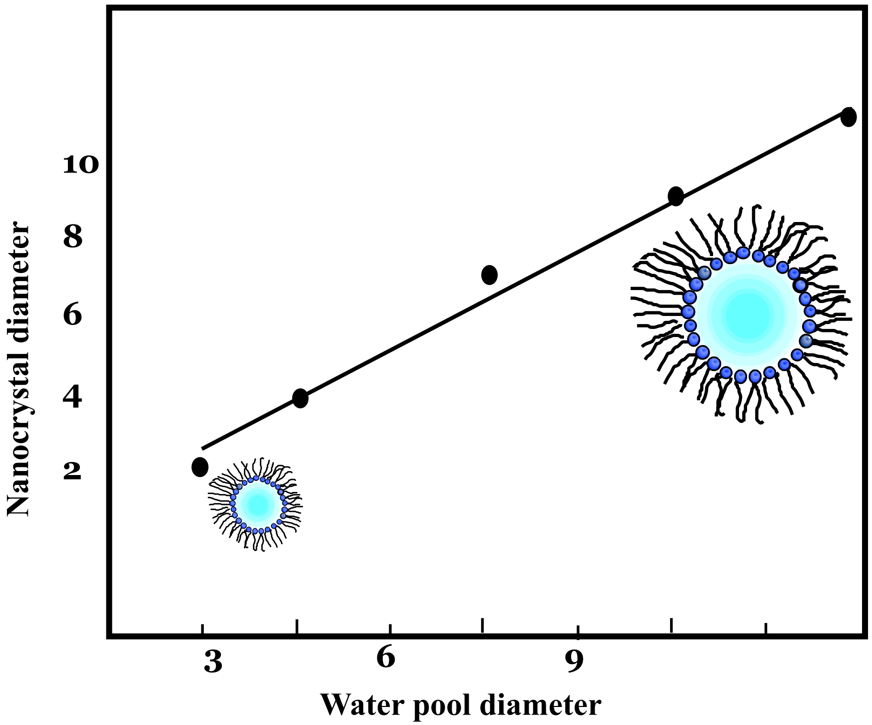


A. Taleb ; C. Petit; M.P. Pileni, Chem Mater. 9, 950-959; (1997) A. Courty, I. Lisiecki and M.P. Pileni, J.Chem.Phys, 116, 8074 (2002)



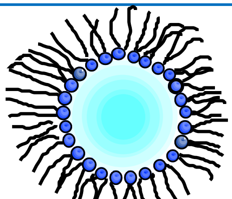
Ag$_2$S NPs

Size control of nanoparticles via the micelle size



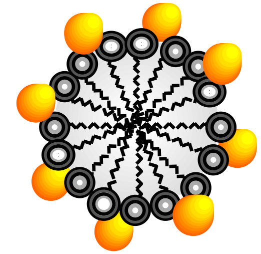
Water content : **W = [H_2O]/[AOT]**

Various nanocrystals made by microemulsion technique

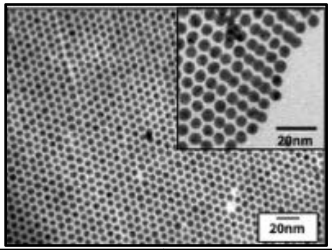


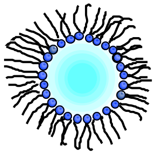
Reverse Micelles

Normal Micelles

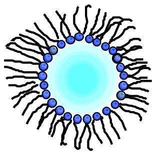


Gold
4nm<D<6nm

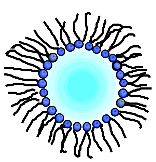




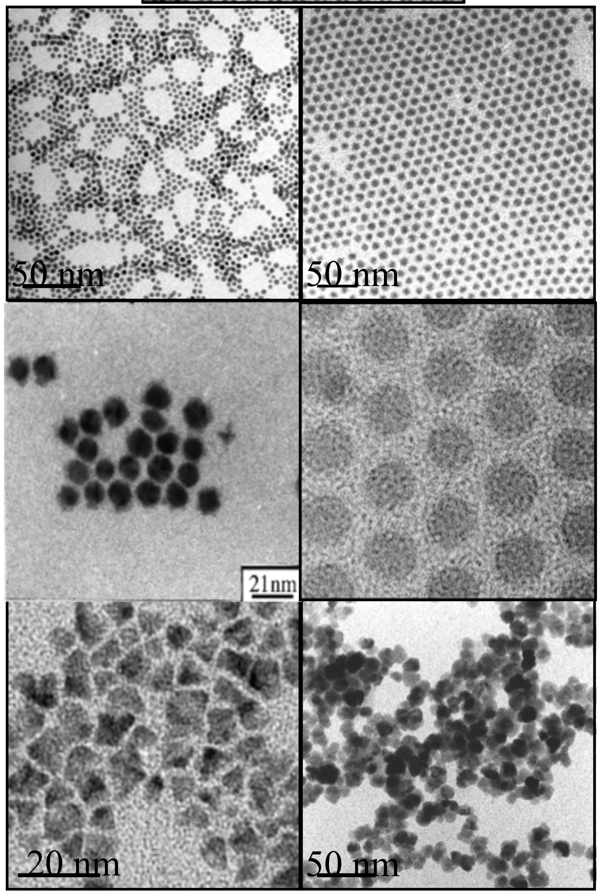
Silver
3nm<D<7nm



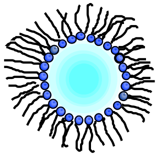
Copper
2nm<D<10nm



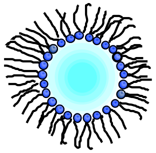
Cadmium sulfide
2nm<D<4nm



Silver sulfide
2nm<D<10nm

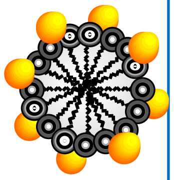


cobalt
4nm<D<10nm

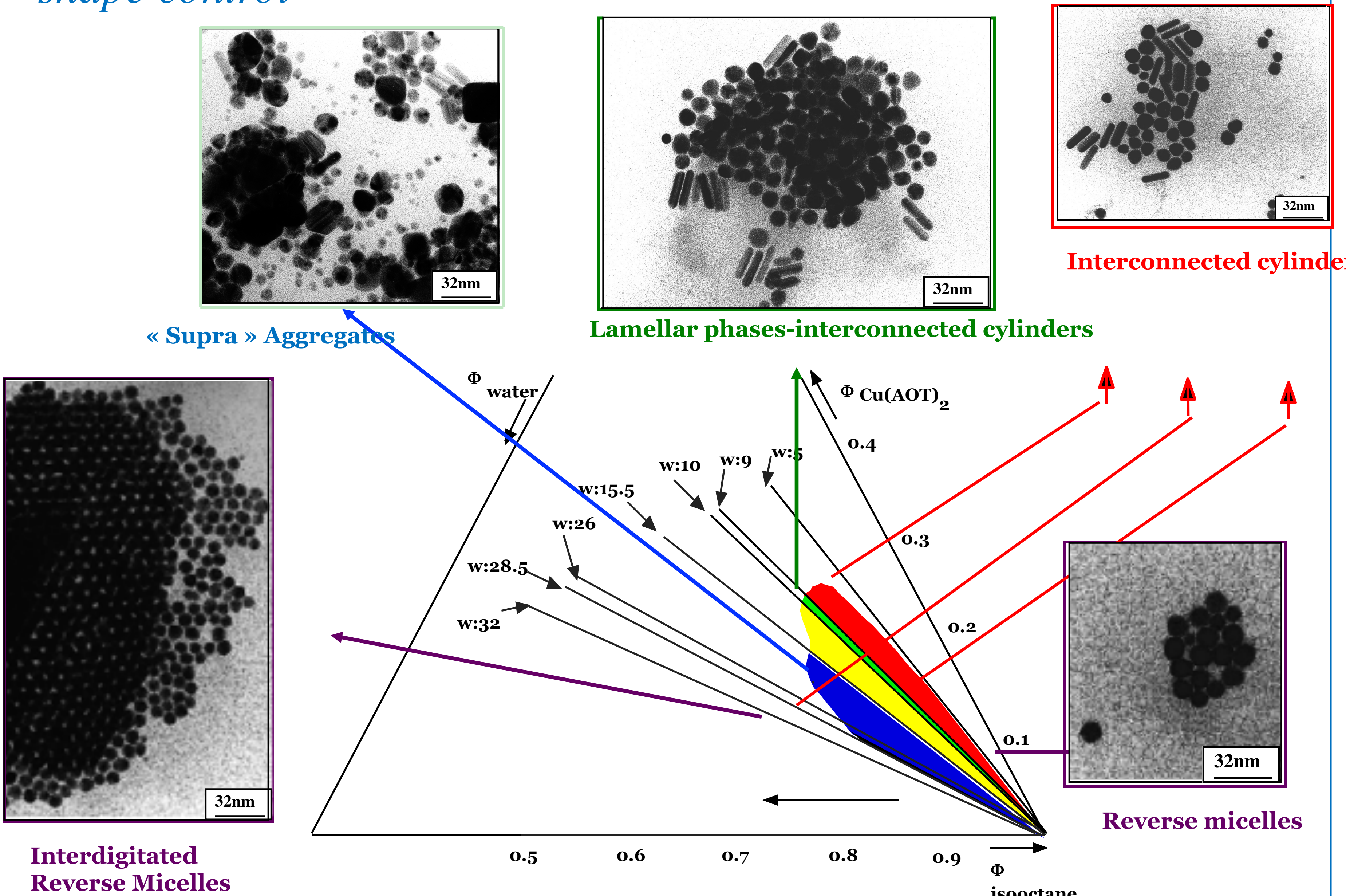


Various Ferrites
Fe_2O_3, $Fe_xCo_yO_4$, $Fe_xCo_yZn_zO_4$,

3nm<D<10nm

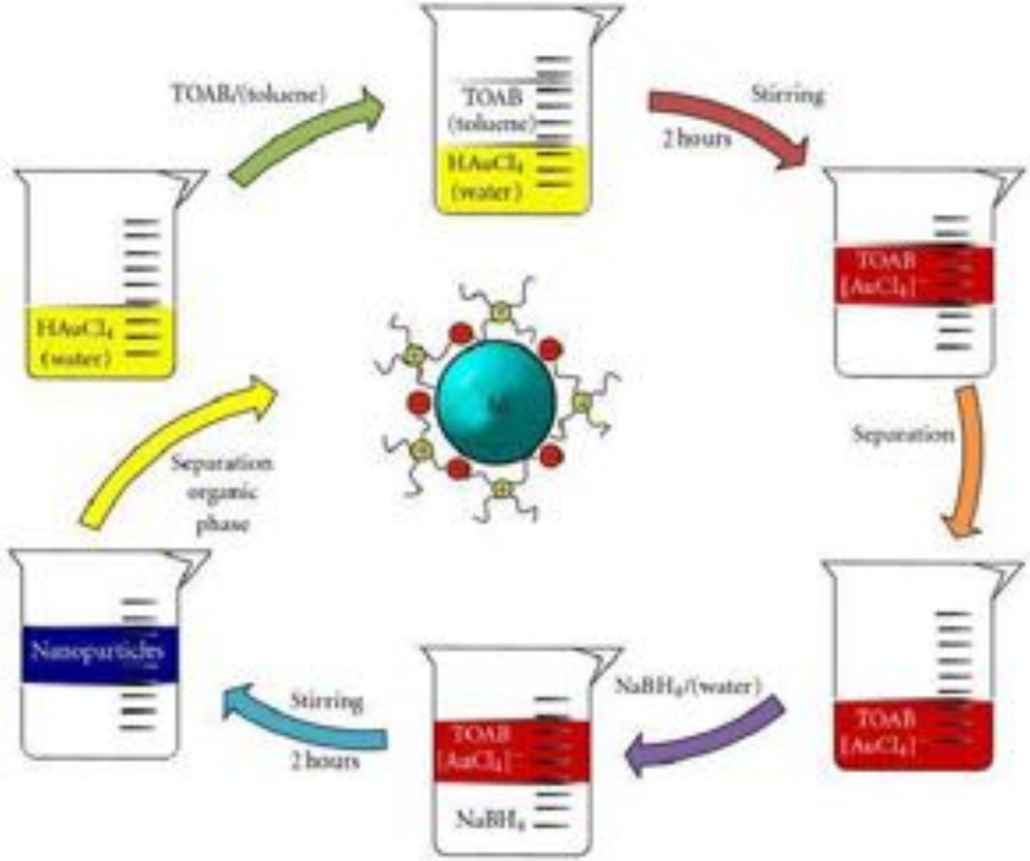


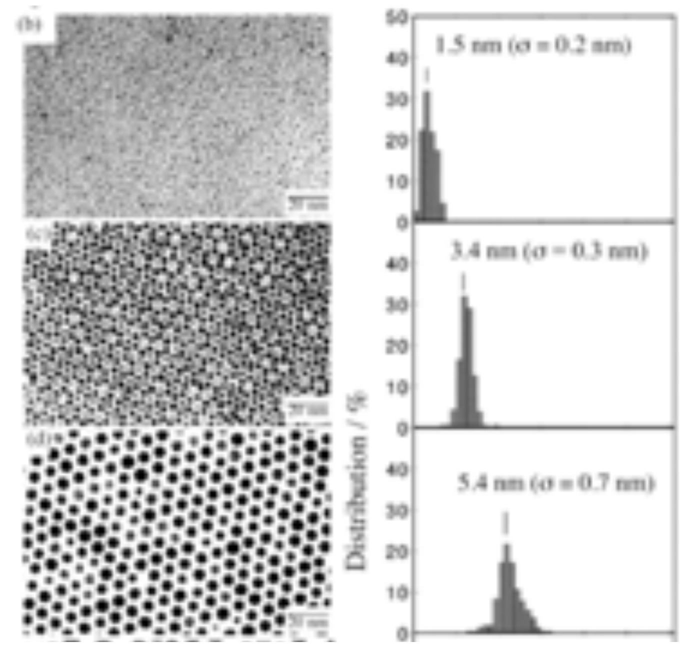
shape control



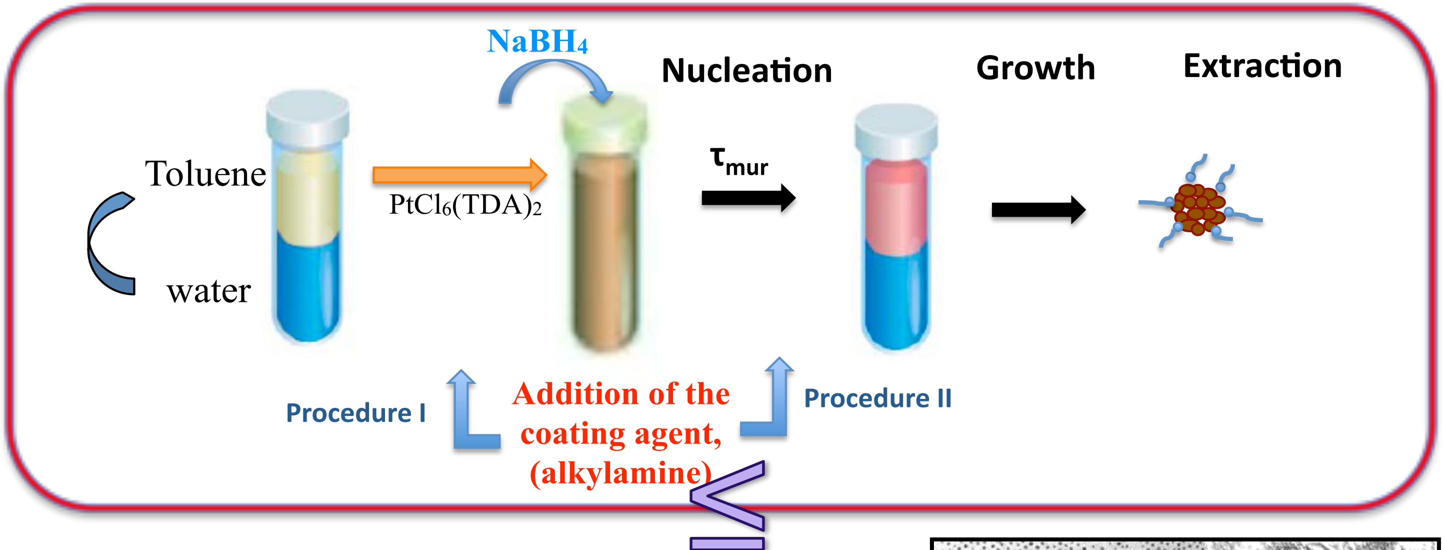
Liquid-liquid phase transfer synthesis: Au NPs

Brust method: A method for synthesizing gold nanoparticles from $HAuCl_4$ in non-aqueous solution (e.g. toluene), using tetraoctylammonium bromide as a phase-transfer catalyst and sodium borohydride to reduce Au(III) to Au(0).



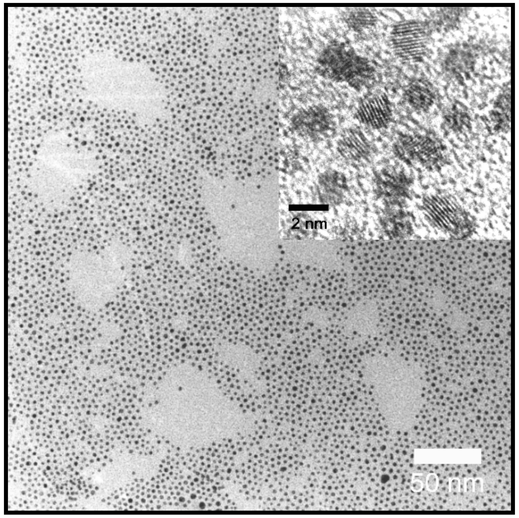


Liquid-liquid phase transfer synthesis: Pt nanoparticles



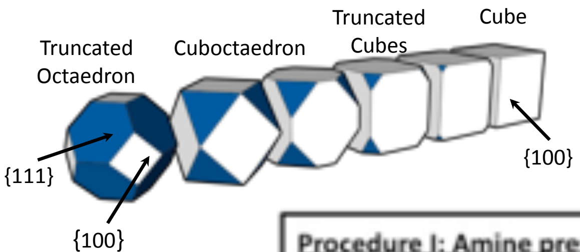
Transfer agent:
Tetrakis (decyl) ammonium bromide (TDAB)

D_{np} around 2nm

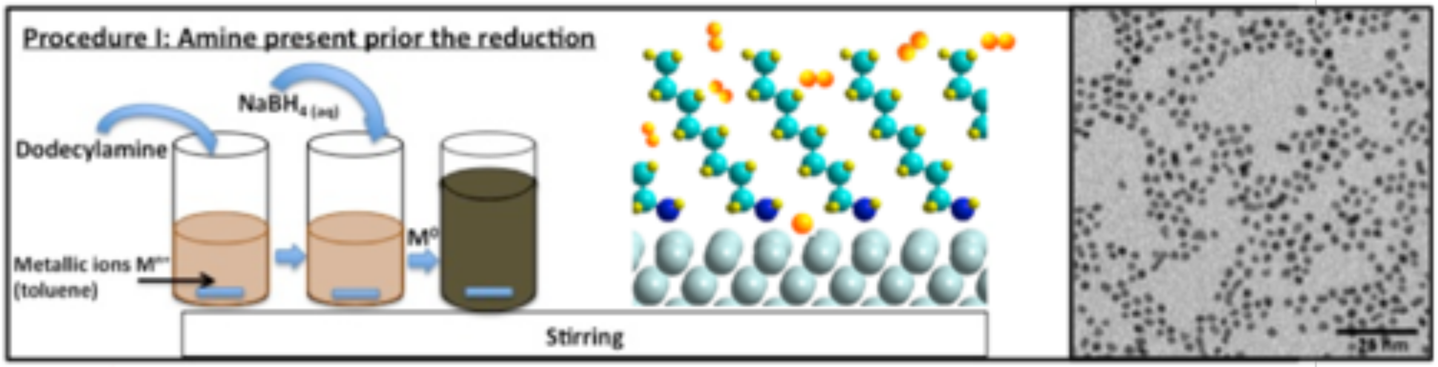


M. Brust et al.J. Chem. Soc. Chem. Com. 801 (1994)

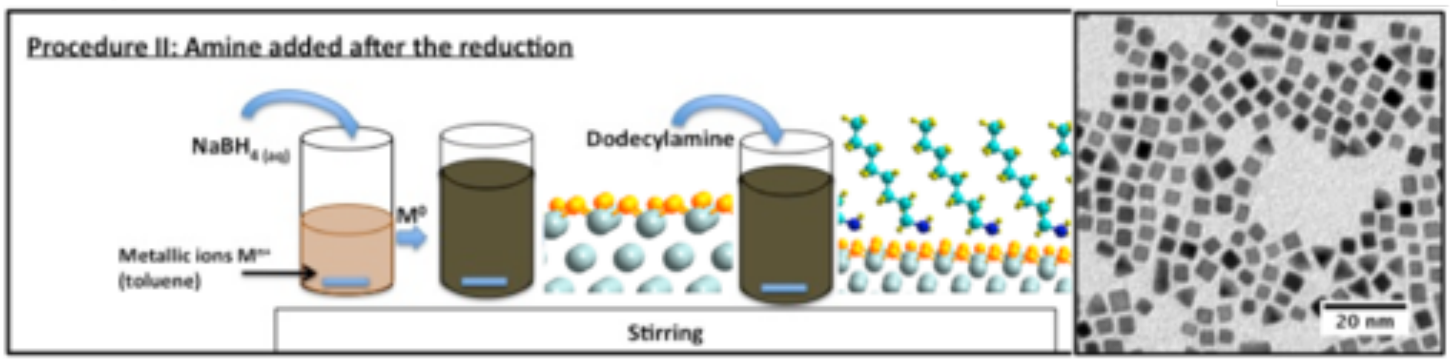
A. Demortiere and C. Petit, Langmuir, 2007, 23, 8575



Liquid-liquid Phase transfer method: shape controlled



Spherical nanoparticles



Nanocubes

The borohydride ion spontaneously hydrolyzes in aqueous solution to give hydrogen as follows: $BH_4^- + 2H_2O \rightarrow BO_2^- + 4H_2$

High temperature synthesis: polyol process

Reduction of $Pt(acac)_2$ and $Co(oleate)_2$ by 1,2 hexadecanediol

$Pt(acac)_2$

Oleylamine

Oleic acid (ligands)

Solvent 1-octadecene

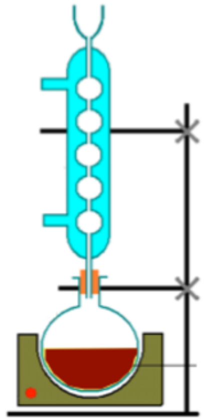
$Co(oleate)_2$

N_2

Δ

Reflux

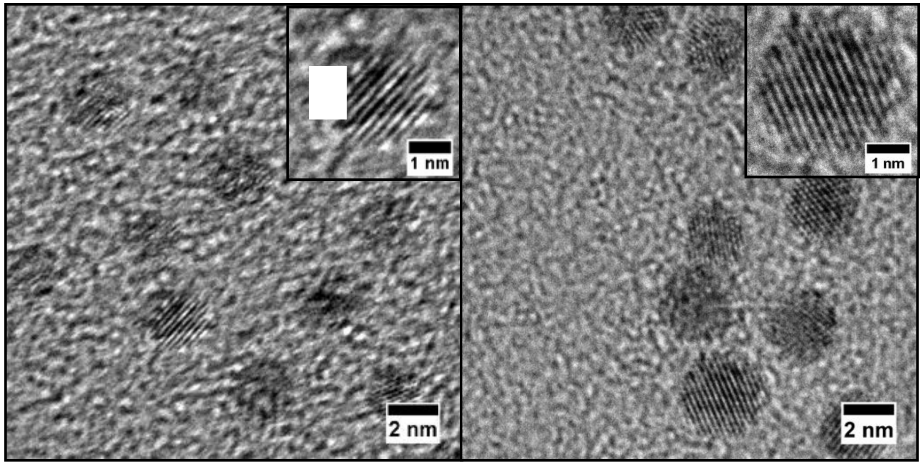
Co_xPt_{100-x}



The mixture is refluxed during 30 min and then cooled to room temperature

T=317°C

Synthesis of CoPt nanoalloys

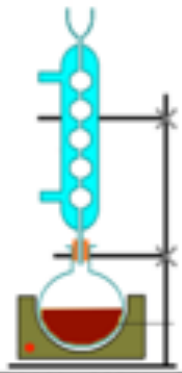


2.5 nm $Co_{52}Pt_{48}$ Polyol $Co_{32}Pt_{68}$ 2.9 nm

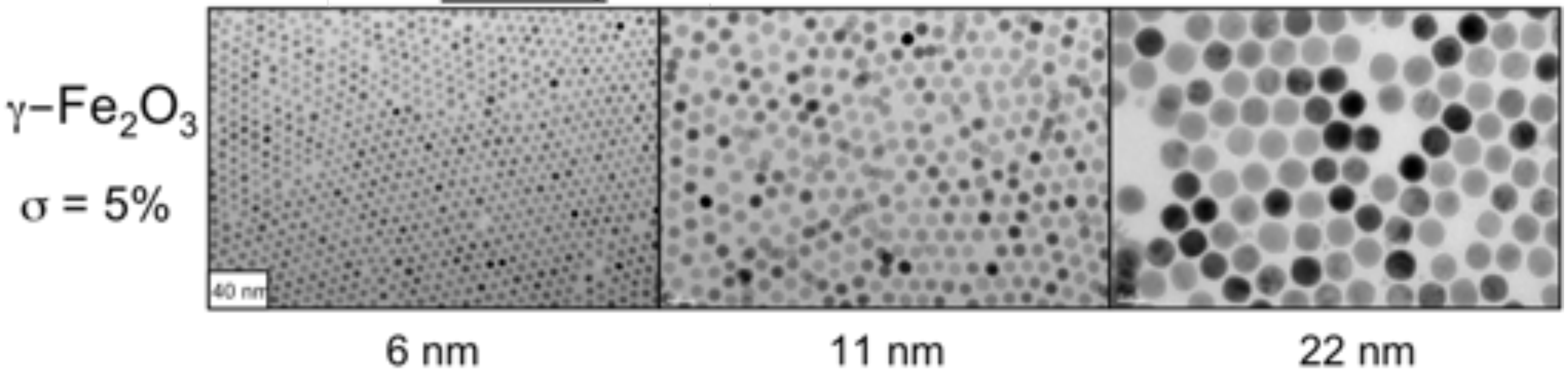
F. Kameche, Phys. Chem. Chem. Phys., 2015, 17, 28162

Thermal decomposition

Synthesis of iron oxide nanoparticles



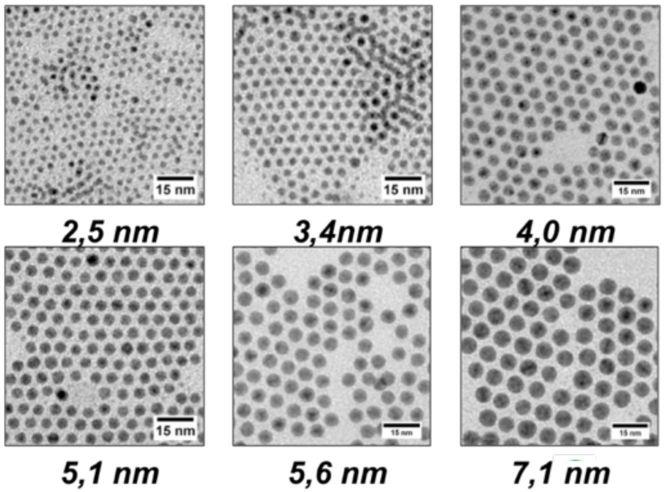
Iron(oleate) 3
+ Oleic acid dissolved in organic solvent
T=b.p of the solvent



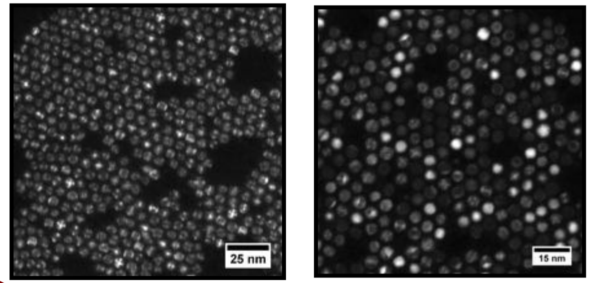
The particle size of the iron oxide nanocrystals (between 6 and 22 nm in diameter) is controlled by using various solvents with different boiling points

Reduction of organometallic salt

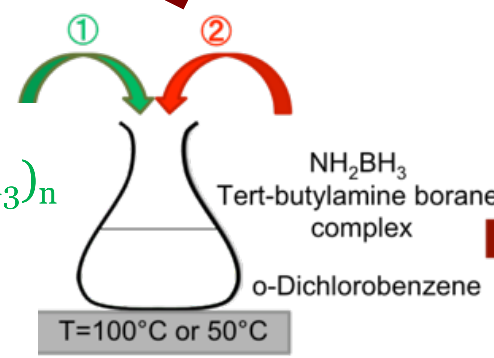
Ag NPs



T=100°C T=50°C

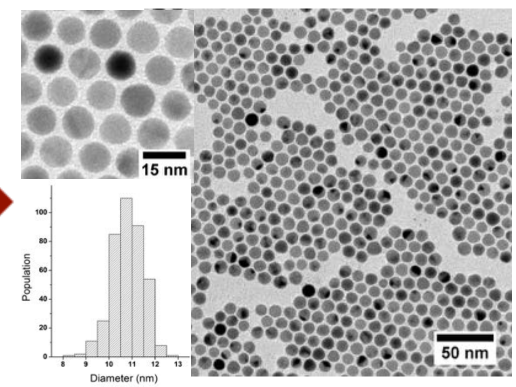


Cu NPs



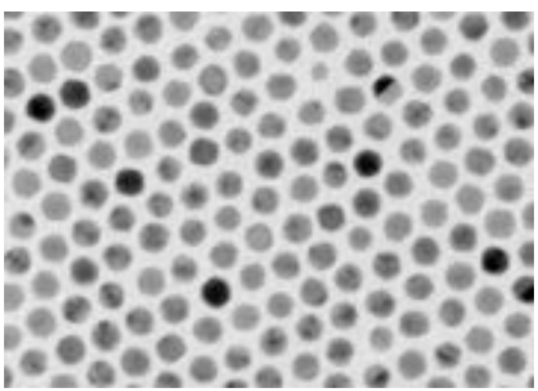
R-M(PPh_3)$_n$

+ ligands (alkylamine/alkanethiols)



T=100°C D= 10.7 ± 0.7 nm

Au NPs



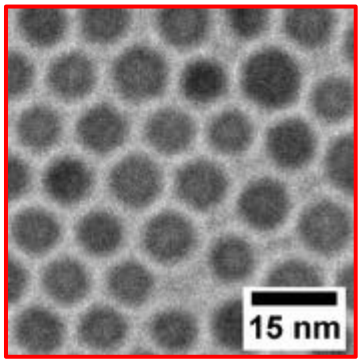
T=100°C D= 5 ± 0.3 nm

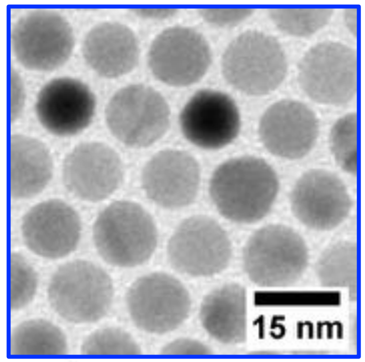
A. Courty *et col*, Nanoscale, 2015, 7, 3189-3195;N. Goubet et al. J.P.C.lett. 5, 2011, 417

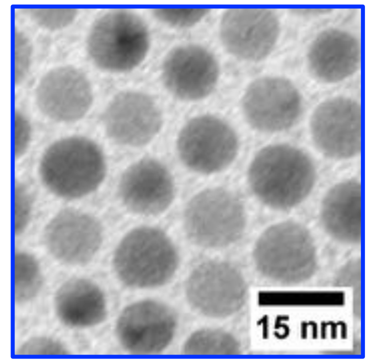
Reduction of organometallic salt: size control

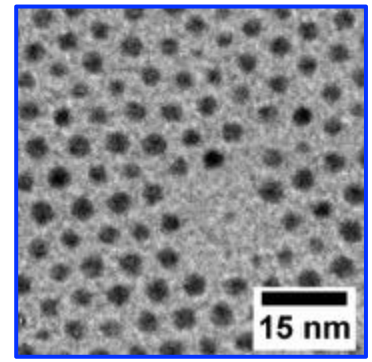
Copper nanoparticles coated with dodecylamine

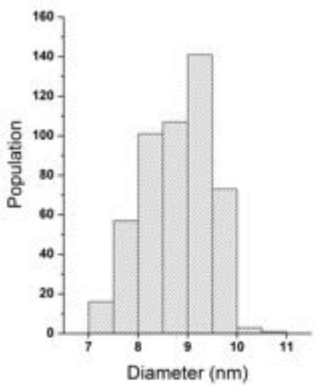
T ← 100°C | 100°C | 80°C | 50°C

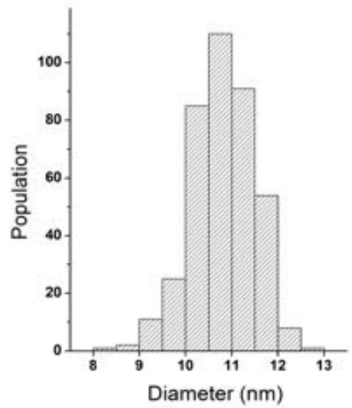


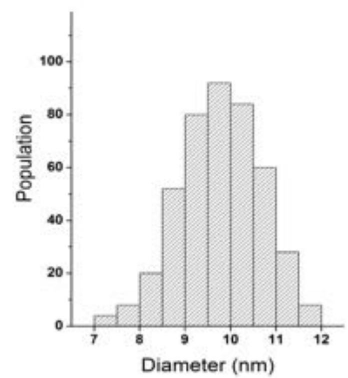


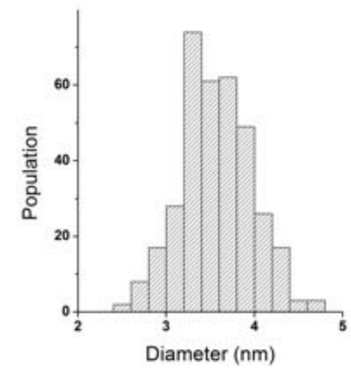












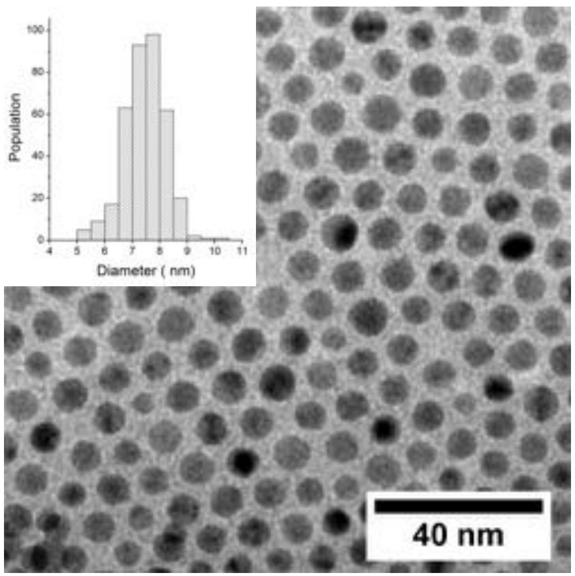
8.7 nm (8%) *10.7 nm (7%)* *9.7 nm (8%)* *3.5 nm (11%)*

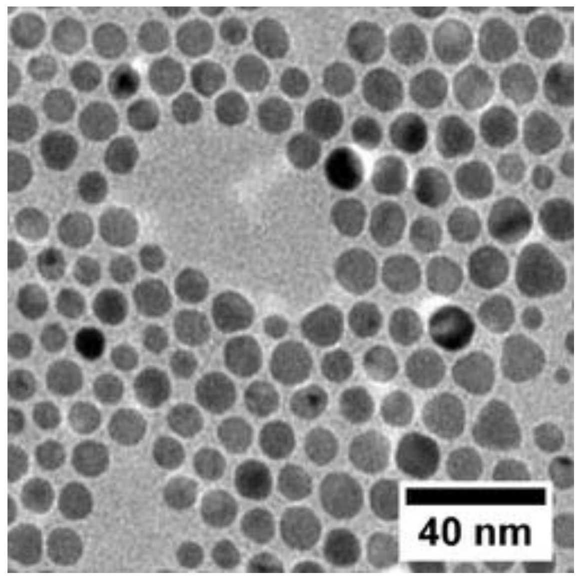
16:1 *Molar ratio of DDA to copper* 8:1

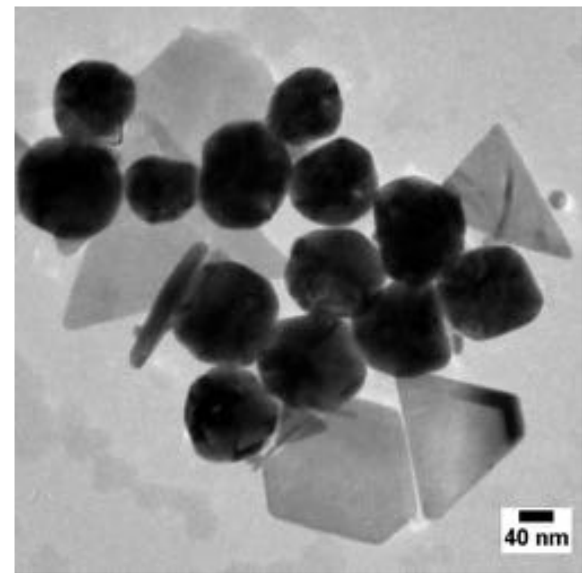
Reduction of organometallic salt: size and shape control

Copper nanoparticles coated with oleylamine

50°C — 80°C — 100°C → T





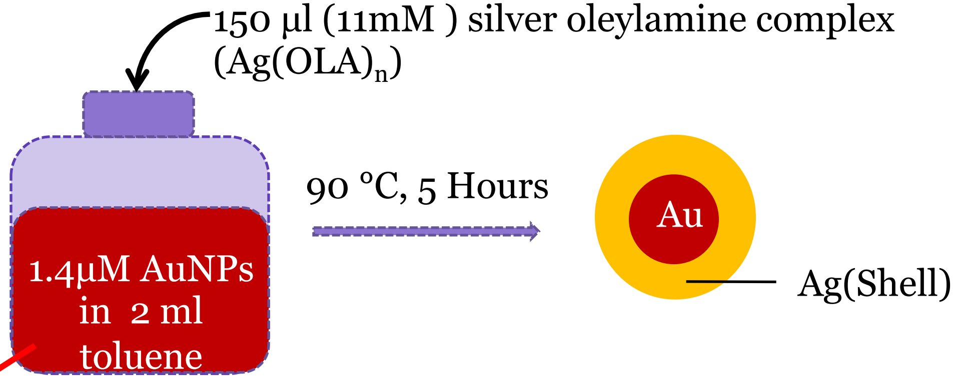


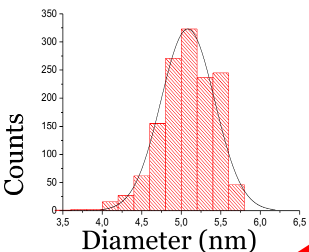
7.4 nm (9%)

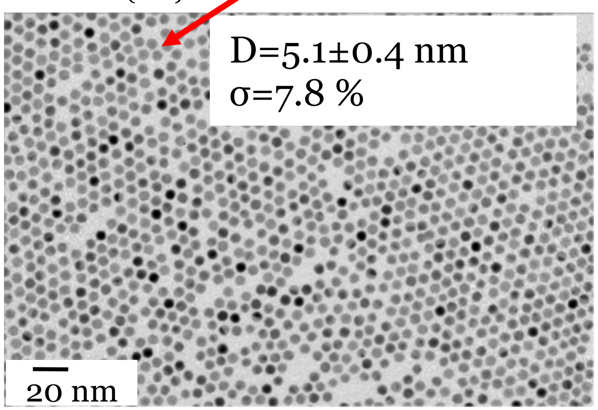
The nature of the amine ligand influences the final size and shape of the CuNPs

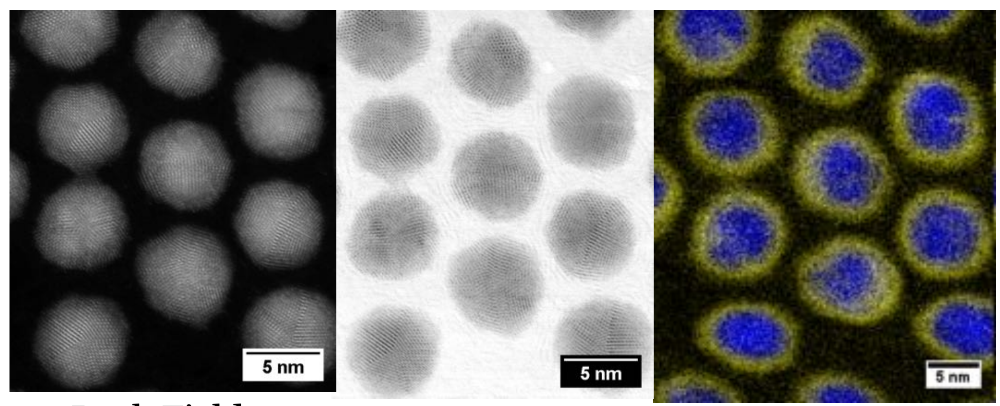
Seed mediated growth : towards core –shell nanoparticles

Seed-mediated growth of Au@Ag NPs









Dark Field
silver
gold

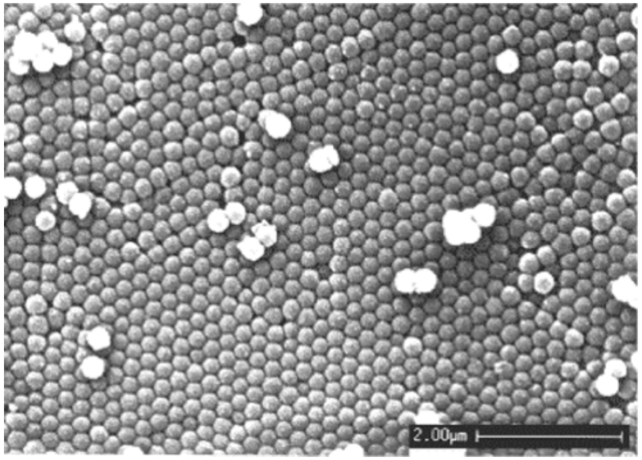
Bright Field
silver
gold

EELS
silver
gold

Self-organizations or Nanoparticles at 2D and 3D

Where does this idea of self-organization?

From the nature!!



SEM image of silica NPs neatly arranged in natural opal

The color of the opal comes from the Bragg diffraction of the light by a regular network of silica particles with 100-500 nm in diameter .

W.Li et al. Materials Chemistry and Physics 116 (2009) 164–168

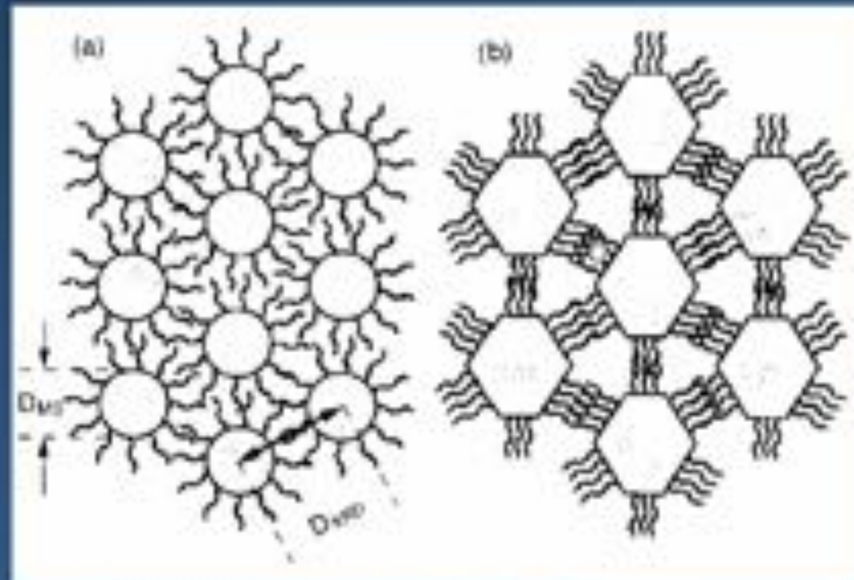
How to assemble Nanoparticles?
Self-assembly of nanoparticles to superlattices

Nanocrystals are able to assemble into close-packed ordered superlattices under the following conditions:

- narrow size distribution

- surfactant that is strong enough to separate the individual nanocrystals

- slow drying rate so that the nanocrystals can move to suitable positions



Schematic illustration of self-assembled, passivated nanocrystal superlattices of spherical (a) and faceted (b) particles.

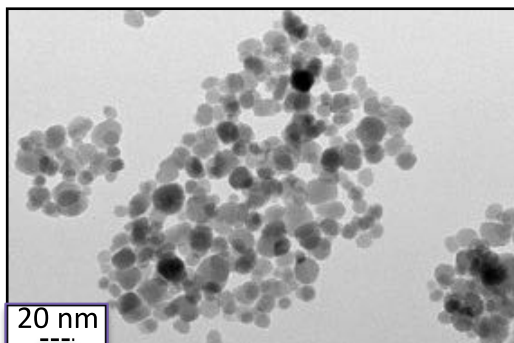
Wang, Adv. Mater. **1998**, *10*, 13-30

Self-organization: influence of the size polydispersity

11 nm

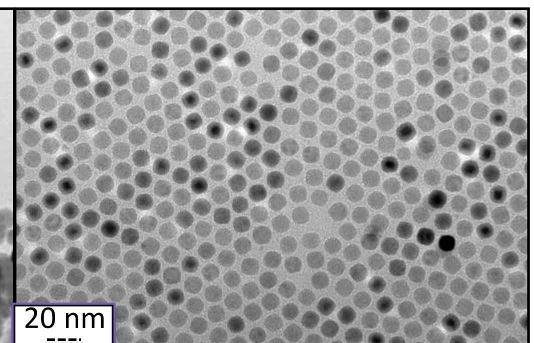
$\gamma\text{–}Fe_2O_3$

Co-precipitation



σ = 25%

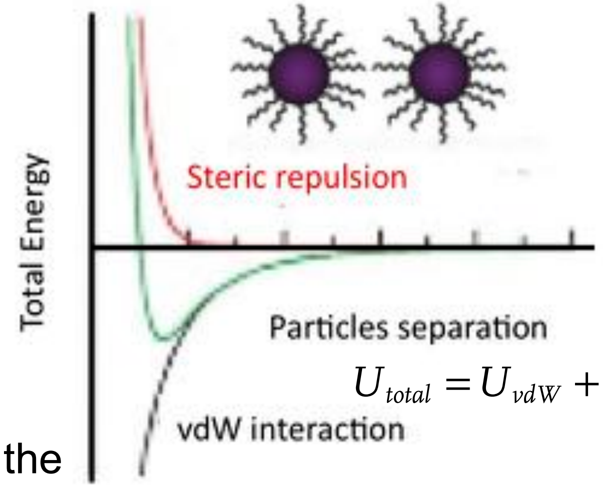
Thermal décomposition



σ = 6%

Dr Anh-tu Nhgo (2013)

Self-organization of nanoparticles: interparticle interaction



$$U_{total} = U_{vdW} + U_{steric}$$

Different type of interactions govern the organization:

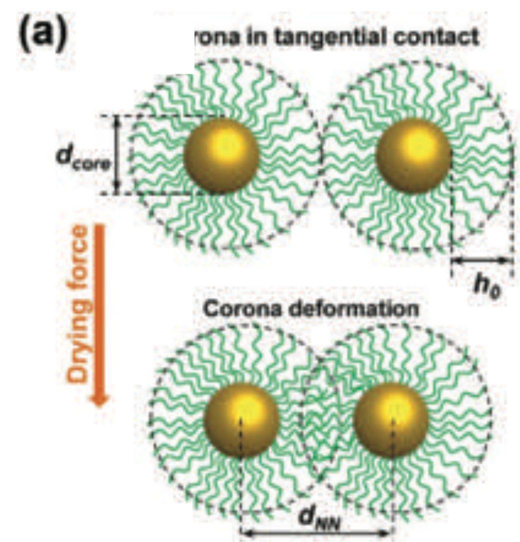
1) Van der Waals interaction (attractive)

$$U_{vdW} = -\frac{A}{12}\left[\frac{d_{core}^2}{d_{NN}^2 - d_{core}^2} + \frac{d_{core}^2}{d_{NN}^2} + 2\ln\left(\frac{d_{NN}^2 - d_{core}^2}{d_{NN}^2}\right)\right]$$

where A is the Hamaker constant that depends on the polarizability of nanoparticle and surrounding medium.

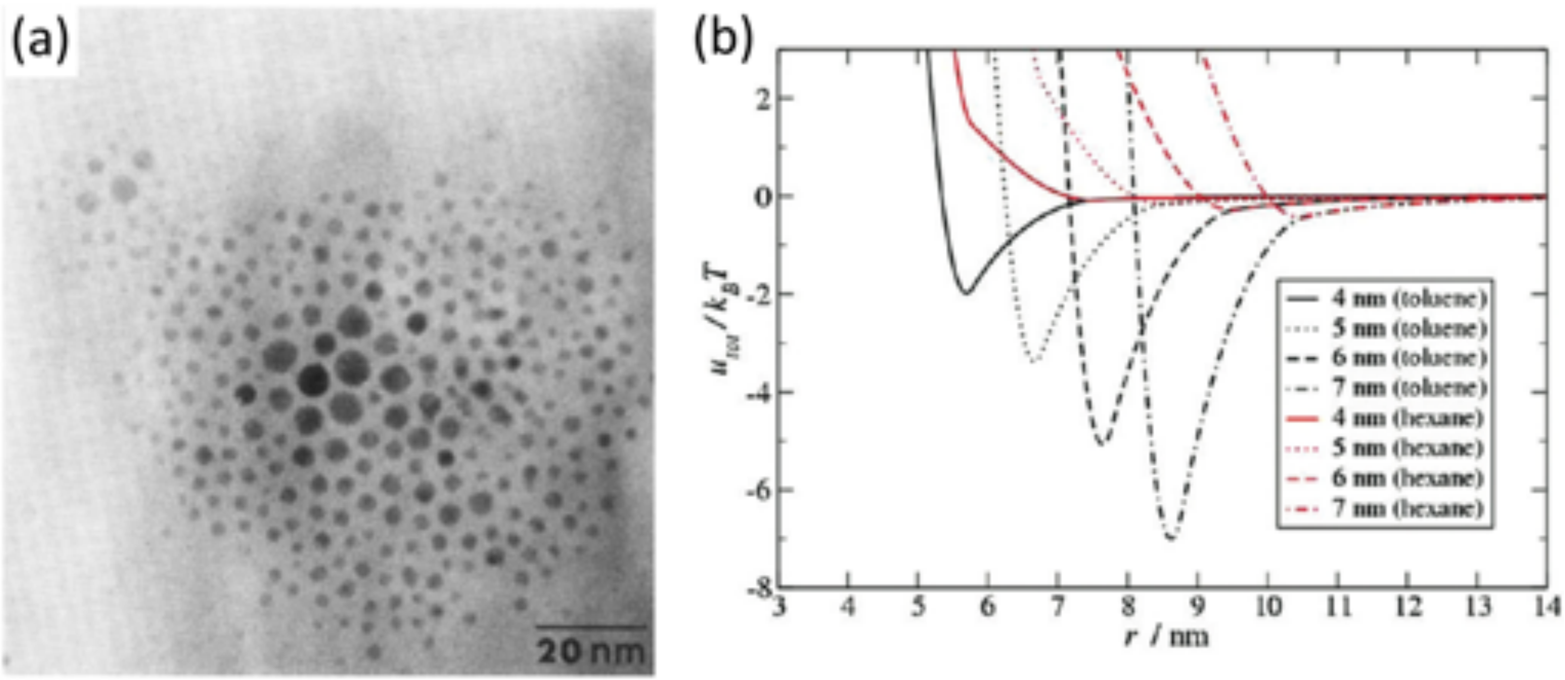
2) Steric interaction (repulsive)

$$U_{steric} = \frac{100 R h_0^2}{(d_{NN} - d_{core})\pi\sigma^3} k_B T \exp\left[-\pi\left(d_{NN} - d_{core}\right)/h_0\right]$$



Self-organization of nanoparticles: interparticle interactions and solvent effects

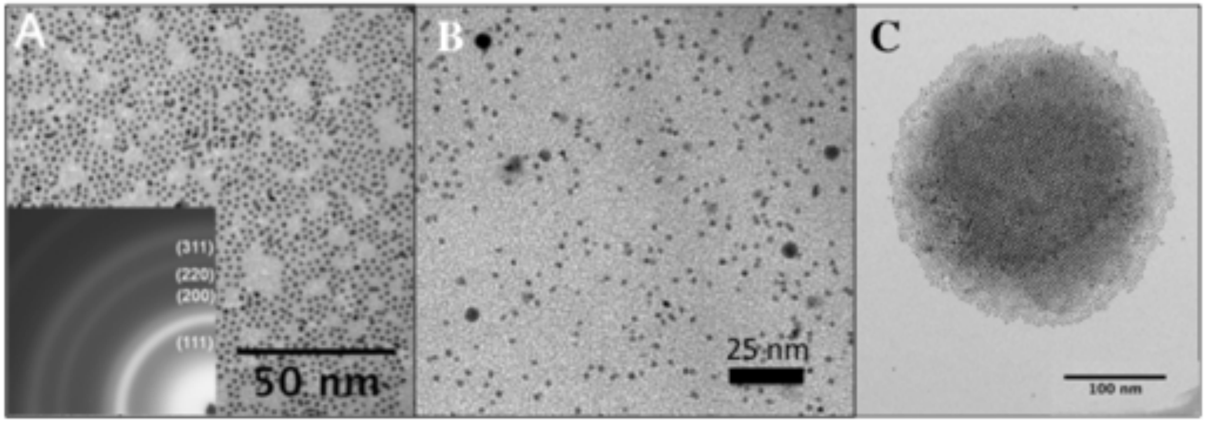
The van der Waals interactions, which are attractive, are highly dependent on the size of the interacting nanoparticles.



N. Goubet, J. Richardi, P.A. Albouy, and M.P. Pileni. Adv. Funct. Mater., 21(14) :2693–2704, 2011.

P. C. Ohara, D. V. Leff, J. R. Heath, and W. M. Gelbart. Phys. Rev. Lett., 75(19) :3466–3469,1995.

Self-organization of nanoparticles: solvent effects

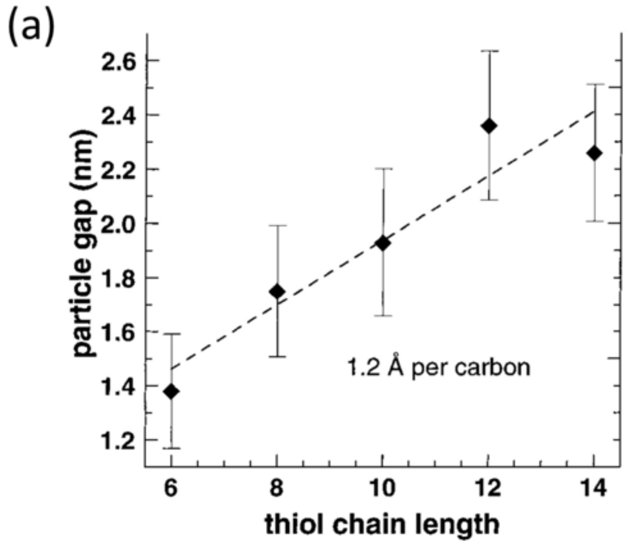


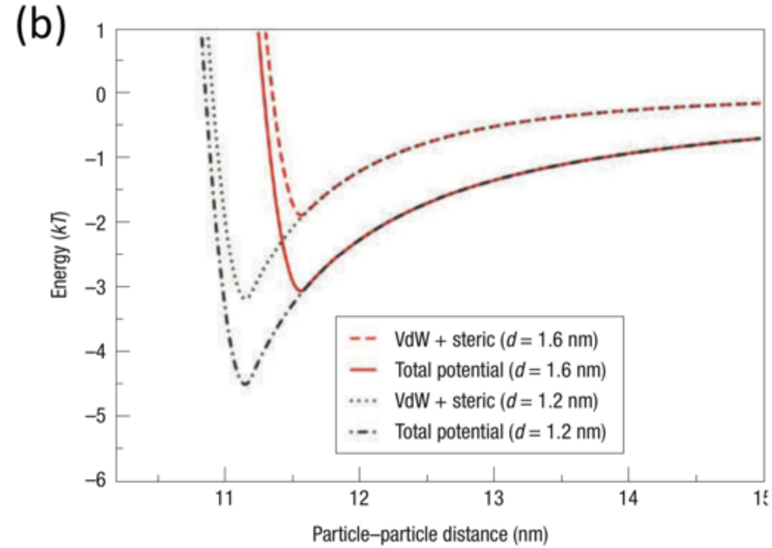
2nm CoPt nanoalloys deposited on amorphous carbon TEM grid, coated by dodecylamine and dispersed in :

(A) Hexane ($T_{boiling}$ = 68°C; viscosity = 0.3 mPa.s)
(B) 1-Phenyl-octane ($T_{boiling}$ = 261°C; viscosity = 1.5 mPa.s)
(C) Toluene ($T_{boiling}$= 111°C, viscosity =0.6 mPa.s)

- The high viscosity of phenyl-octane hinders the diffusion of the NC at the surface in addition to the interdigitation between the ligands chains
- With toluene (a bad solvent of the capping chains) the inter-particles interactions are more attractive than in hexane yielding some aggregation in solution

Self-organization of nanoparticles: steric interaction effect





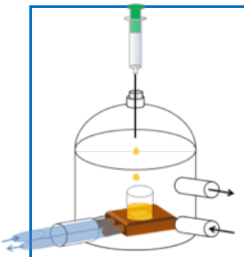
Control of the interparticle distance between gold nanoparticles by tuning the alkyl chain lenght

Modulation of interaction potential by varying the alkyl chain length of coating agent of 10 nm maghemite NPs

J. E. Martin, J. P. Wilcoxon, J. Odinek, and P. Provencio.. J. Phys. Chem. B, 104(40) :9475–9486, 2000.

Y. Lalatonne, J. Richardi, and M. P. Pileni.. Nat. Mater. 3(2) :121–125, 2004.

Self-organization of nanoparticles: interparticle interaction and solvent effects

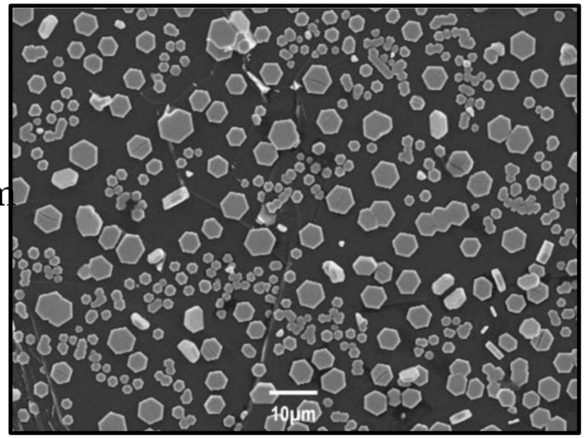


T = 50° C

AgC$_{10}$ 5nm

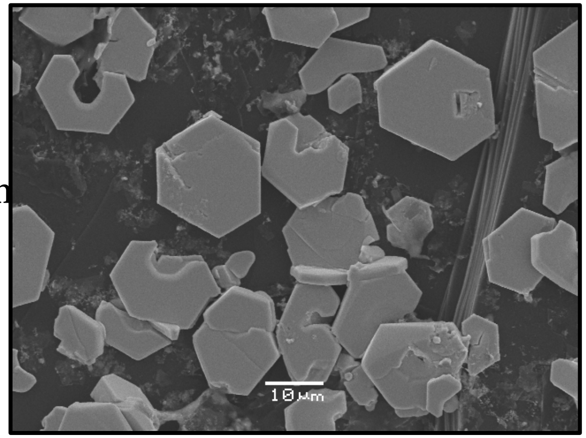
Hexane

2 to 10 µm



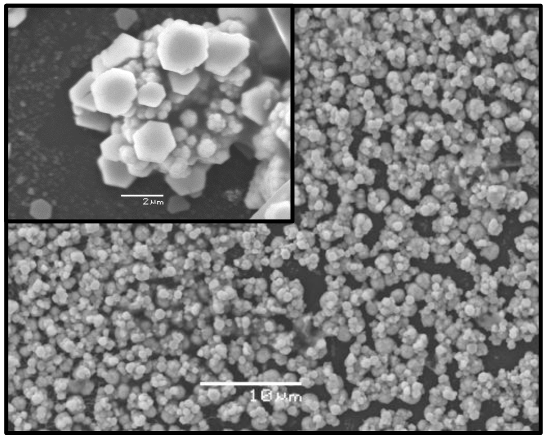
Toluene

20 to 100 µm



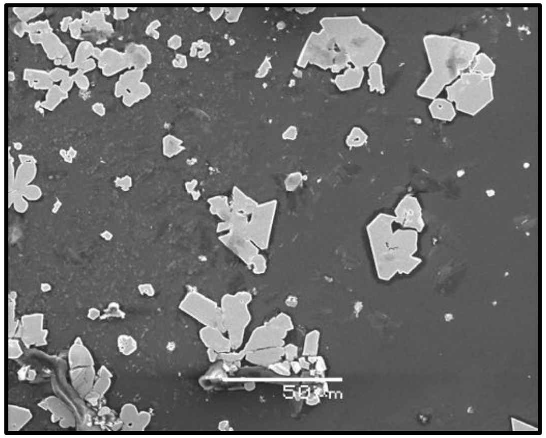
AgC$_{12}$ 5nm

Hexane



~ 2 µm

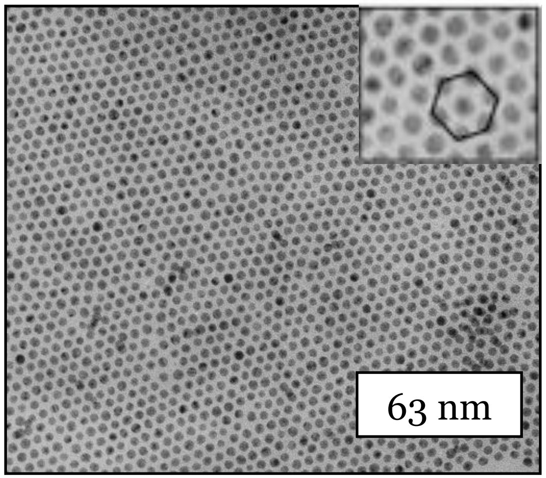
Toluene



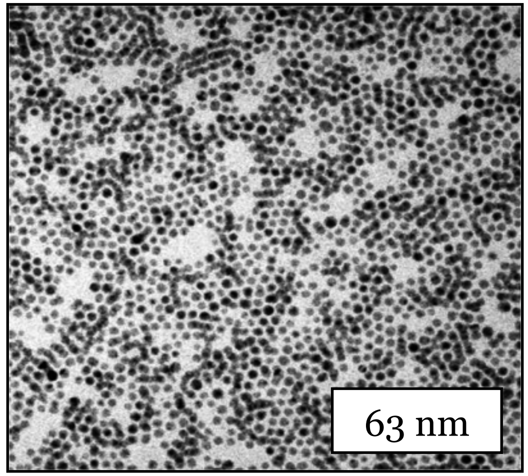
5 to 50 µm

A.Courty et coll, Chem Mater 2016, DOI: 10.1021/acs.chemmater.6b01374

Self-organization of nanoparticles: substrate effects



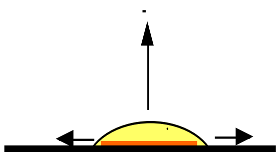
HOPG
2D hexagonal network



Amorphous carbon

5nm AgC_{12} NCs

Solvent hexane



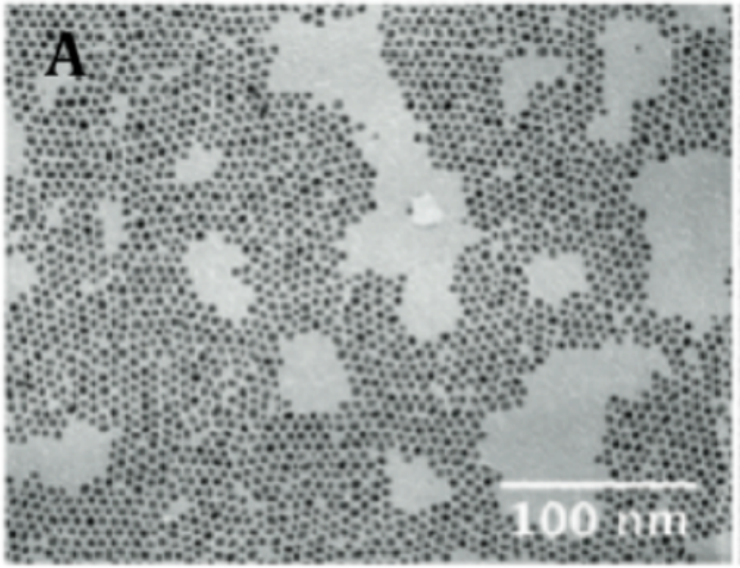
- Change in the compactness of the film due to a change of the wettability of the substrate.

- The hydrophobicity of the substrates increases with its roughness.

M.P. Pileni, Y. Lalatone, D. Ingert, I. Lisiecki and A. Courty, *Faraday Discussion*, 125, 251- (2004).

Self-organization of nanoparticles: interparticle interaction effects

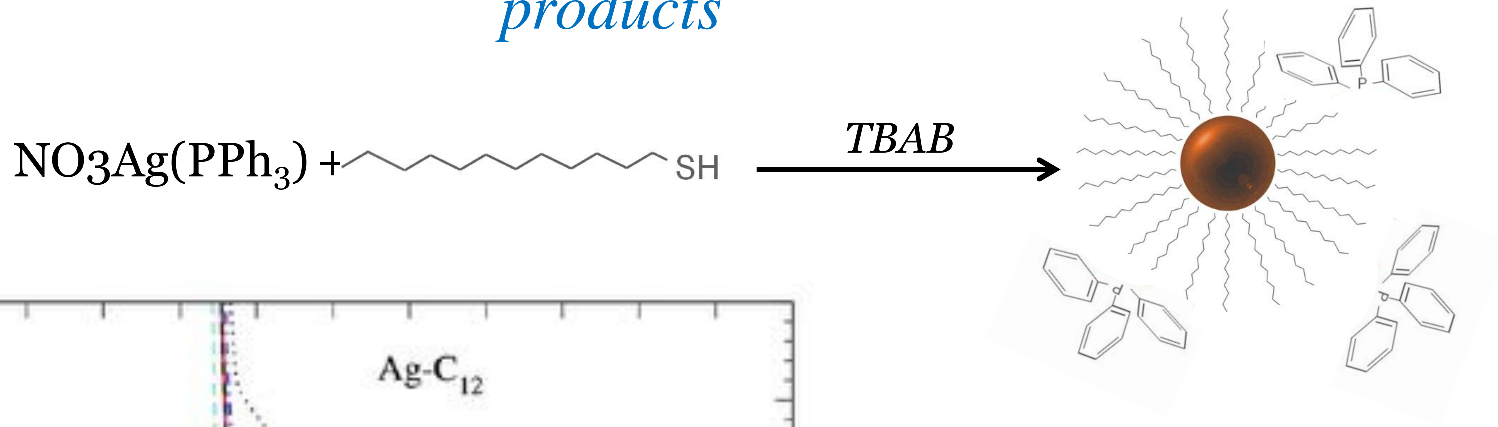
HOPG

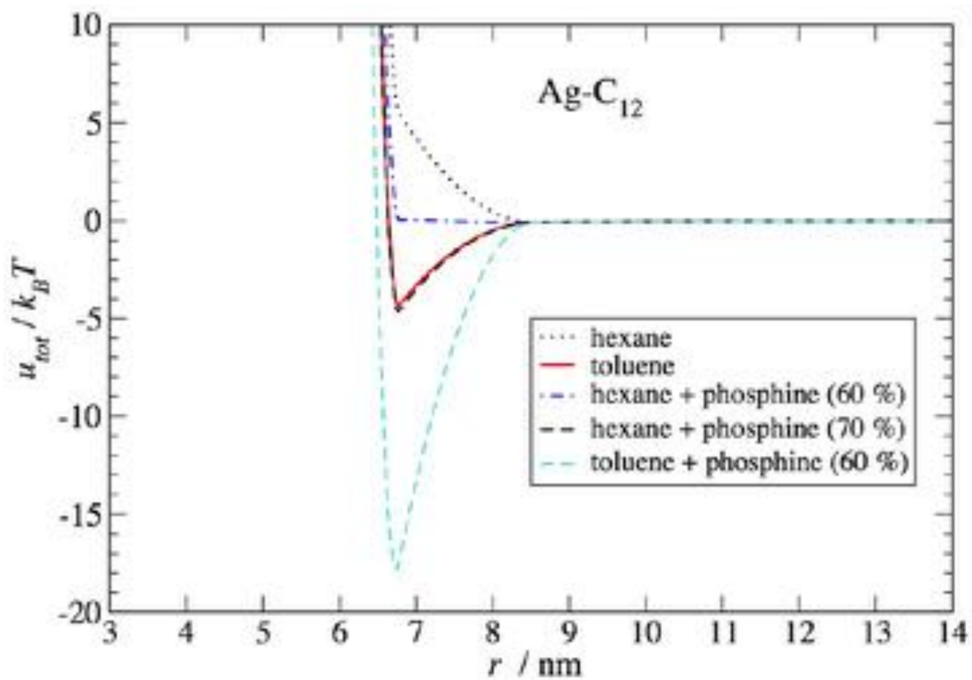


2nm CoPt nanoalloys

- This decrease in the ordering compared to assemblies of AgNCs is due to a decrease in the interaction energy with the size of the NCs

Self organization of nanoparticles: role of the solvent and by products

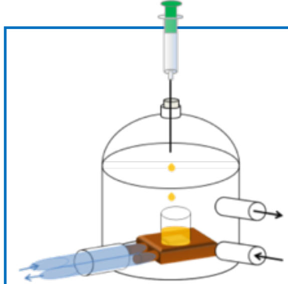




- The presence of PPh_3 in hexane leads to an attraction between the nanocrystals which is favorable for SL growth in solution. In toluene the interaction potential becomes even more attractive

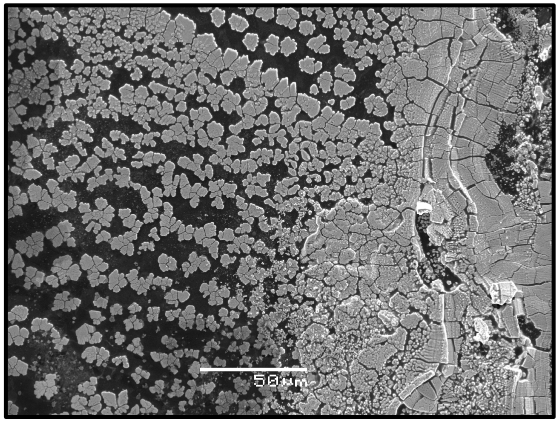
Calculation from a Flory-type model of the interaction potential between silver NCs

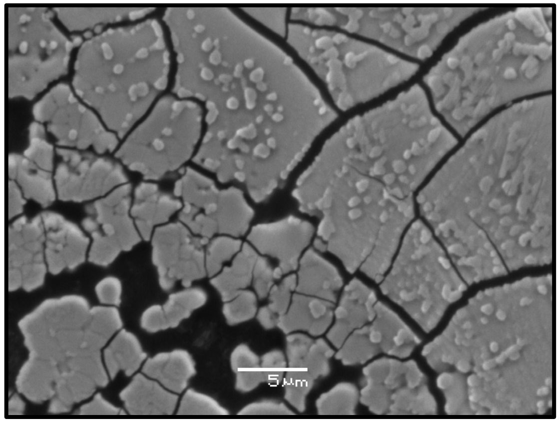
A.Courty et coll, Chem Mater 2016, DOI: 10.1021/acs.chemmater.6b01374



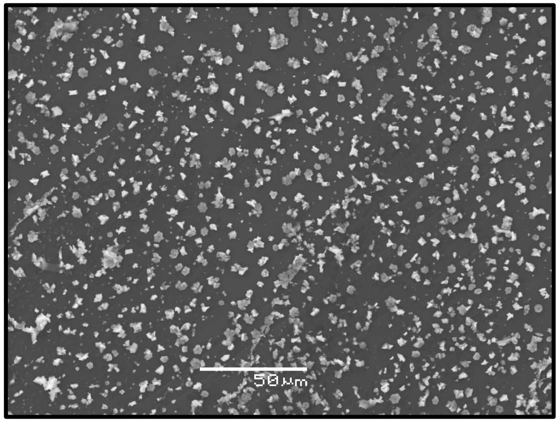
Self organization of nanoparticles: role of the solvent and by products

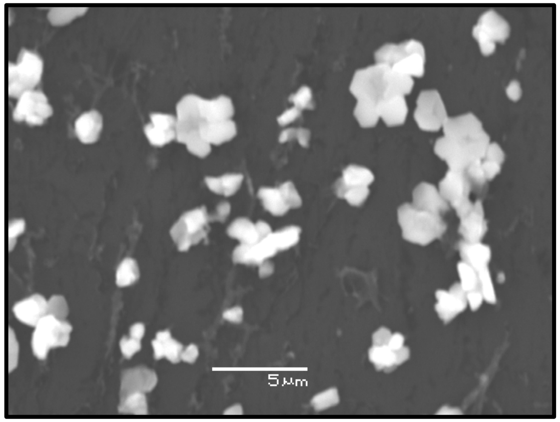
$Ag_{5nm}C_{12}$

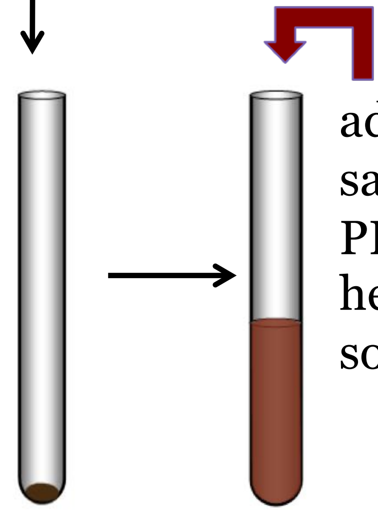




AgC_{12} NPs washed by acetone and dispersed in pure hexane solution (No more PPh_3)







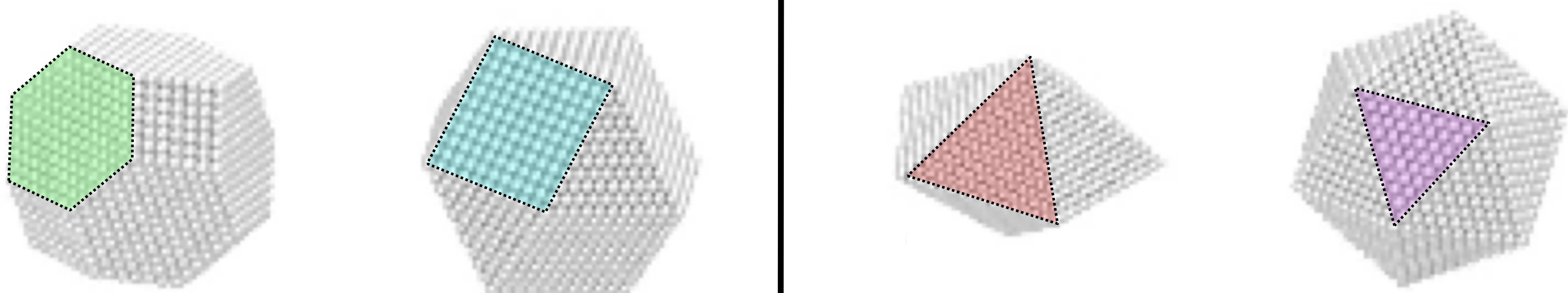
addition of saturated PPh_3 hexane solution

A.Courty et coll, Chem Mater 2016, DOI: 10.1021/acs.chemmater.6b01374

Self-Organization : Influence of the NP morphology &crystallinity

The NP morphology, which depends on crystallinity, can influence the ability of NPs to interact with neighboring NPs and to self-assemble in close-packed superlattices

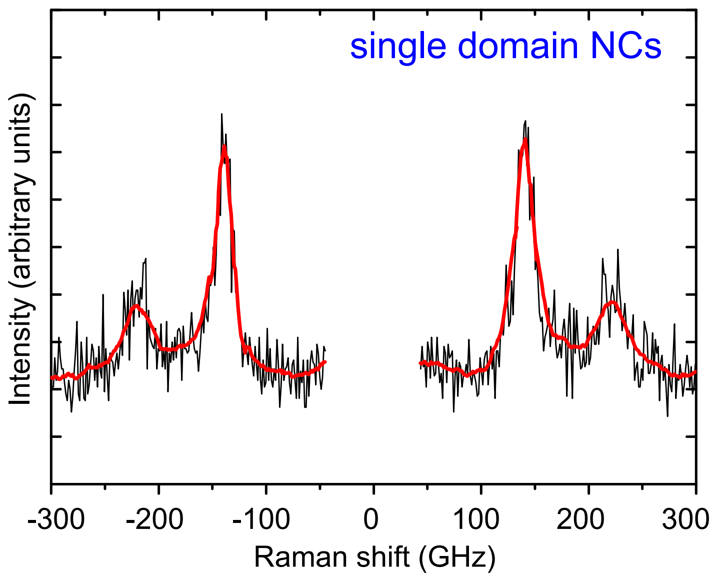
single domain NCs | multiply twinned particles

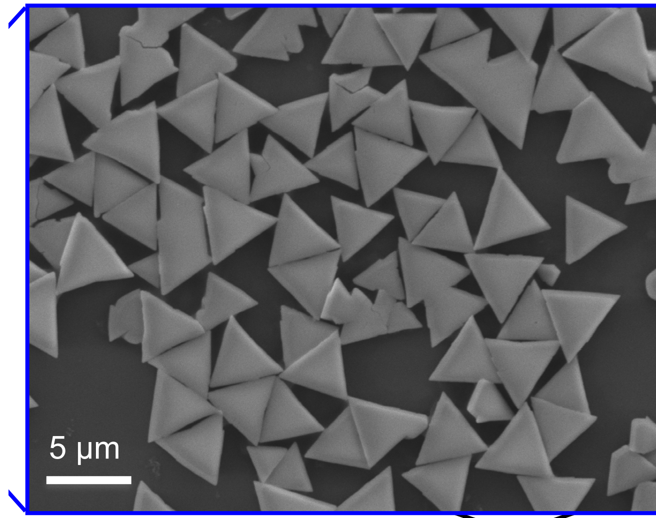


truncated octahedron — cuboctahedron — decahedron — icosahedron

- Larger **facets in single domain NCs** compared to those in MTPs
- Larger facets ➔ stronger NP-NP interactions

Self-Organization : Influence of the NP crystallinity



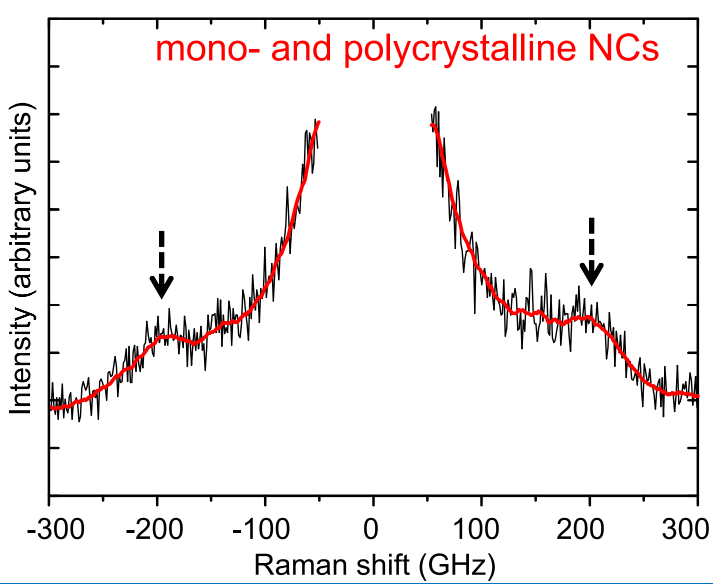


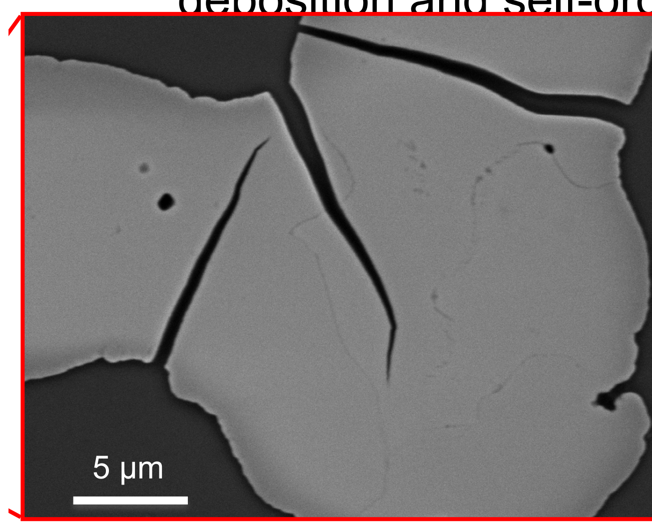
truncated-
tetrahedral
colloidal
crystals

of
ated NCs
aker

deposition and self-organization

Si wafer



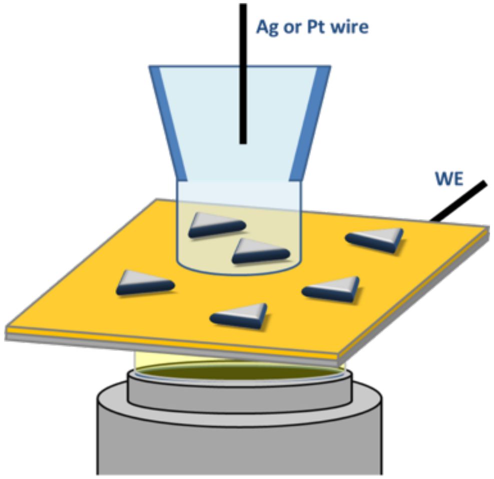


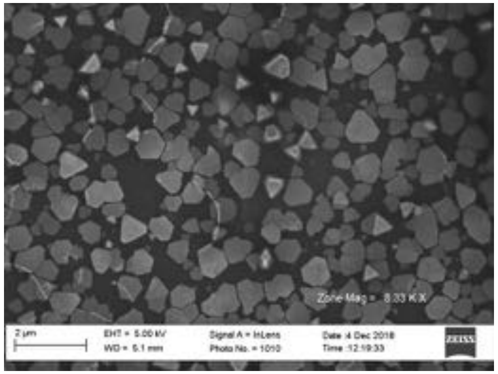
nanocrystal film
with irregular
outlines

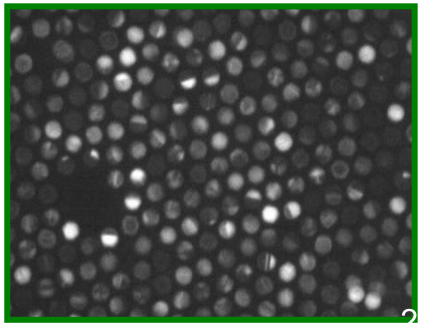
Y. Wan, H. Portalès, N. Goubet, A. Mermet and M.P. Pileni Nano Research **6**, 611-618 (2013)

Self-Organization : Influence of the NP crystallinity

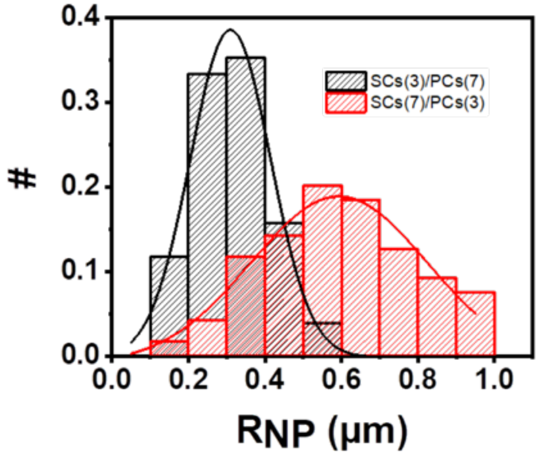
High resolution <u>optical</u> microscopies for studying the formation of Ag superlattices

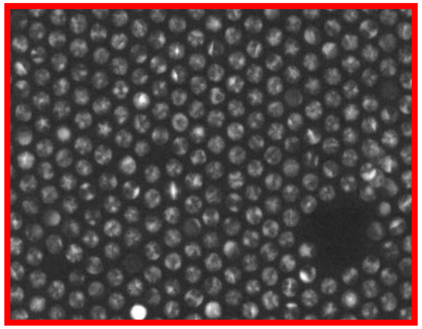






single crystals (SC)



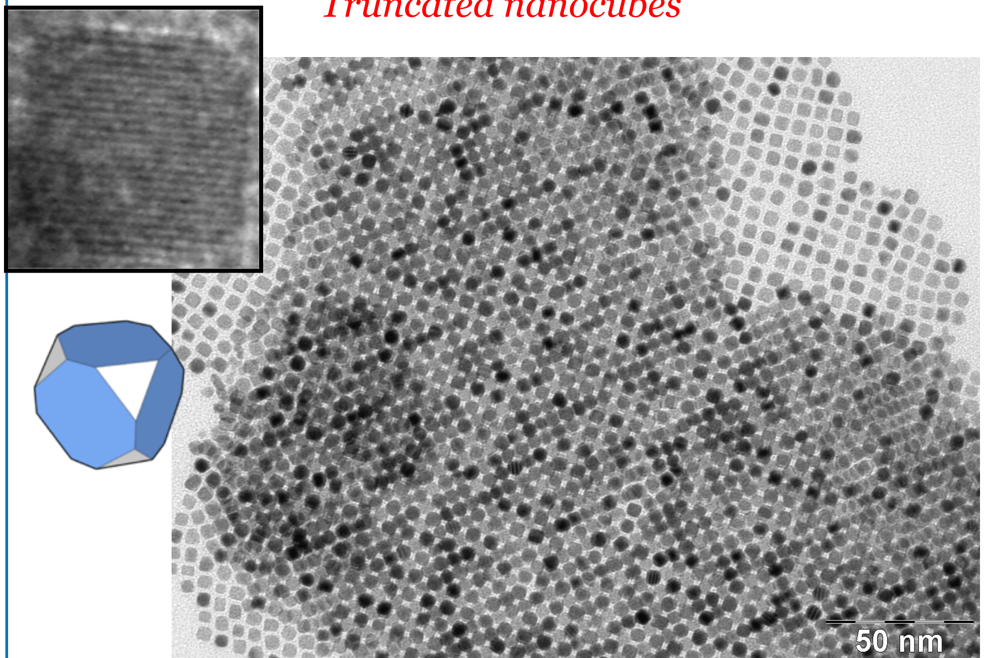


Polycrystals (PC)

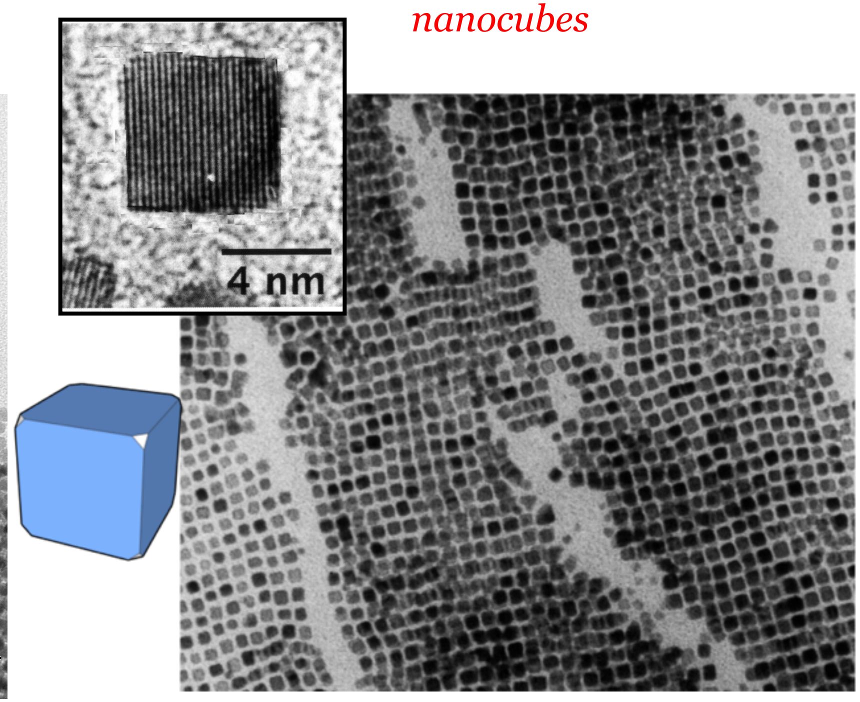
Self-Organization : Influence of the NP shape

Assemblies of Pt nanoparticles

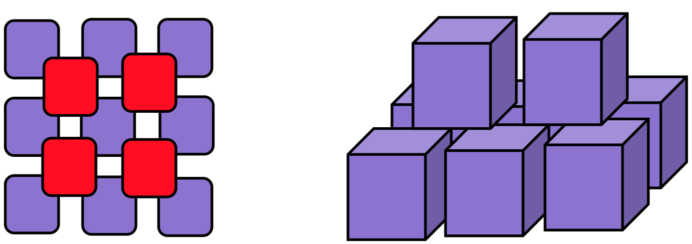
Truncated nanocubes



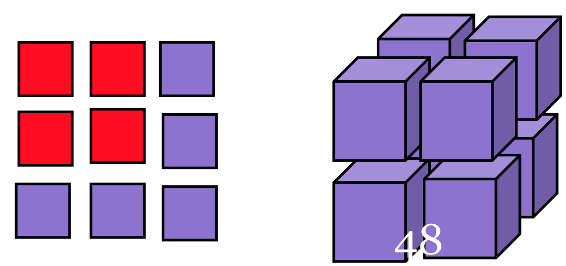
nanocubes



Face centered cubic 3D arrangement

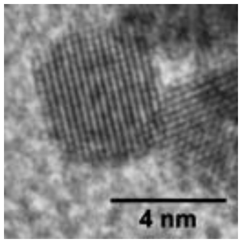


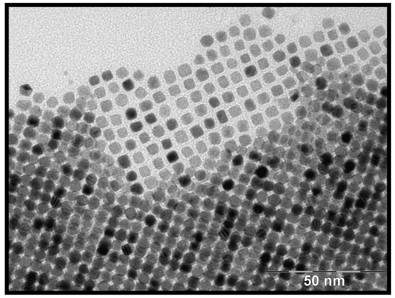
Simple cubic 3D arrangement



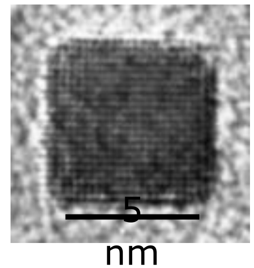
Self-Organization : Influence of the NP shape

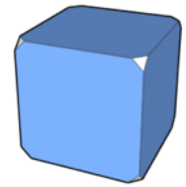
Truncated nanocubes

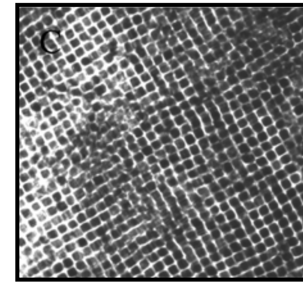




nanocubes

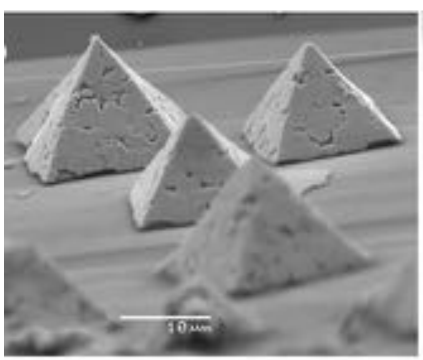


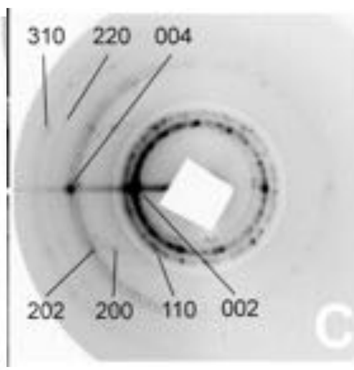


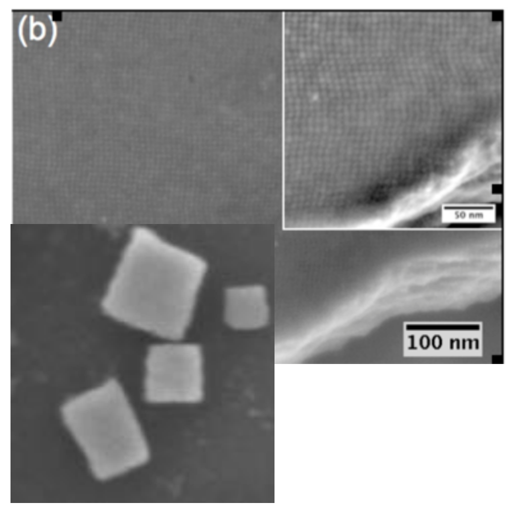


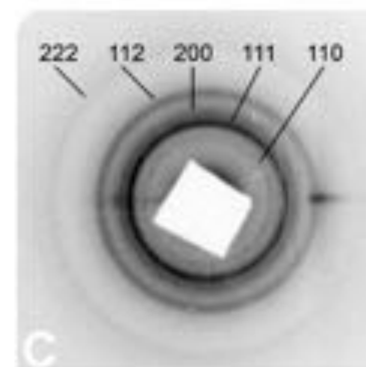
Face centered cubic 3D superlattices

Simple cubic 3D superlattices

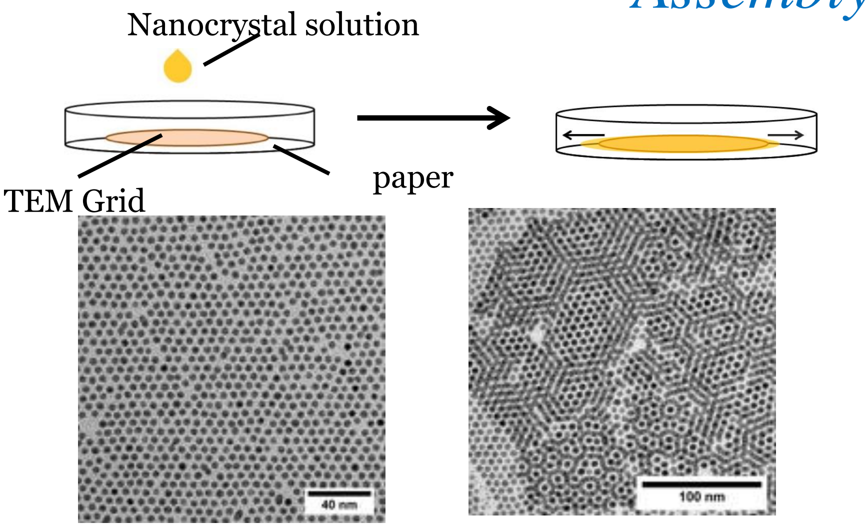


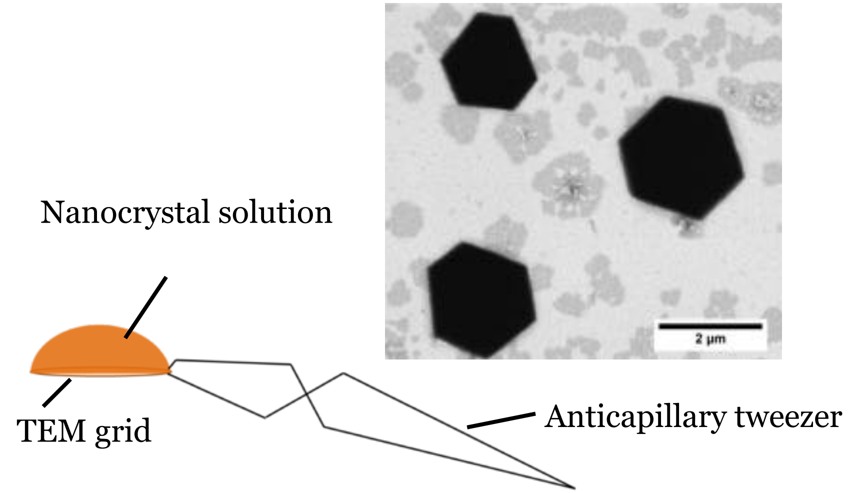


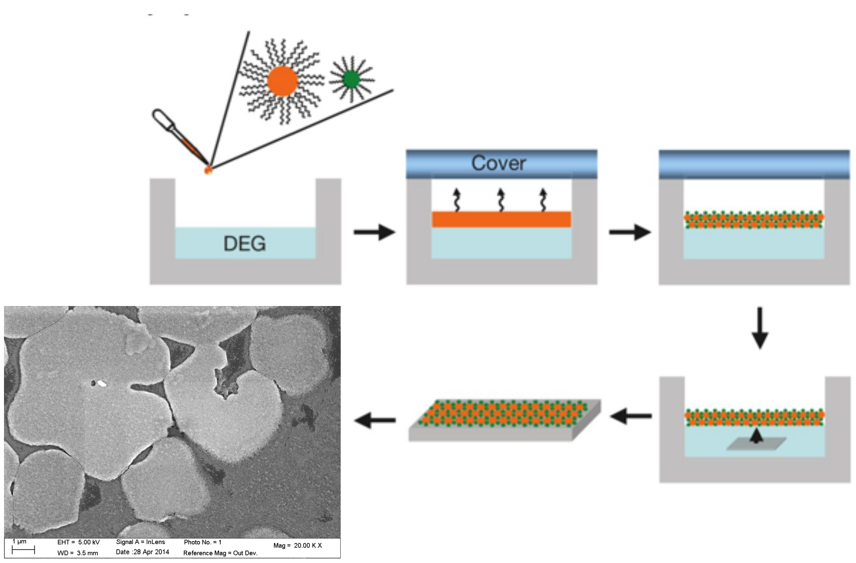


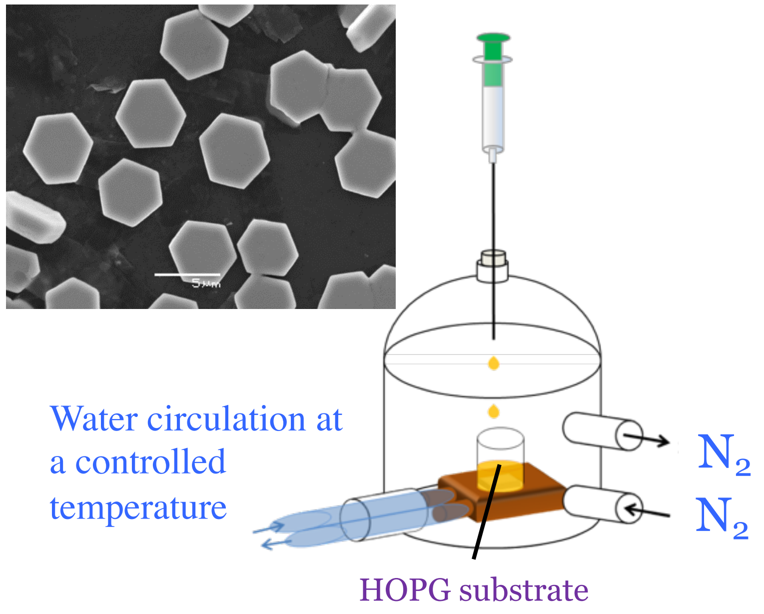


Assembly methods



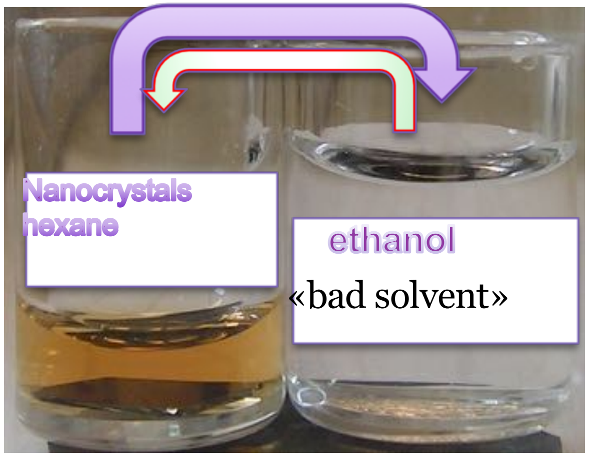






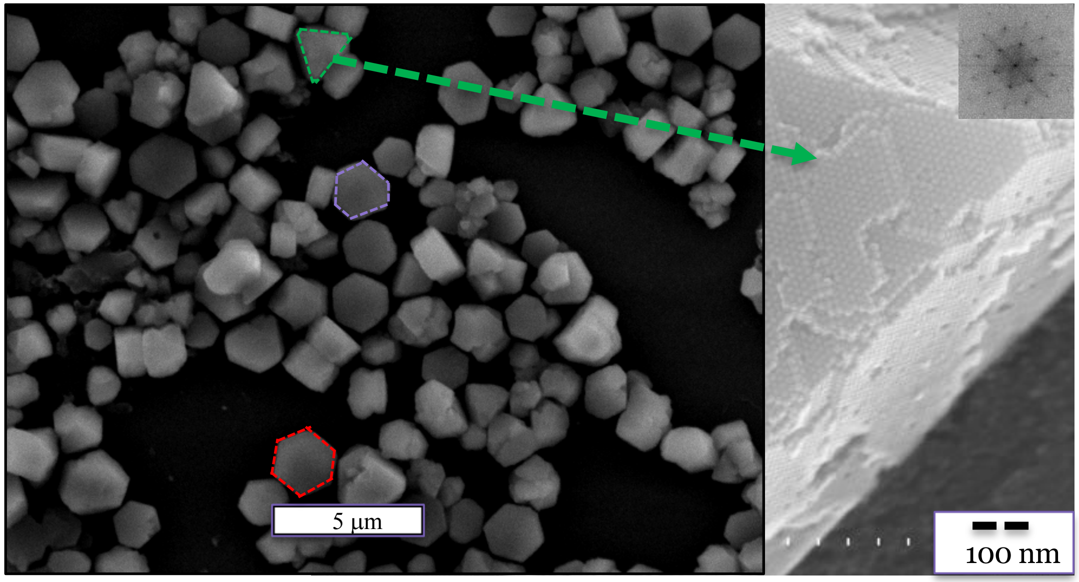
Nanoalloys for fundamental energy to application, (2013), Ed. by F. Calvo CNRS and University of Lyon, France, Elsevier,. H. Brune, A. Courty, C. Petit, V. Repain

Assembly methods



Fabrication of colloidal crystals
Made of ferrite NPs 12.7 nm in diameter
by a co-evaporation method

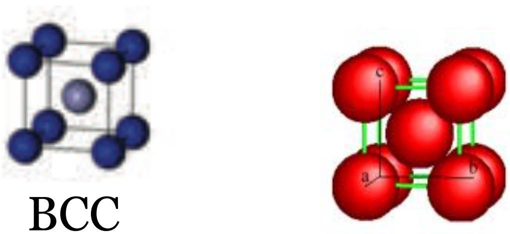
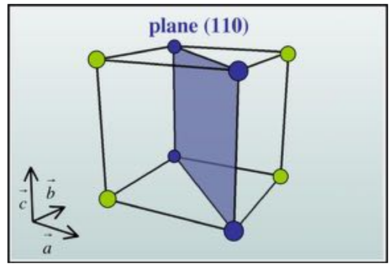
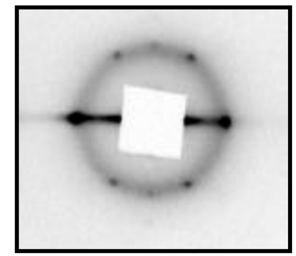
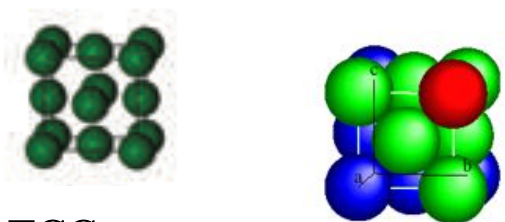
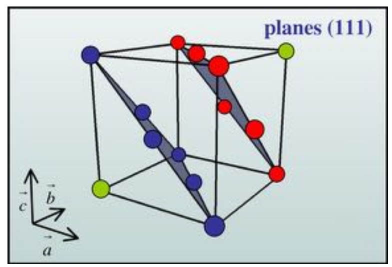
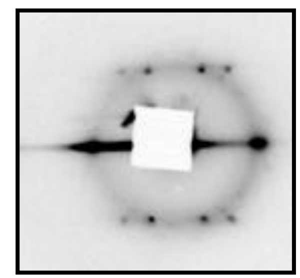
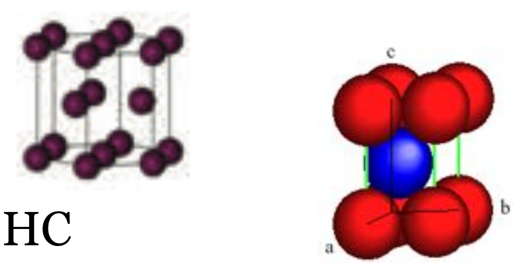
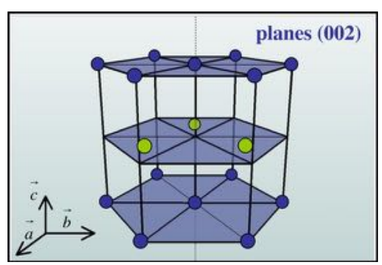
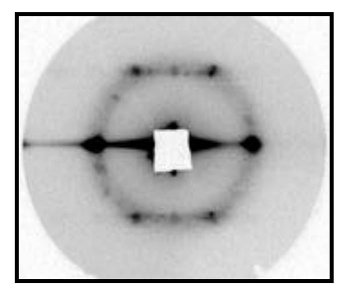
- Well-facetted fcc colloidal crystals of size reaching 2.4 µm, with various morphologies including triangles and hexagons



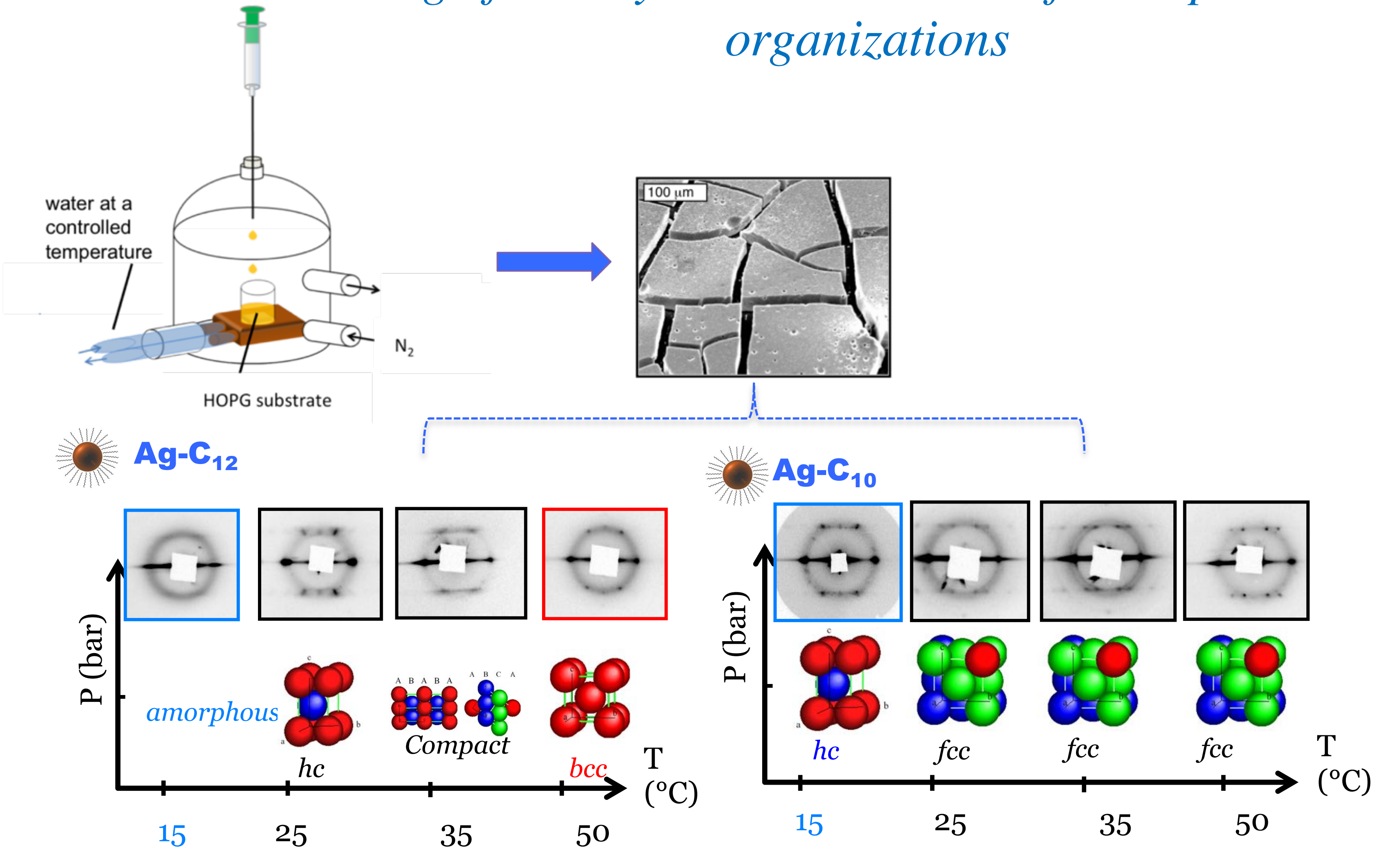
A-T Ngo, S. Costanzoa, P-A Albouy, V. Russier, S. Nakamae, J. Richardi,, I. Lisiecki Colloids and Surfaces A 560 (2019) 270-277

Crystalline structure of nanocrystal 3D organizations

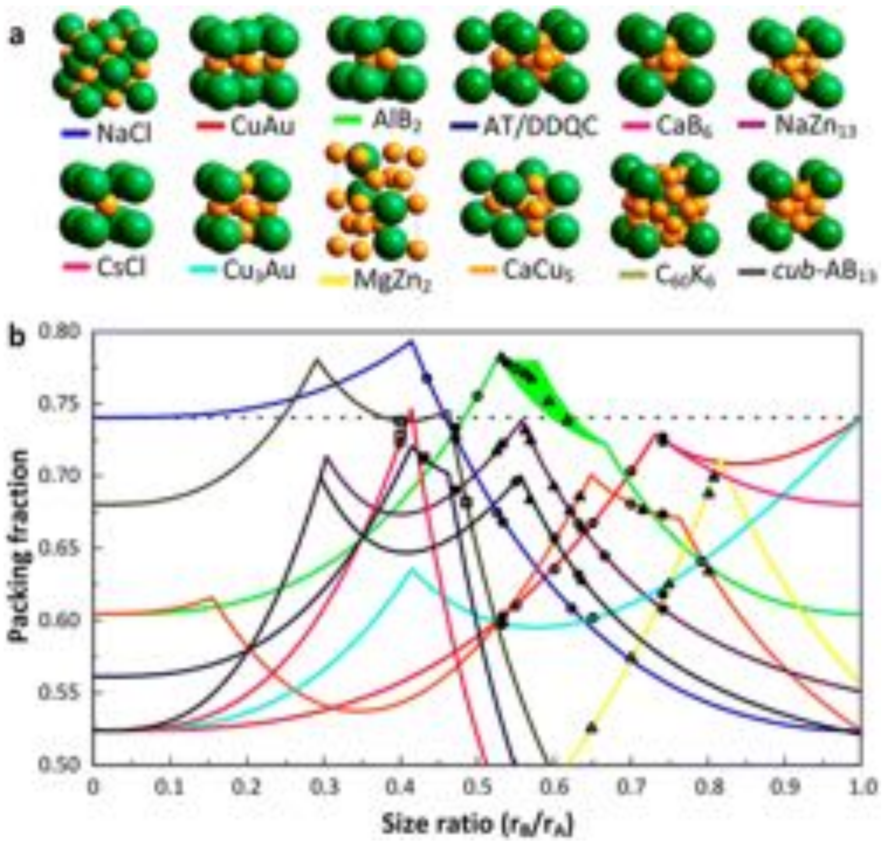
Self-assembled colloidal NPs can reprint the same structure as colloidal atoms

Typical sphere packing	In blue the planes of highest density in lattices	Typical SAXRD patterns Obtained in the case of Ag NP superlattices
 BCC		
 FCC		
 HC		

Tuning of the crystalline structure of Nanoparticle 3D organizations



*Self-Organization : periodical arrangement of nanocrystals of different nature and/or size (**Binary nanocrystals superlattices**, BNSLs)*



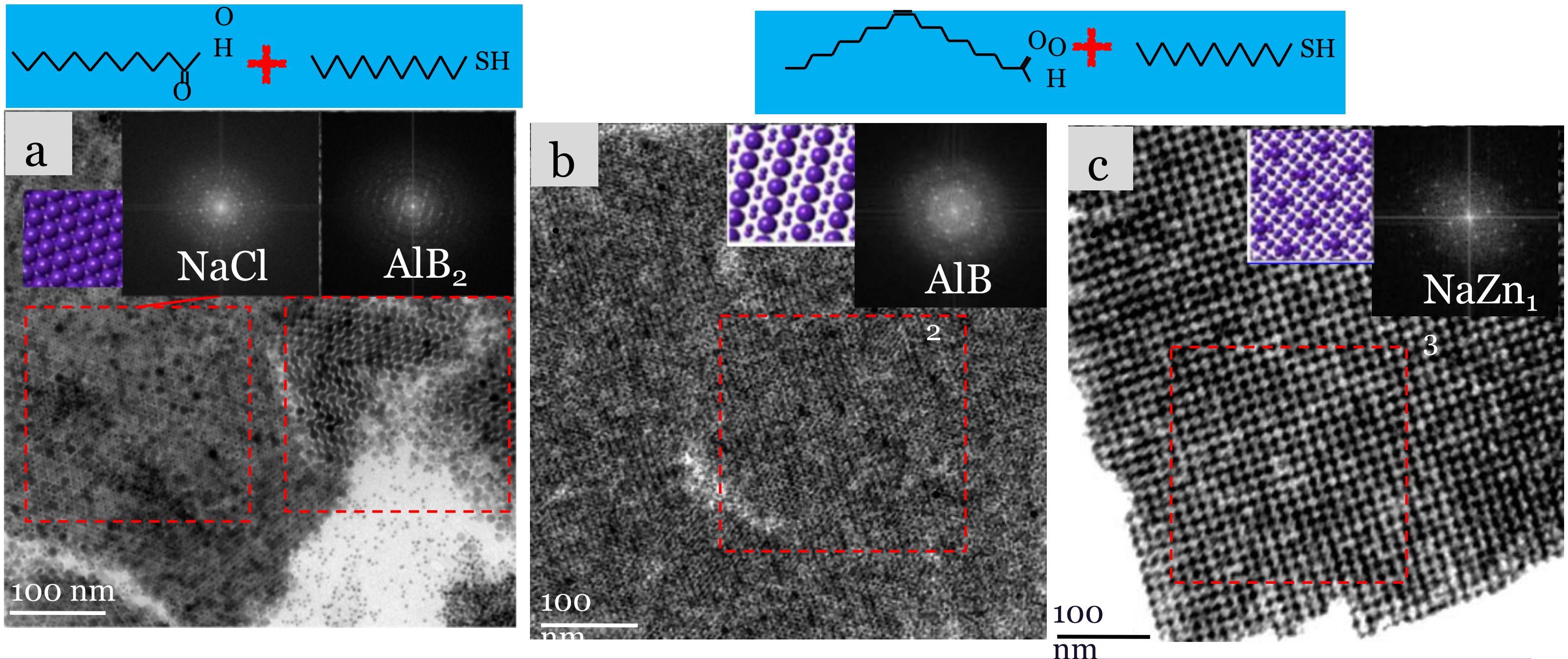
Two important parameters can determine the possibility of formation of BNSLs with A and B components differing by their size:

- The size ratio $\gamma = R_B/R_A$ with $R_A > R_B$
- The stoichiometry $x = n_B/(n_A+n_B)$.

For capped nanoparticle with length L of the hydrocarbon chain:

$\gamma_L = (R_B+L_B)/(R_A+L_A)$.

Self-organization: Control of crystalline structure with different effective size ratio (Fe_2O_3-Au)

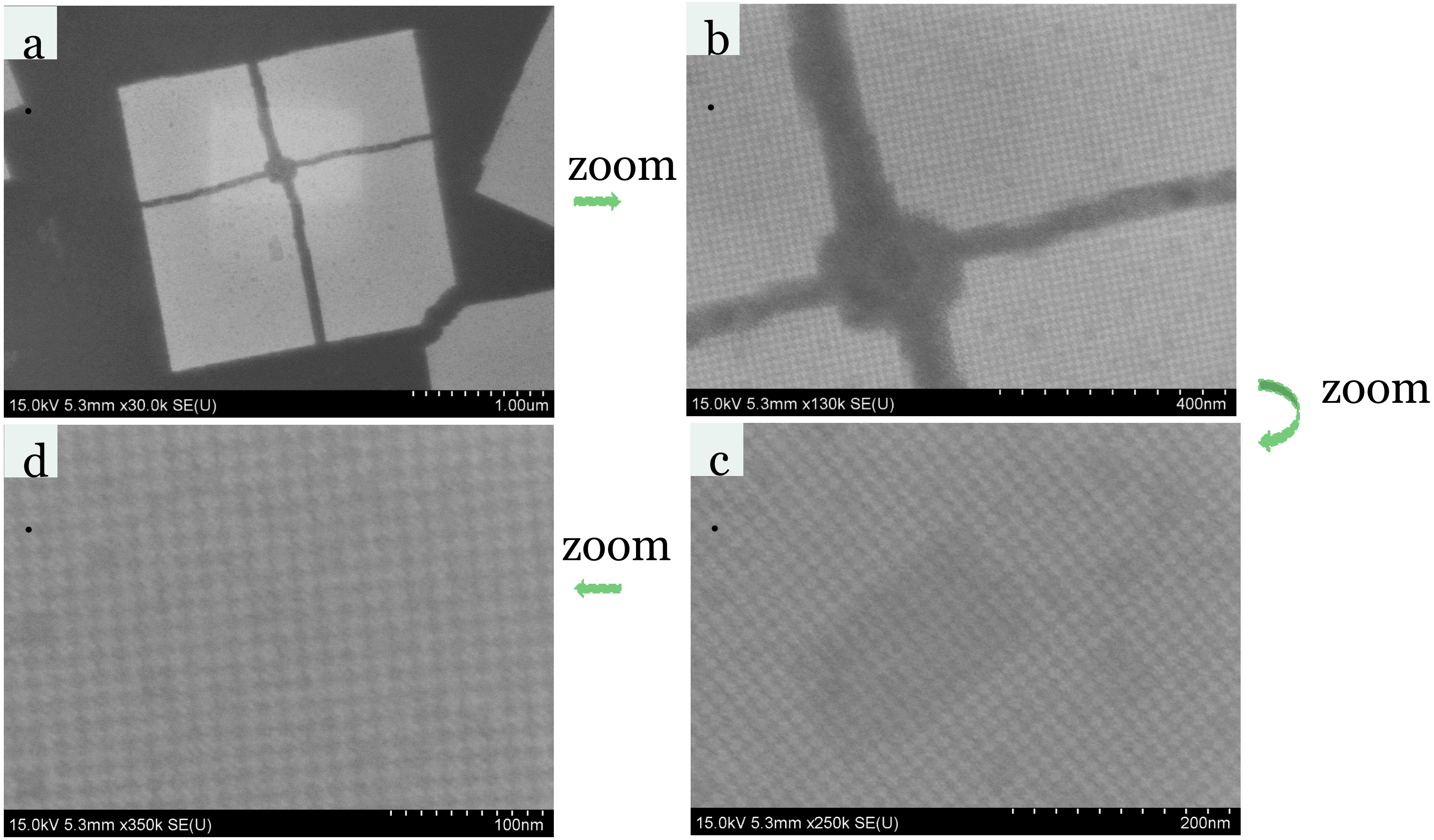


NP_1	NP_2	Concentration ($5x10^{-7}$ M)	Effective size ratio (NP_2/NP_1)	Structure
Fe_2O_3 (12.4nm, C_{12})			0.46	NaCl+AlB_2
Fe_2O_3 (11.4nm, C_{18})	Au(4.6nm, C_{12})	1:4	0.49	AlB_2
Fe_2O_3 (7.9nm, C_{18})			0.66	$NaZn_{13}$

S. Lee, A. Ngo, I.Lisiecki and A. Courty, to be published

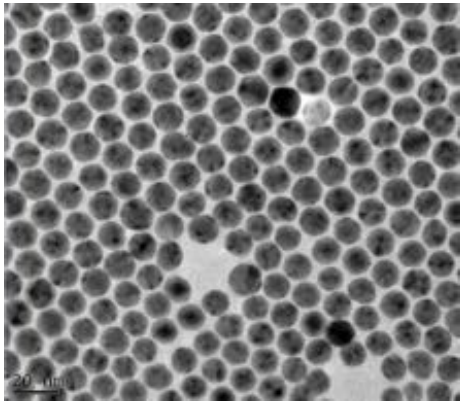
Self-organization: 3D BNSLs of $NaZn_{13}$ structure (Fe_2O_3-Au)

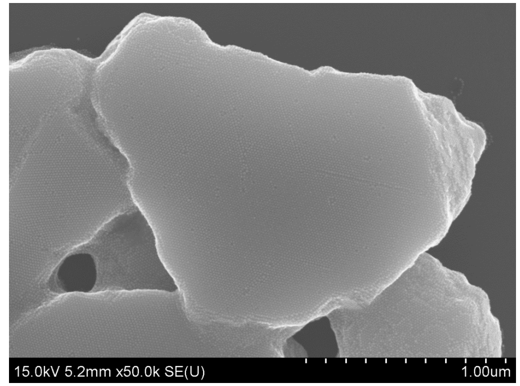
Long range organisation confirmed by HR-SEM

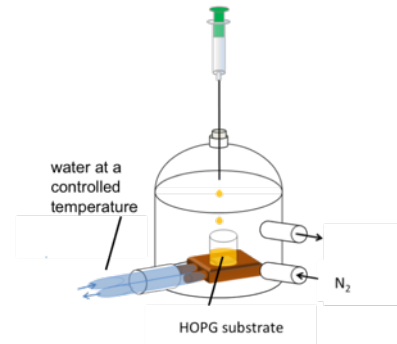


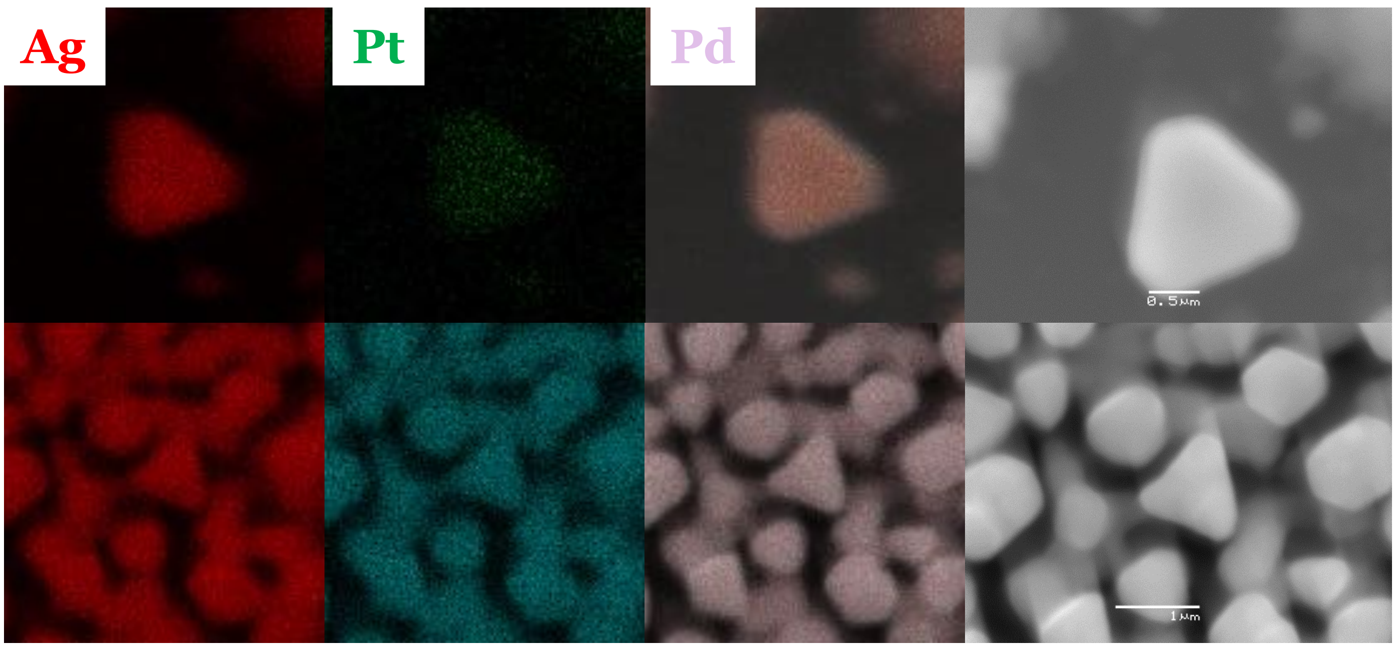
S. Lee, A. Ngo, I.Lisiecki and A. Courty, to be published

Self-organization of core-shell Ag@Pt and Ag@Pd NPs







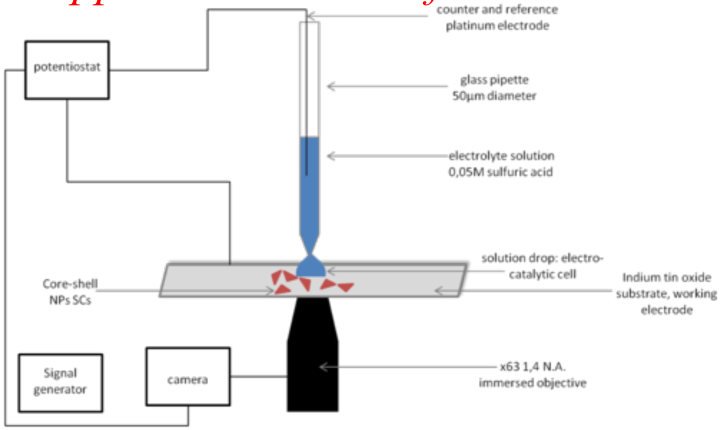


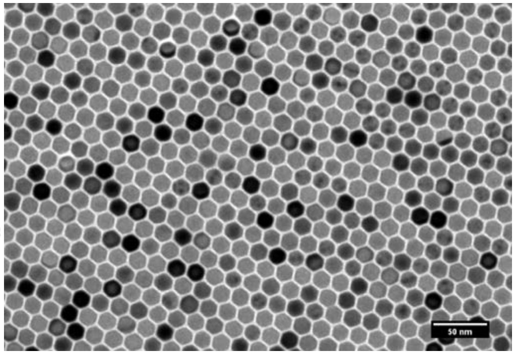
S. Hadaoui, Y. Yinan, S.Salzemann and A. Courty, to be published

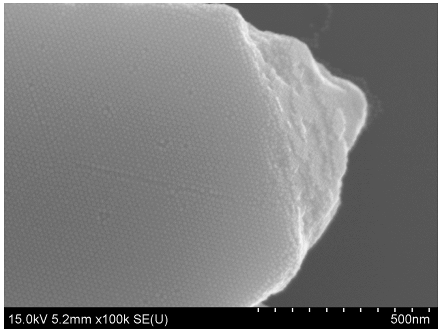
Properties

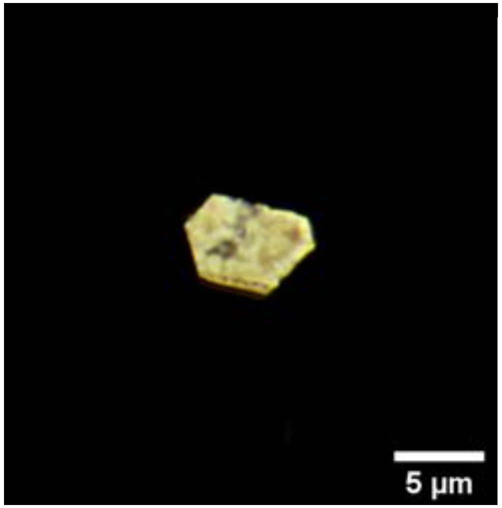
Electro-catalytic activity of core-shell Ag@Pt NP 3D organizations

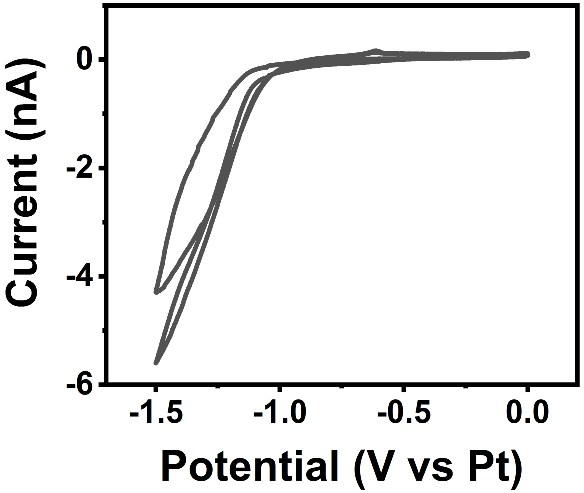
SCs appear to be active for the electro-catalytic reduction of H^+

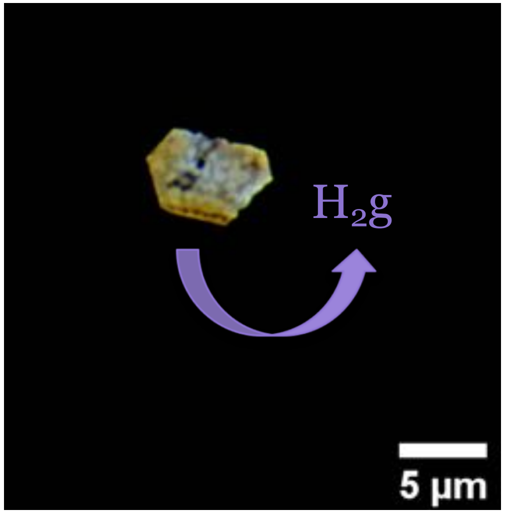








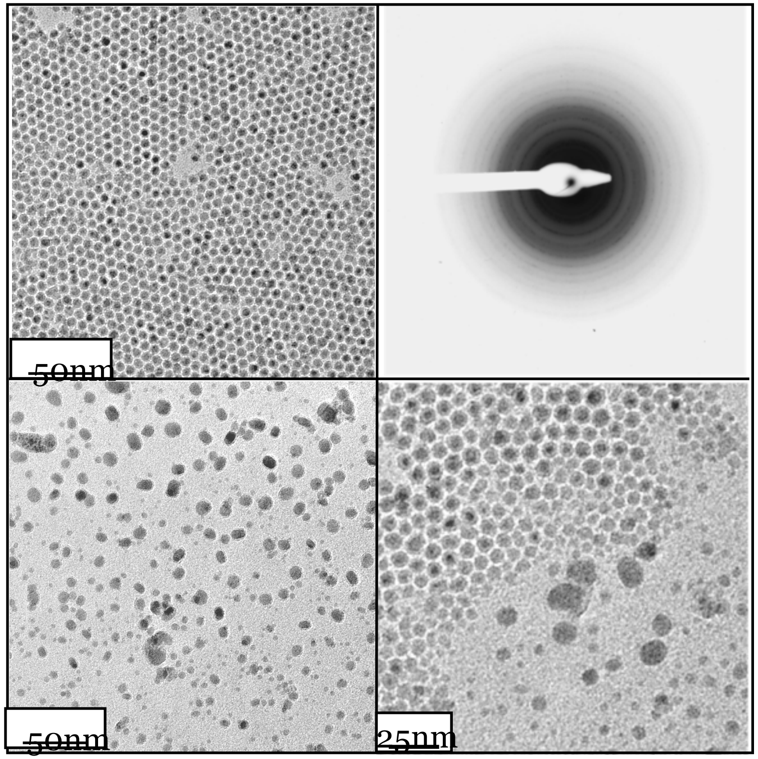


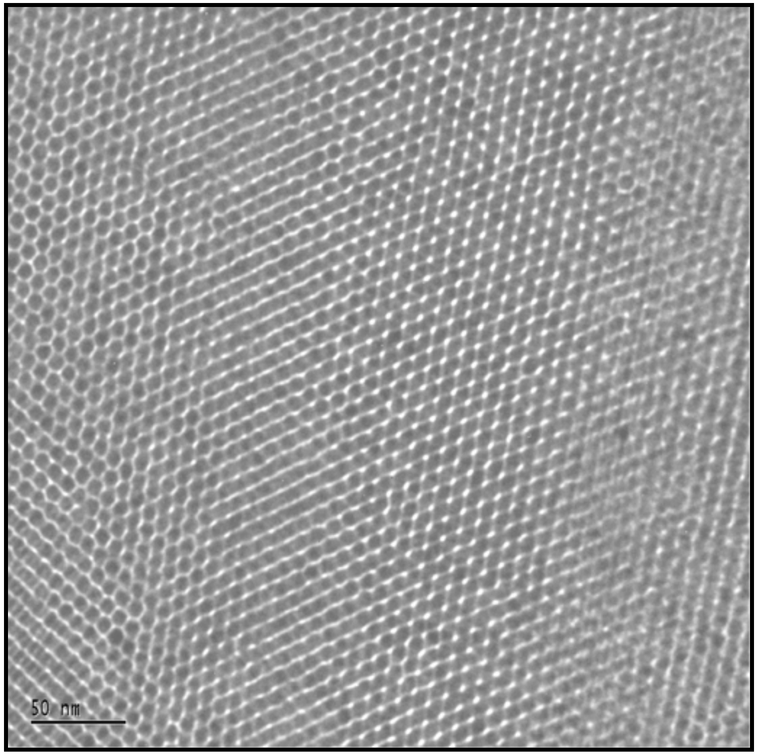


voltammetry (50mV/s) of proton reduction on Ag@Pt2 SCs.

Air annealing of cobalt nanocrystals
Effect of the organization

T_a: 300°C

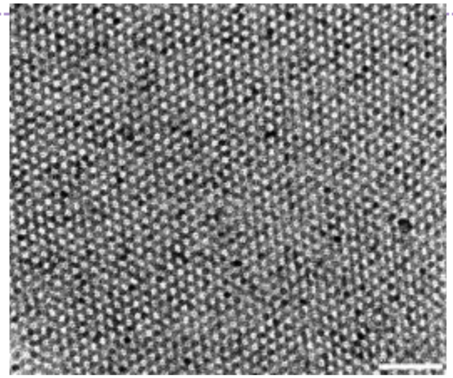


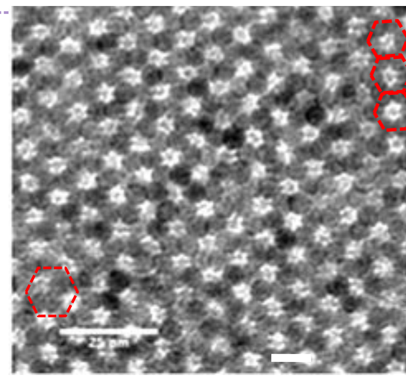


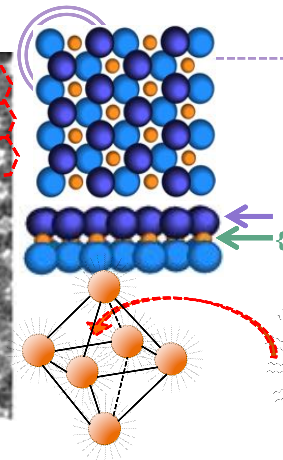
Self-oragnized nanocrystals are stable during annealing while they coalesce when isolated

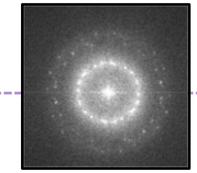
Formation of AB type binary superlattices of Fe_2O_3 NPs/ POMs

γ-Fe_2O_3@OA / Mo_{132}@DDA

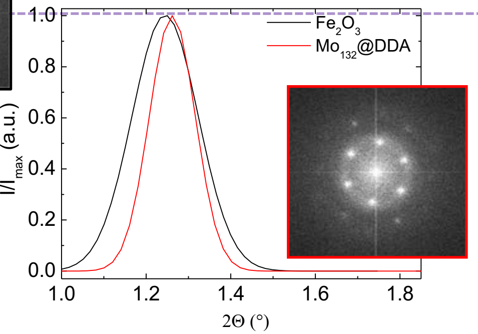








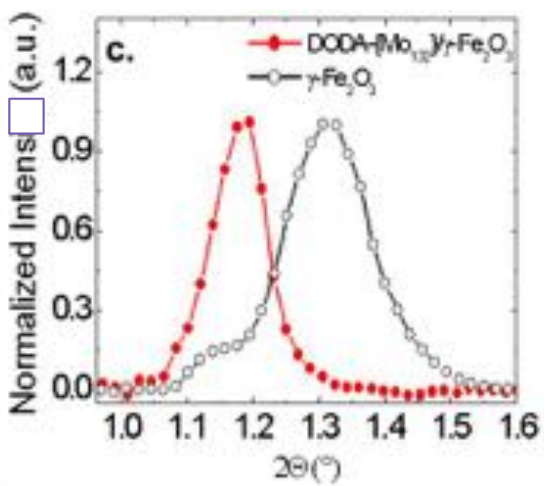
SAXS

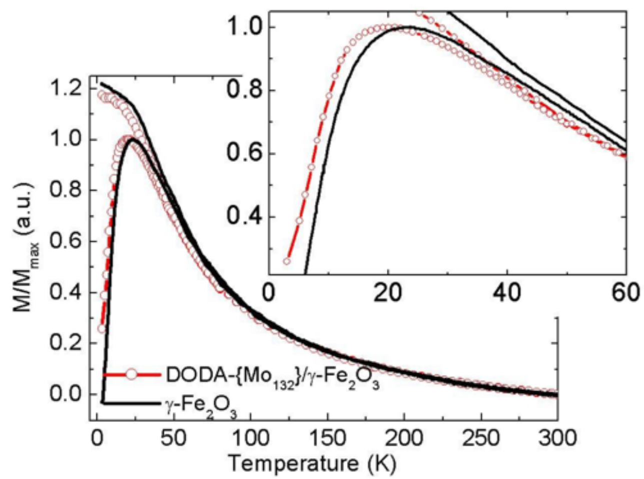


➢ *structuring effect of the POMs.*

γ-Fe_2O_3@OA / Mo_{132}@DODA

SAXS





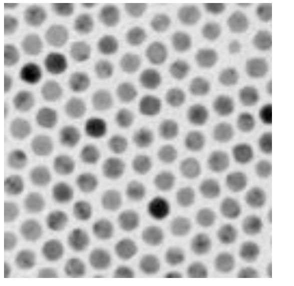
➢ *decrease of the blocking temperature of 10%. (decrease of dipolar interaction)*

Modulation of the magnetic interaction by the control of magnetic dipolar interaction in the mesostructure of the BNSL

R. Breitwieser, et al.. *Small*, Wiley-VCH Verlag, 2016, 12 (2), pp.220-228.

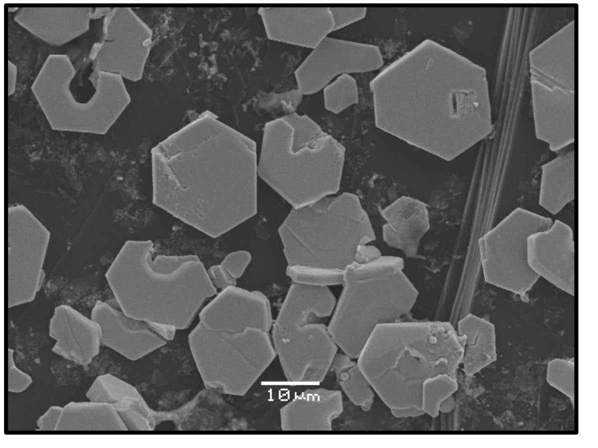
Conclusions

Colloidal chemistry is a powerful and versatile tool for synthesizing inorganic nanocrystals of controlled size, composition and shape.



Nanocrystals are elementary bricks allowing the construction of more elaborate materials by 2D and 3D self-assembly

These colloidal supercrystal have intrinsic properties that allow new physical properties to be considered



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