Now in the 64^{th} year

Atomically Precise Metal Clusters

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International Winter School on Frontiers in Materials Science, JNCASR, Bengaluru, December 4-6, 2023

Volume 91, number 6 CHEMICAL PHYSICS LETTERS 1 October 1982

Gas phase

PRODUCTION OF LARGE SODIUM CLUSTERS (Na_x, $x \leqslant 65$) BY SEEDED BEAM EXPANSIONS

Manfred M. KAPPES, Roland W. KUNZ * and Ernst SCHUMACHER
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Received 10 August 1982

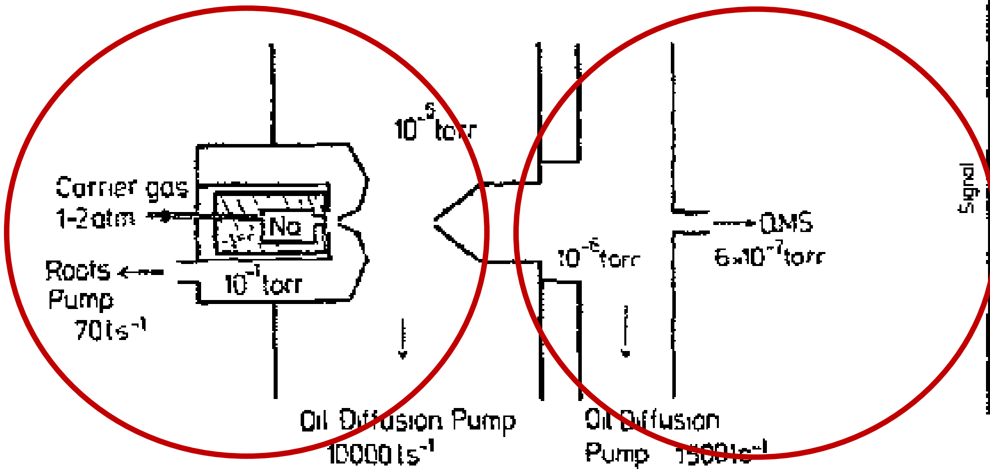


Fig. 1. Schematic of the seeded-beam apparatus.

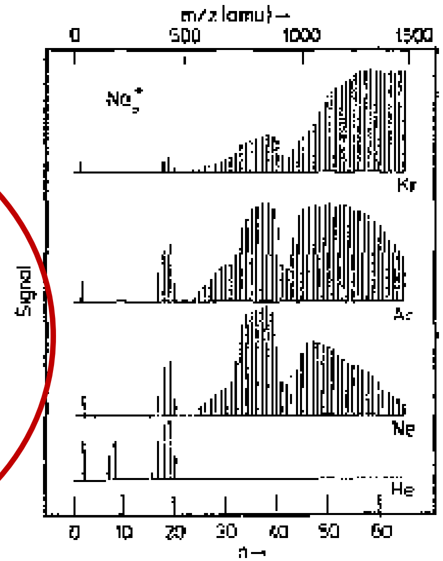
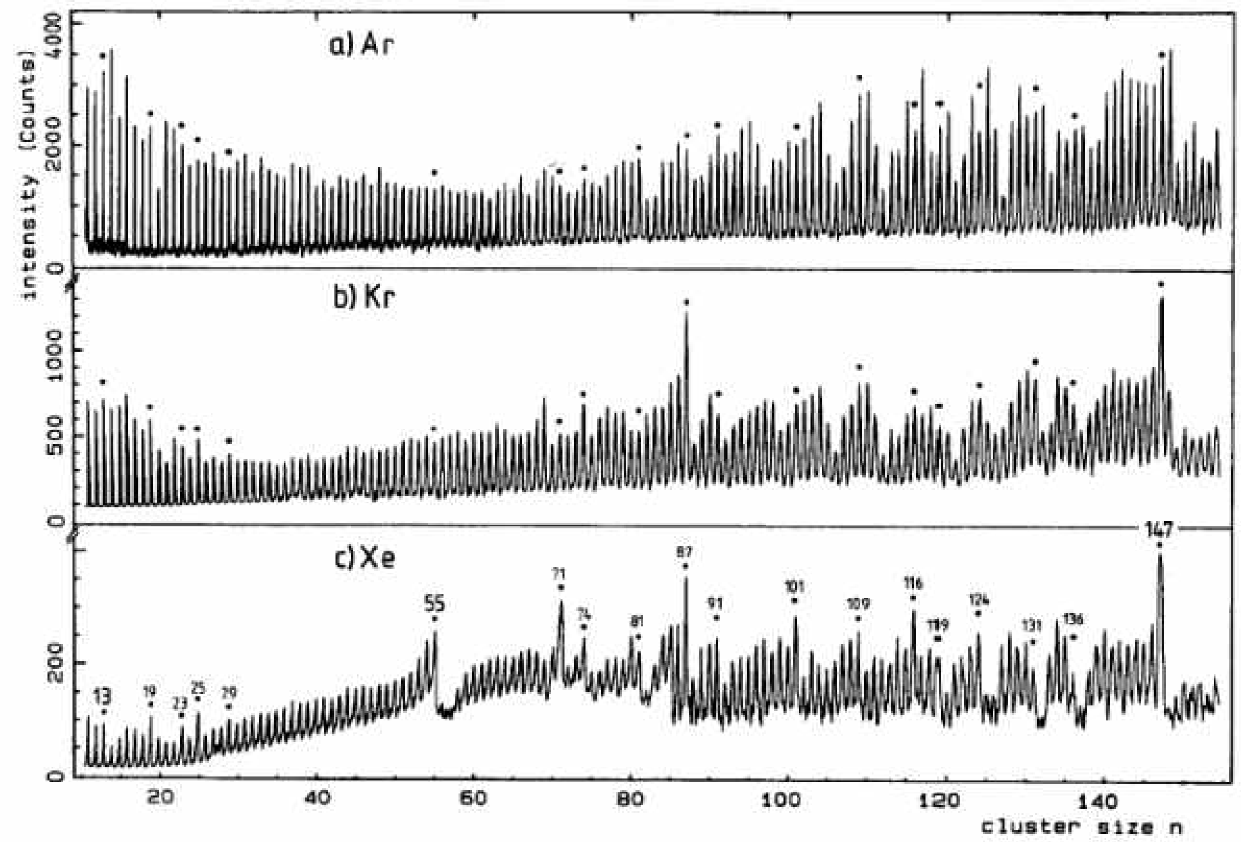


Fig. 3. Data sets showing the results of high-mass scans measured for four different backing gases. The mass spectra upon which these representations are based were obtained at a resolution ($m/\Delta m$) of 400 and a maximum signal-to-noise ratio of 20. Backing pressures were 1.3 atm for all three gases. A 1.5 mm aperture skimmer was used. With krypton, argon and neon, ions were detected up to m/z = 1495 corresponding to Na_{65}^+.

Gas phase cluster spectroscopy

Gas phase



Ti_8C_{12}
Fe, Al clusters

Mass spectra of positively charged Ar, Kr, Xe clusters, W. Miehle, O. Kandler, T. Leisner, and O. Echt. (**1989**) *J. Chem. Phys.*, 91, 5940.

Gas phase

International Journal of Mass Spectrometry and Ion Processes, 74 (1986) 33–41 33
Elsevier Science Publishers B.V., Amsterdam – Printed in The Netherlands

MASS DISTRIBUTIONS OF NEGATIVE CLUSTER IONS OF COPPER, SILVER, AND GOLD

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(First received 6 May 1986; in final form 27 August 1986)

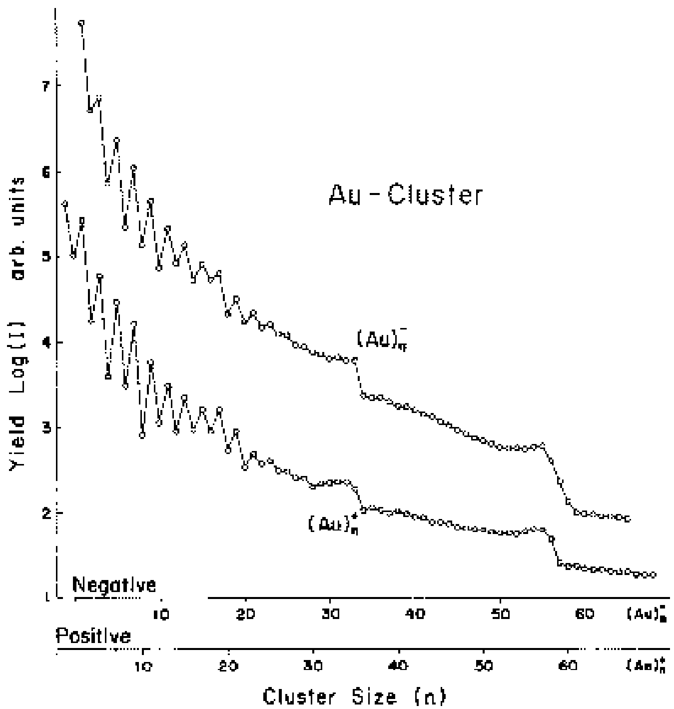


Fig. 2. Size distributions of gold clusters, $(Au)_n^-$ (upper curve) and $(Au)_n^+$ (lower curve), plotted on a logarithmic scale.

Gas phase and solution phase

Magic clusters

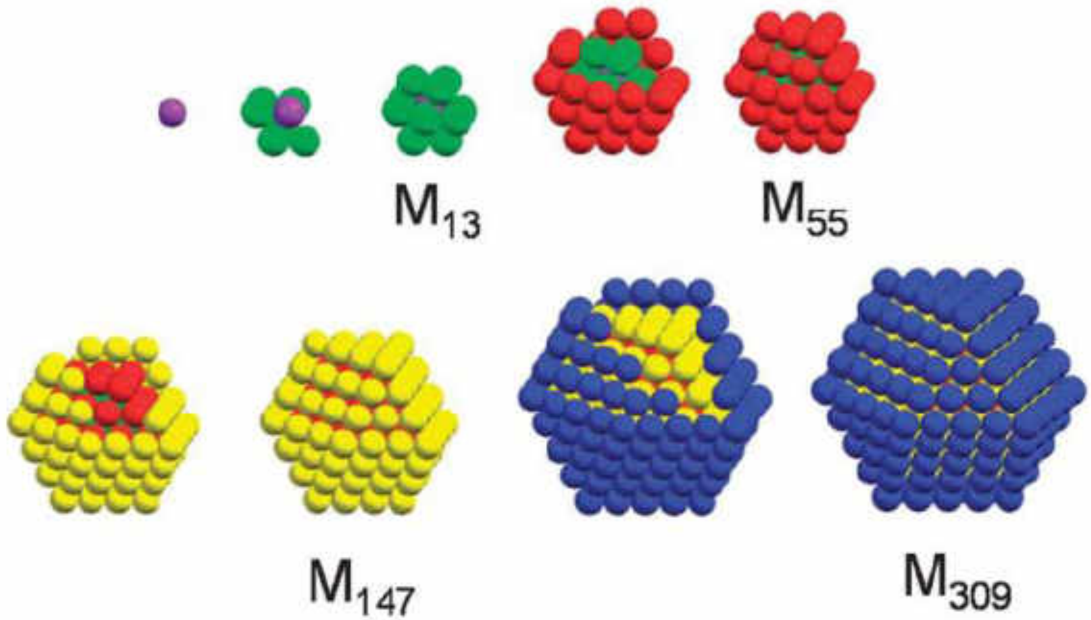


Fig. 1 Organization of full-shell clusters: a first single atom (purple) is surrounded by 12 others (green) to give a one-shell cluster M_{13}. 42 atoms (red) can be densely packed on the 12 green atoms ending with the M_{55} two-shell cluster, followed by 92 atoms (yellow) and 162 atoms (blue) to give M_{147} and M_{309}, respectively.

From Gunter Schmidt, *Chem. Soc. Rev.* **2008**, *37*, 1909–1930

C_{60}: Buckminsterfullerene

H. W. Kroto*, J. R. Heath, S. C. O'Brien, R. F. Curl & R. E. Smalley

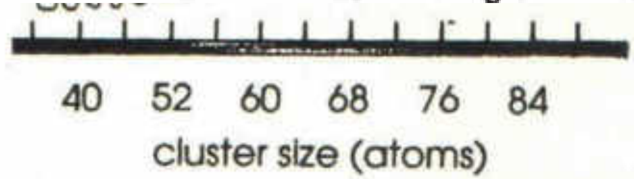
Rice Quantum Institute and Departments of Chemistry and Electrical Engineering, Rice University, Houston, Texas 77251, USA

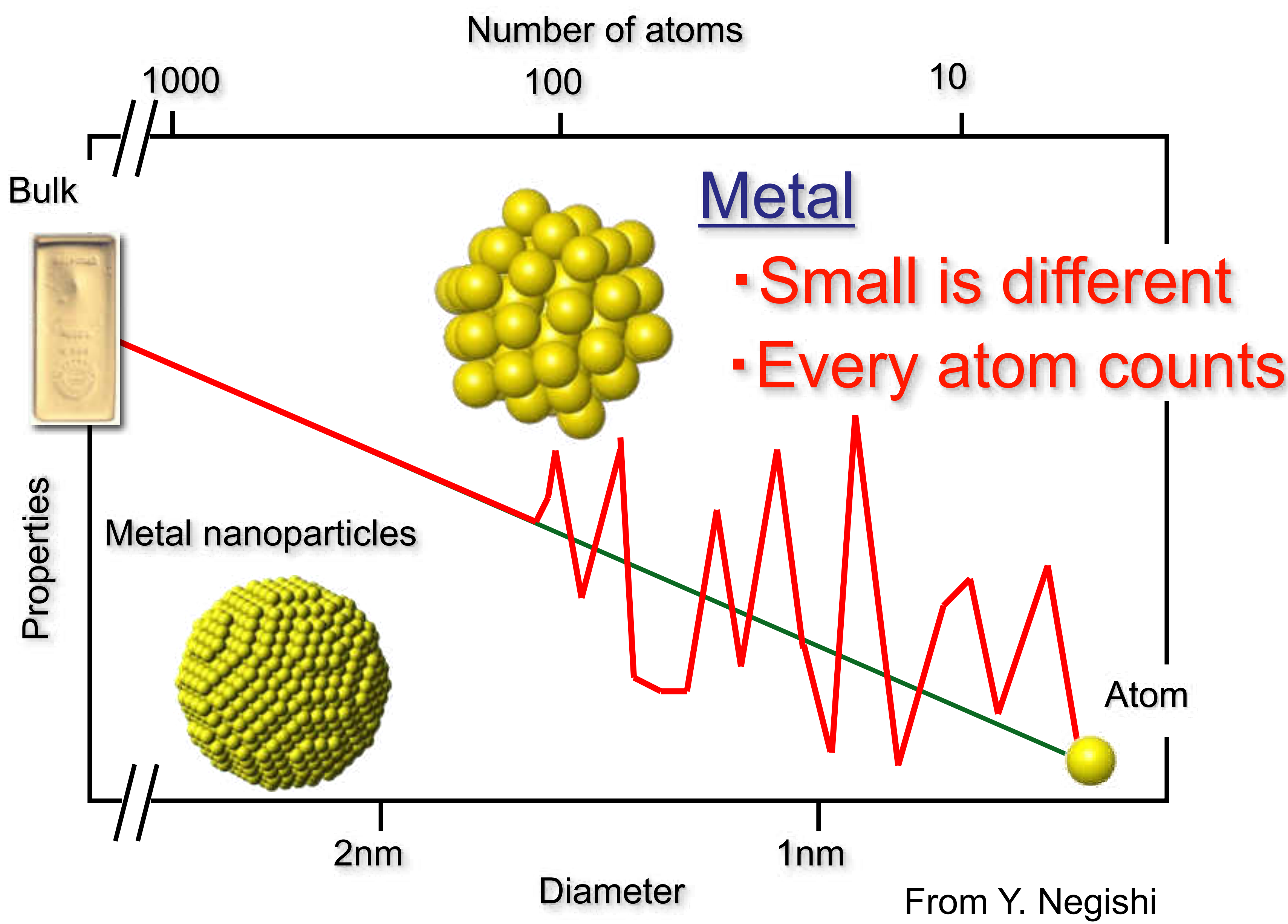
Fig. 1 A football (in the United States, a soccerball) on Texas grass. The C_{60} molecule featured in this letter is suggested to have the truncated icosahedral structure formed by replacing each vertex on the seams of such a ball by a carbon atom.

During experiments aimed at understanding the mechanisms by which long-chain carbon molecules are formed in interstellar space and circumstellar shells[1], graphite has been vaporized by laser irradiation, producing a remarkably stable cluster consisting of 60 carbon atoms. Concerning the question of what kind of 60-carbon atom structure might give rise to a superstable species, we suggest a truncated icosahedron, a polygon with 60 vertices and 32 faces, 12 of which are pentagonal and 20 hexagonal. This object is commonly encountered as the football shown in Fig. 1. The C_{60} molecule which results when a carbon atom is placed at each vertex of this structure has all valences satisfied by two single bonds and one double bond, has many resonance structures, and appears to be aromatic.

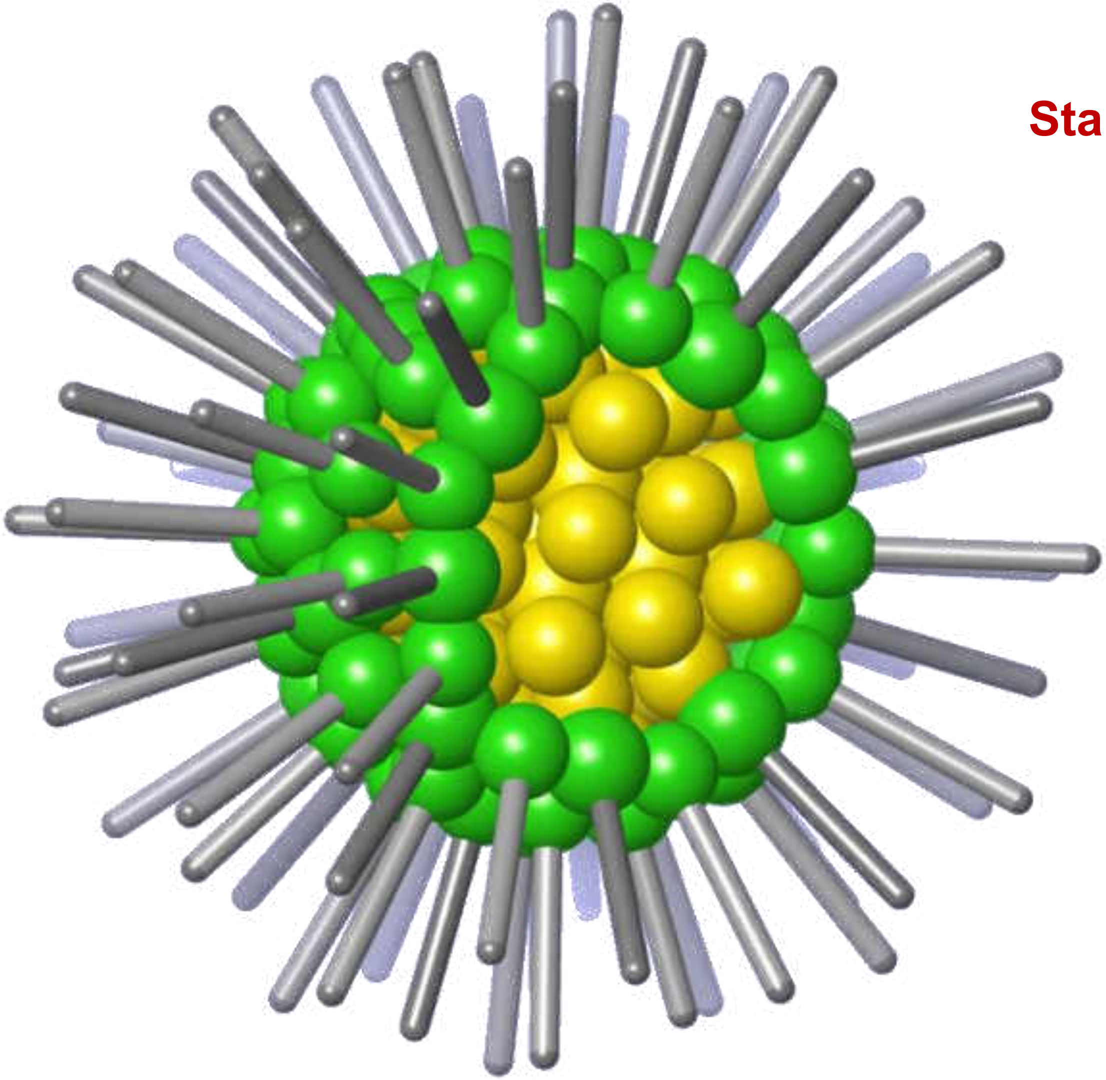
graphite fused six-membered ring structure. We believe that the distribution in Fig. 3c is fairly representative of the nascent distribution of larger ring fragments. When these hot ring clusters are left in contact with high-density helium, the clusters equilibrate by two- and three-body collisions towards the most stable species, which appears to be a unique cluster containing 60 atoms.

When one thinks in terms of the many fused-ring isomers with unsatisfied valences at the edges that would naturally arise





Number of atoms
1000
100
10
Bulk
Metal
・Small is different
・Every atom counts
Properties
Metal nanoparticles
Atom
2nm
1nm
Diameter
From Y. Negishi



Stable clusters

From Y. Negishi


How do we know that they exist?

Table 1 Landmark events in the history of mass spectrometry and their importance in enabling the characterization of materials

Progress in instrumentation	Systems studied by MS		Resolution[6,134*]	Mass range (m/z)[134,*]
1912 Measurement of m/z values by Thomson[3] 1918 Electron ionization[135] 1936-37 Secondary ion MS[136]	Isotopes of elements Atomic weights using MS[6]	early studies by MS	100 Aston (130)[5] 1,000	100 Aston (~100)[5] 1,000
1946 Time of flight[139] 1952 Double-focussing instruments[140] 1955 Advanced TOF[141] 1956 GC–MS,[7,8] high-resolution MS[142] 1953-58 Quadrupole analyzers[143] 1962 Ion mobility[144] 1966 Chemical ionization[145] 1967 Tandem MS[146]	1940s Organic mass spectrometry, Mixture of organic analytes could be separated by GC–MS[6]	Analysis of organic molecules	TOF (4000-5000 at m/z ~100)[137] 10,000 W geometry ortho-TOF (70,000 at m/z 316)[147]	Magnetic sector (~2000)[138] 10,000 FTICR (~29,000)[148]
1968 Electrospray ionization (ESI)[149] 1974 Fourier transform (FT) Ion cyclotron resonance[13], Atmospheric pressure chemical ionization[150] 1975 Surface-induced dissociation[151] 1978 Triple quadruple[152] 1981 Fast atom bombardment MS[153] 1987 MALDI[10,154] 1999 Orbitrap[14] 2004 Desorption electrospray ionization[155]	1980s high molecular-weight polymers, peptides, proteins, nucleic acids, ESI for macromolecules[6] 1996 Analysis of intact live viruses[156]	MS in proteomics and living cells	1,00,000 Orbitrap (6,00,000 at m/z 195)[15]	1,00,000 MALDI TOF (~2,00,000)[158]
	1985 Discovery of fullerenes by laser-induced vaporization[38] 1996 LDI for characterization of thiol-protected clusters[47] 2008 MS of intact $Au_{25}(PET)_{18}$ clusters[52] 2018 MS of $Au_{\sim 2000}$ NPs[124]	Analysis of materials with MS	10,00,000 FTICR (20,00,000 at m/z 66,000)[157]	10,00,000 Cryo MALDI TOF (~20,00,000)[159]

Charge detection MS

*Does not strictly correspond to the time evolution presented in the left column

Chakraborty and Pradeep *NPG Asia Materials* (2019) 11:48
https://doi.org/10.1038/s41427-019-0149-3

Synthetic methods

J. CHEM. SOC., CHEM. COMMUN., 1994

Synthesis of Thiol-derivatised Gold Nanoparticles in a Two-phase Liquid–Liquid System

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Department of Chemistry, The University of Liverpool, PO Box 147, Liverpool, UK L69 3BX

Using two-phase (water–toluene) reduction of $AuCl_4^-$ by sodium borohydride in the presence of an alkanethiol, solutions of 1–3 nm gold particles bearing a surface coating of thiol have been prepared and characterised; this novel material can be handled as a simple chemical compound.

Colloidal solutions of metals have been known for a long time[1] and a large variety of preparative techniques is now available.[2,3] Depending on the preparative conditions, the particles have a tendency to agglomerate slowly, eventually lose their disperse character and flocculate. The removal of the solvent generally leads to the complete loss of the ability to reform a colloidal solution. Preparation of colloidal metals in a two-phase system was introduced by Faraday,[4] who reduced an aqueous gold salt with phosphorus in carbon disulfide and obtained a ruby coloured aqueous solution of dispersed gold particles. Combining this two phase approach with the more recent techniques of ion extraction and monolayer self-assembly with alkane thiols,[5] a one-step method for the preparation of an unusual new metallic material of derivatised nanometre-sized gold particles is described.

The strategy followed consisted in growing the metallic clusters with the simultaneous attachment of self-assembled thiol monolayers on the growing nuclei. In order to allow the surface reaction to take place during metal nucleation and growth, the particles were grown in a two-phase system. Two-phase redox reactions can be carried out by an appropriate choice of redox reagents present in the adjoining phases.[6] In the present case, $AuCl_4^-$ was transferred from aqueous solution to toluene using tetraoctylammonium bromide as the phase-transfer reagent and reduced with aqueous sodium borohydride in the presence of dodecanethiol ($C_{12}H_{25}SH$). On addition of the reducing agent, the organic phase changes colour from orange to deep brown within a few seconds. The overall reaction is summarized by eqns. (1) and (2), where the

$$AuCl_4^-(aq) + N(C_8H_{17})_4^+(C_6H_5Me) \rightarrow N(C_8H_{17})_4^+AuCl_4^-(C_6H_5Me) \quad (1)$$

$$mAuCl_4^-(C_6H_5Me) + nC_{12}H_{25}SH(C_6H_5Me) + 3me^- \rightarrow 4mCl^-(aq) + (Au_m)(C_{12}H_{25}SH)_n(C_6H_5Me) \quad (2)$$

source of electrons is BH_4^-. The conditions of the reaction determine the ratio of thiol to gold, *i.e.* the ratio *n/m*. The preparation technique was as follows. An aqueous solution of hydrogen tetrachloroaurate (30 ml, 30 mmol dm^{-3}) was mixed with a solution of tetraoctylammonium bromide in toluene (80 ml, 50 mmol dm^{-3}). The two phase mixture was vigorously stirred until all the tetrachloroaurate was transferred into

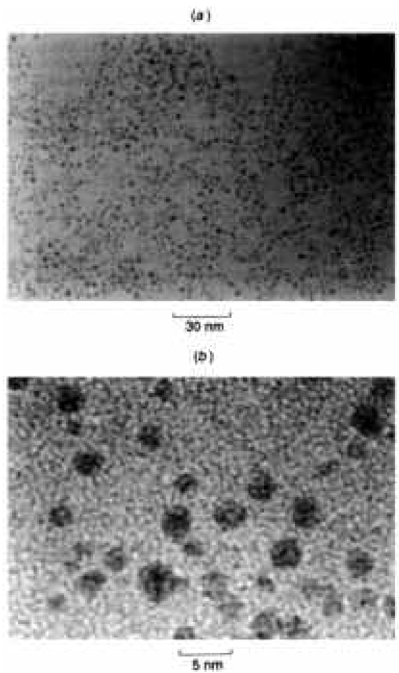


Fig. 1 TEM pictures of the thiol derivatised gold nanoparticles at (a) low and (b) high magnification

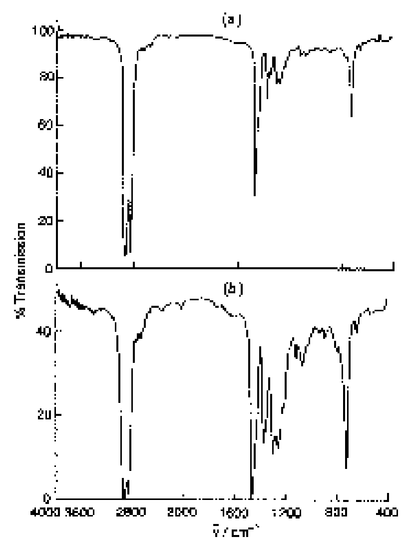


Fig. 2 IR spectra of (a) dodecanethiol and (b) nanoparticles prepared in the present work. The particles were deposited on an NaCl disc by evaporation of a drop of a toluene solution.

Observing such clusters

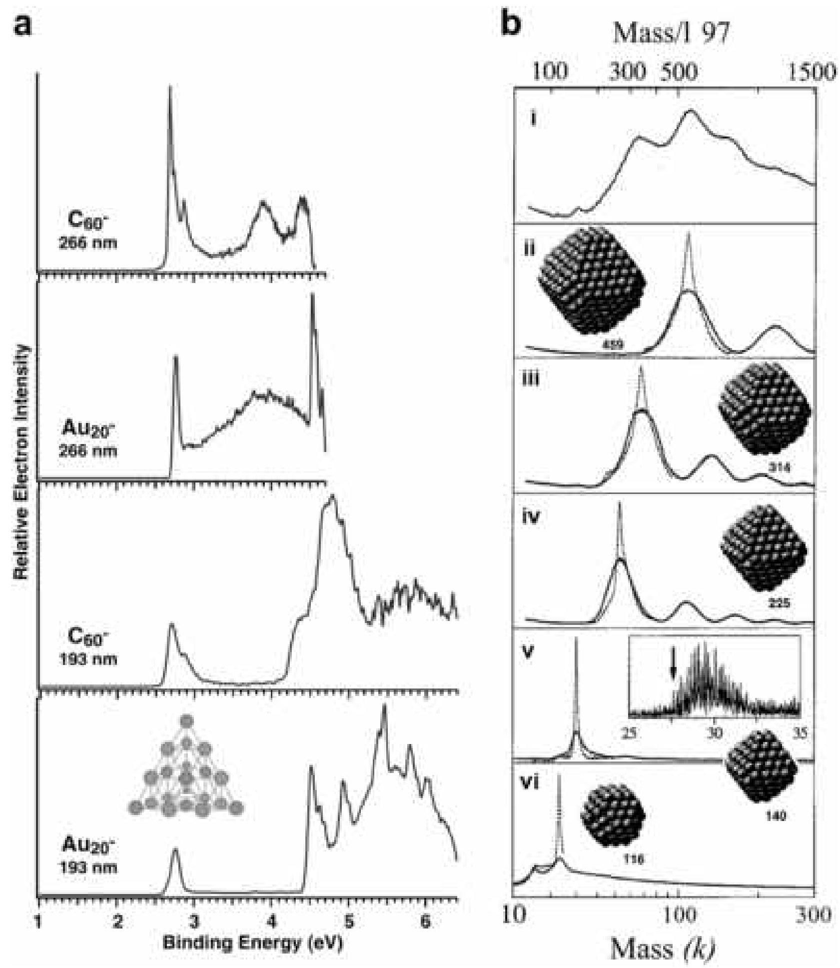
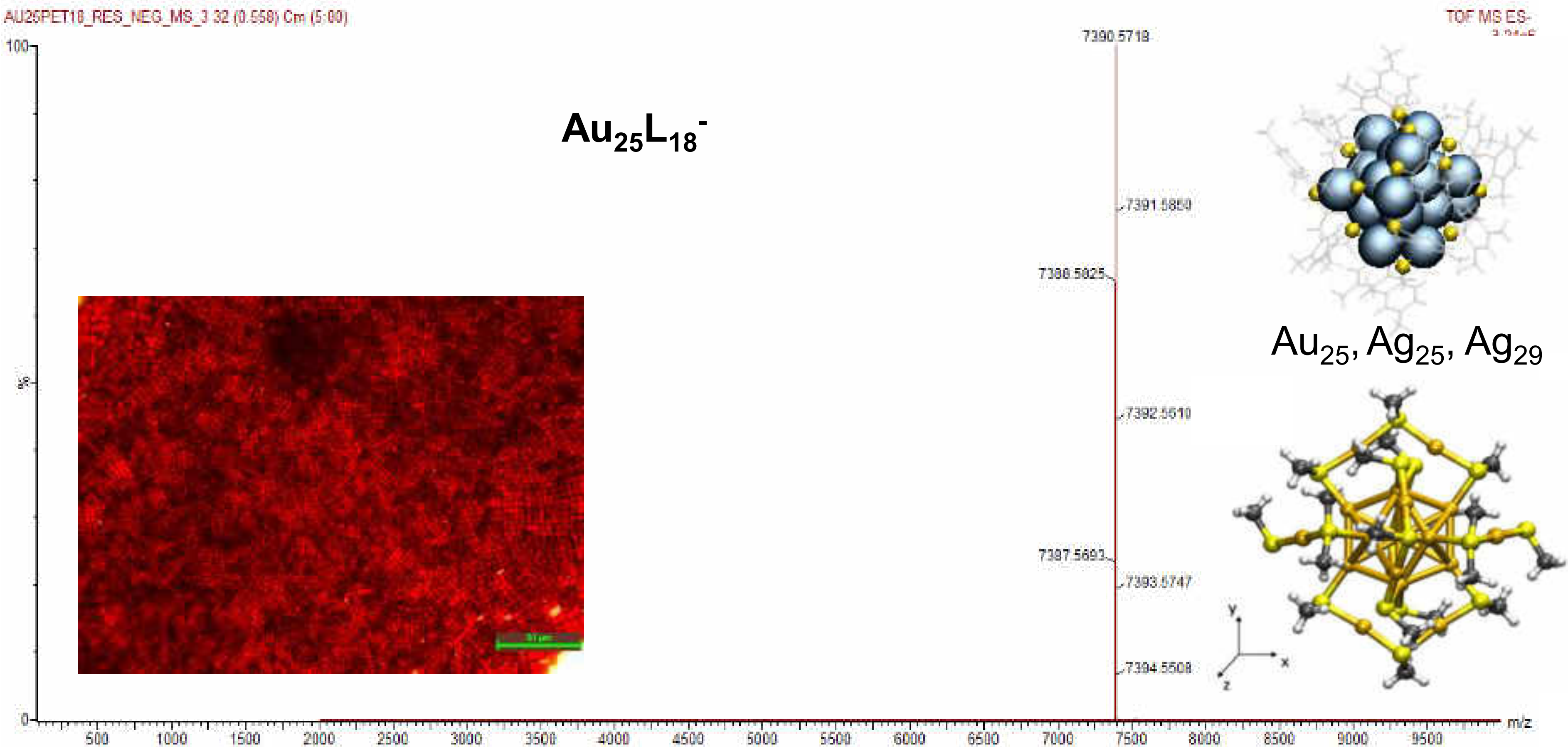


Fig. 2 Early stages of MS of noble metal clusters. a Photoelectron spectra of Au_{20}^- cluster compared with C_{60}^- at 193 nm and 266 nm[45]. (Copyright© 2003 the American Association for the Advancement of Science) **b** Mass spectra obtained by laser desorption ionization of dodecanethiol thiol-protected gold clusters, (i) crude mixture of clusters and (ii–vi) separated fractions[47]. (Copyright© 1996 John Wiley and Sons)

Chakraborty and Pradeep *NPG Asia Materials* (2019) 11:48
https://doi.org/10.1038/s41427-019-0149-3

Atomically precise metal clusters as materials

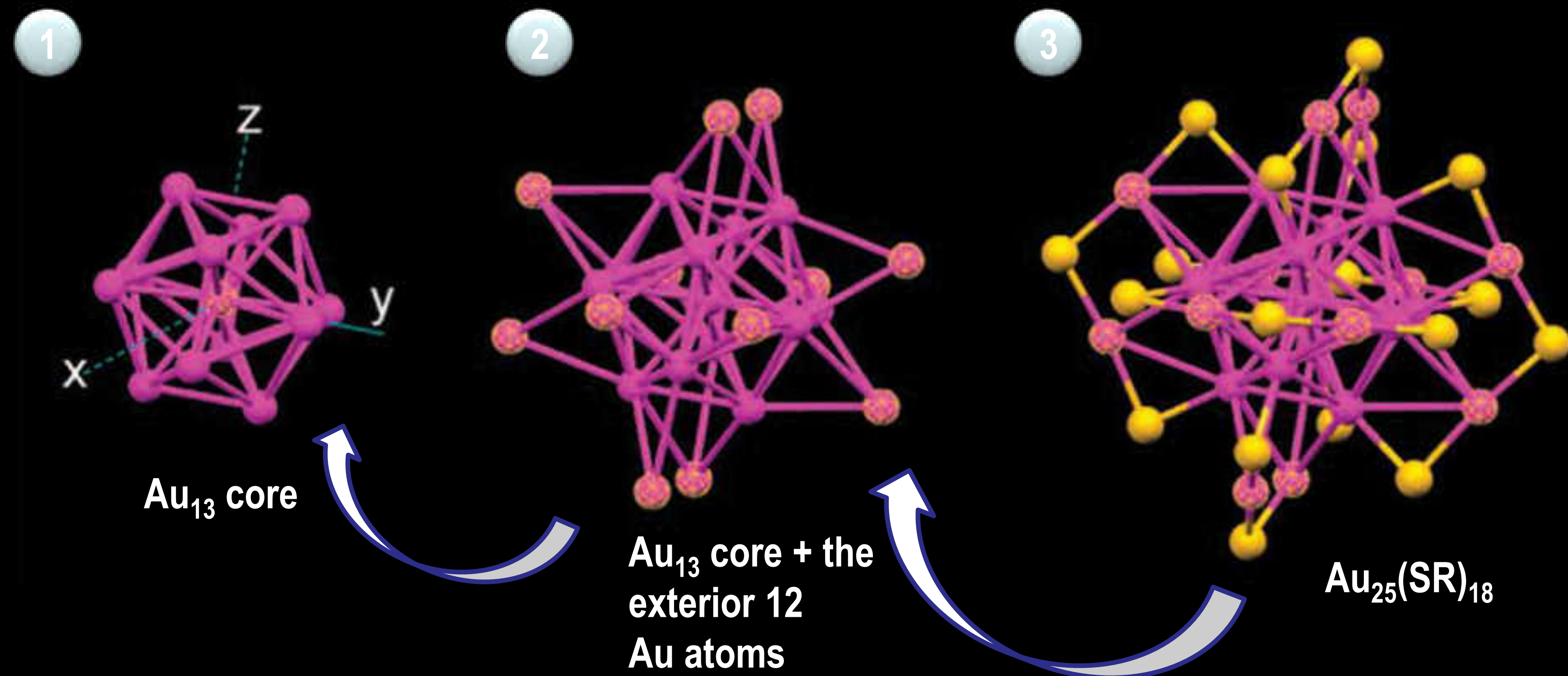


T. Pradeep et. al. *Acc. Chem. Res.* 2018; 2019.

Au
S

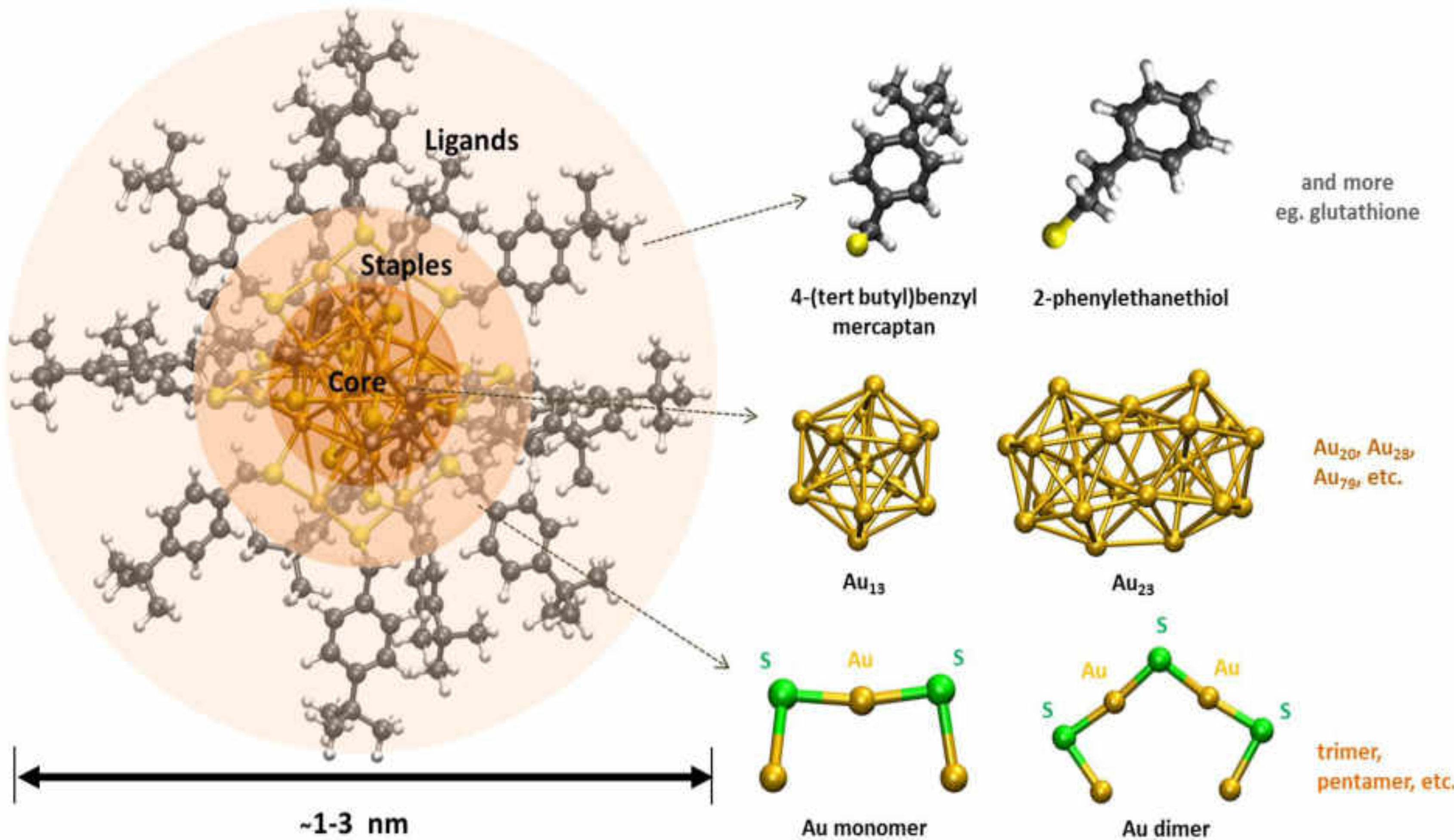
Structures

$Au_{25}(SR)_{18}$



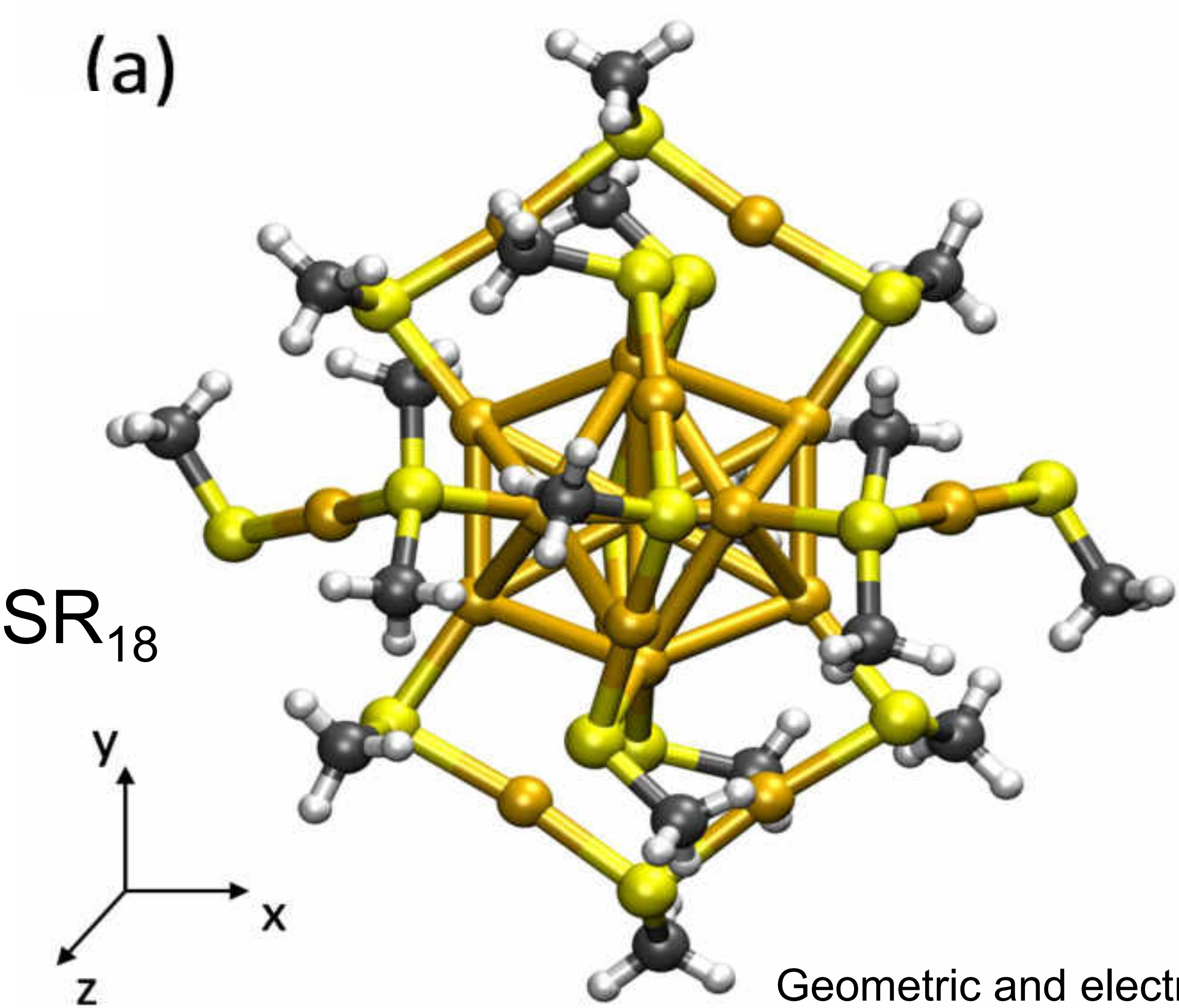
>150 such clusters

Zhu, M.; Aikens, C. M.; Hollander, F. J.; Schatz, G. C.; Jin, R. *J. Am. Chem. Soc.* **2008**, *130*, 5883-5885.

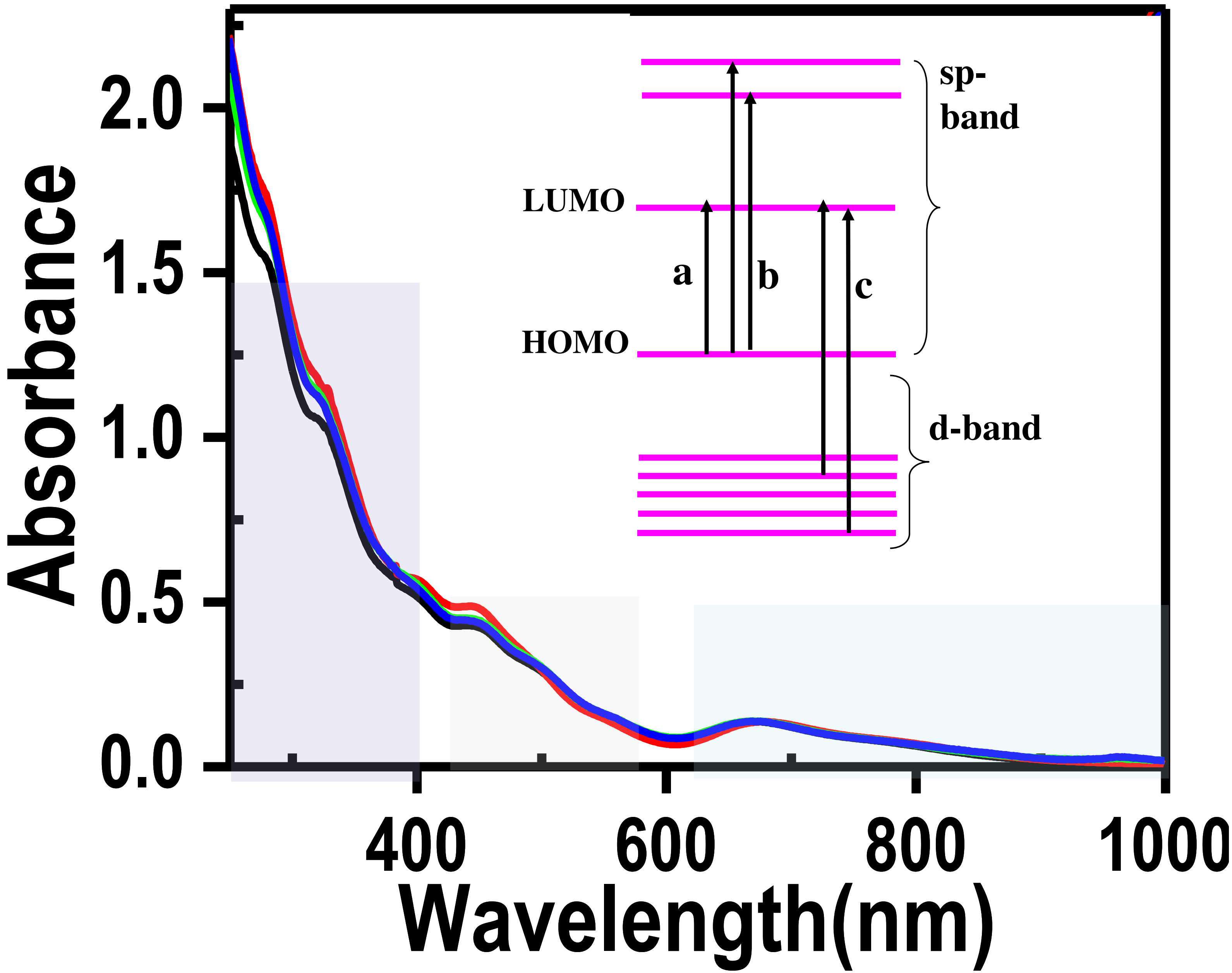


Ligands
Staples
Core
~1-3 nm
and more
eg. glutathione
4-(tert butyl)benzyl
mercaptan
2-phenylethanethiol
Au_{13}
Au_{23}
Au_{20}, Au_{28},
Au_{79}, etc.
S
Au
S
Au monomer
S
Au
Au
S
S
Au dimer
trimer,
pentamer, etc.

$Au_{25}SR_{18}$



Geometric and electronic stability



Absorbance
0.0
0.5
1.0
1.5
2.0
400
600
800
1000
Wavelength(nm)
LUMO
HOMO
sp-
band
d-band
a
b
c

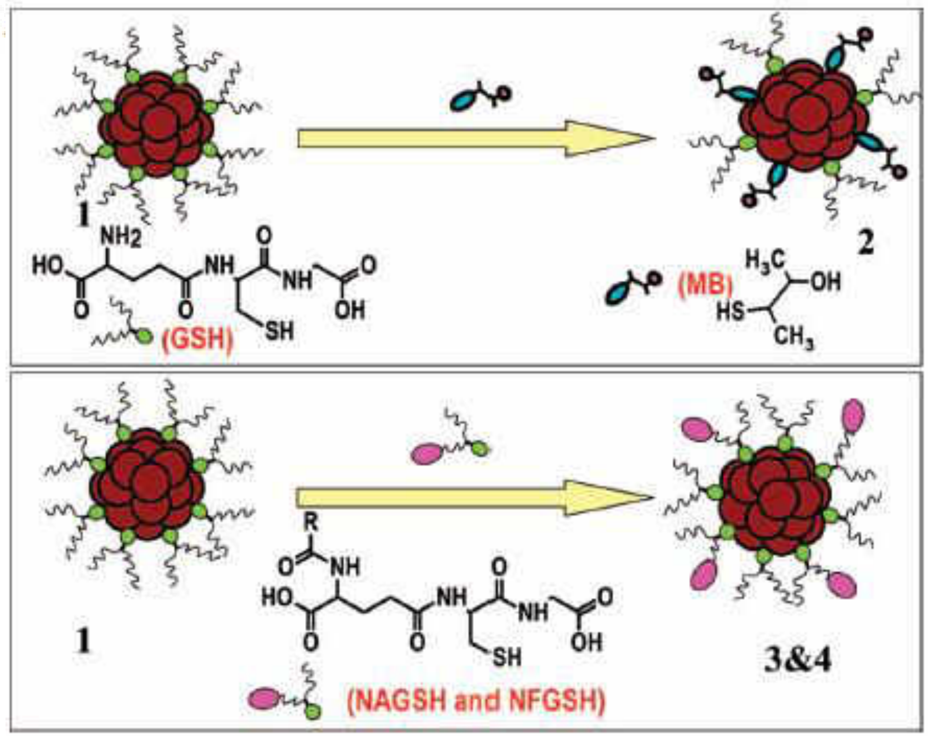
12168 *J. Phys. Chem. C* 2008, *112*, 12168–12176

Ligand Exchange of $Au_{25}SG_{18}$ Leading to Functionalized Gold Clusters: Spectroscopy, Kinetics, and Luminescence

E. S. Shibu,† M. A. Habeeb Muhammed,† T. Tsukuda,‡ and T. Pradeep*,†

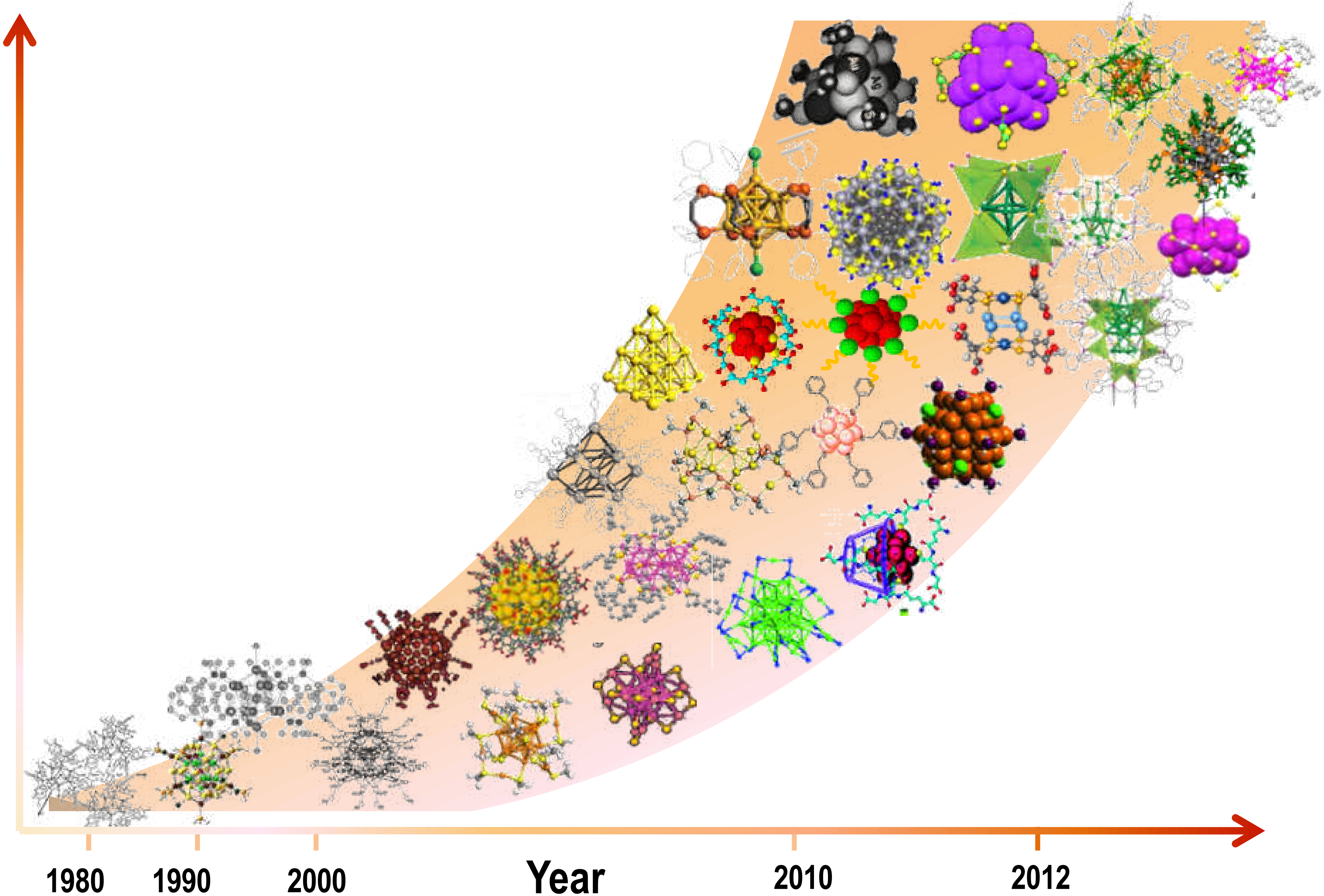
DST Unit on Nanoscience (DST UNS), Department of Chemistry and Sophisticated Analytical Instrument Facility, Indian Institute of Technology, Madras, Chennai 600 036, India and Institute for Molecular Science, Myodaiji, Okazaki 444-8585, Japan

Received: January 18, 2008;



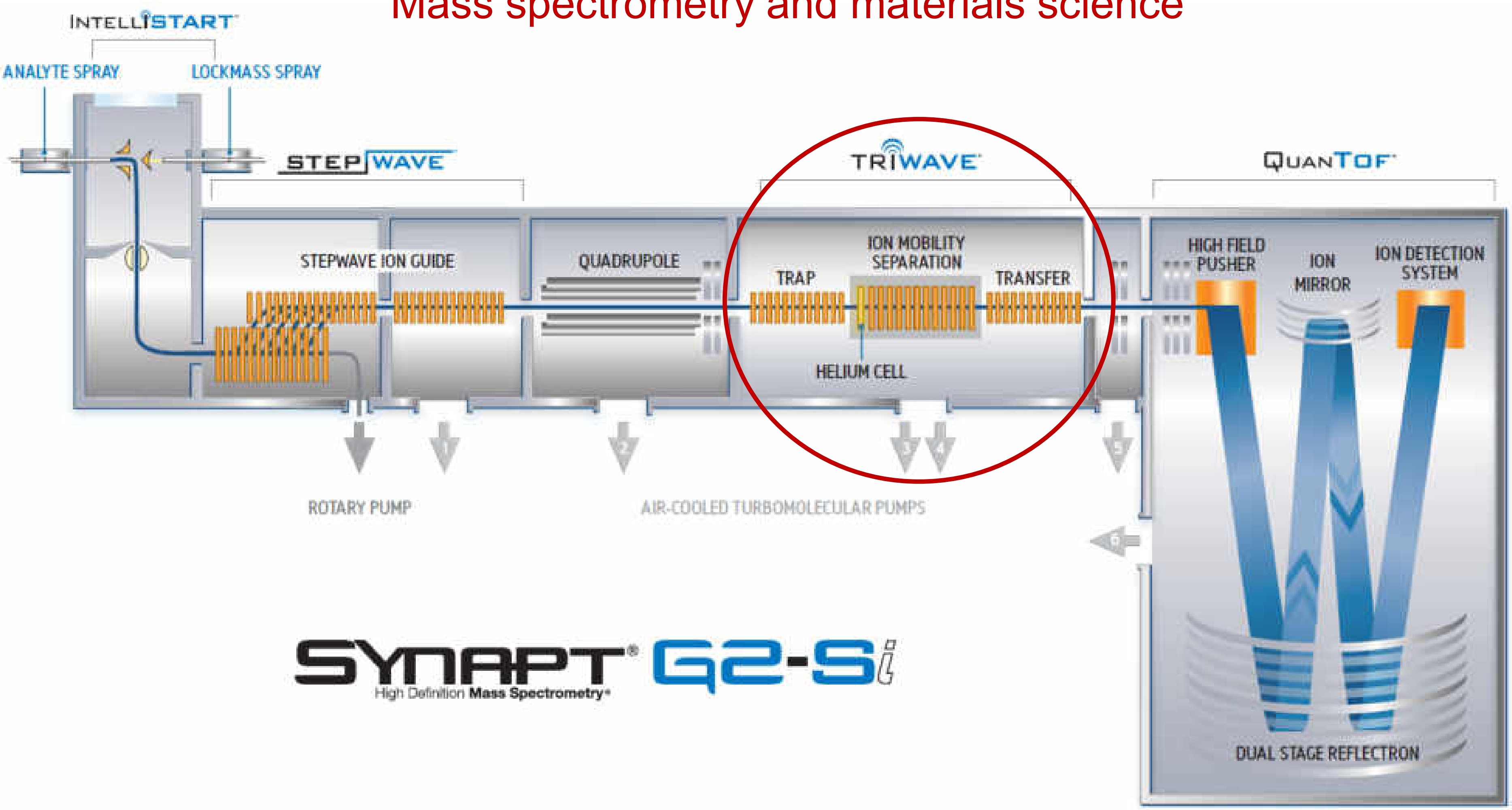
With Tatsuya Tsukuda

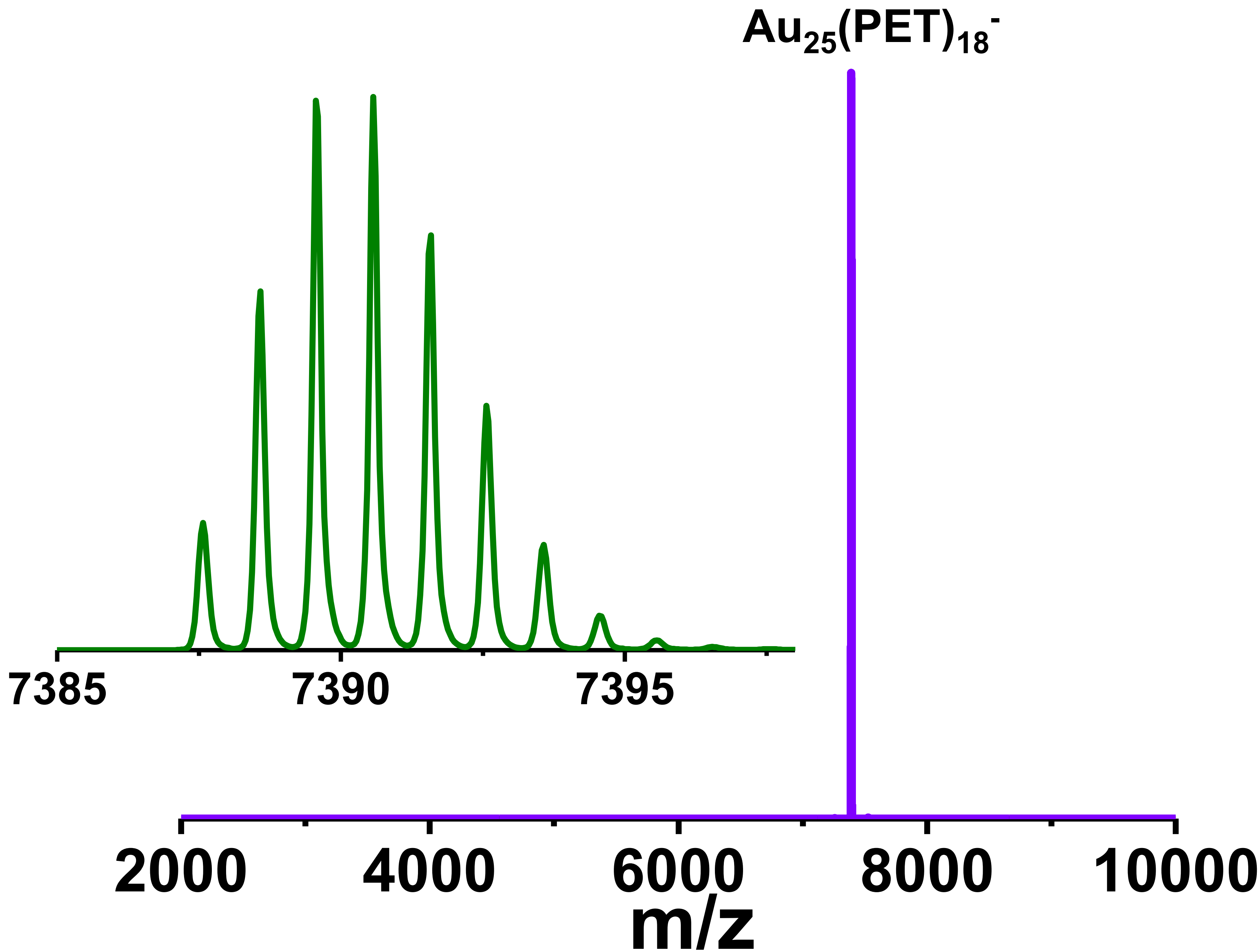
Evolution of noble metal clusters



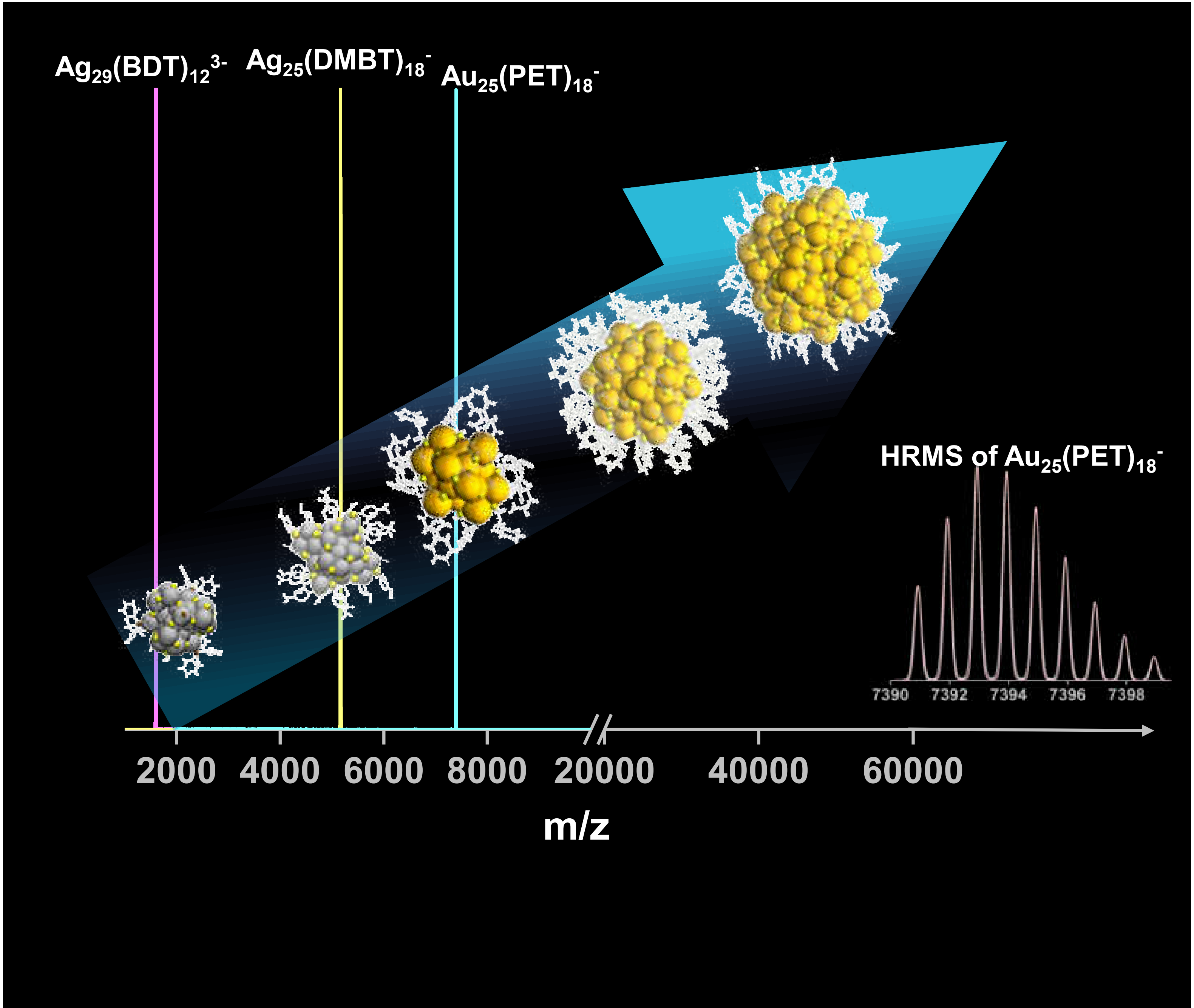
I. Chakraborty and T. Pradeep, *Chem. Rev.* 2017

Mass spectrometry and materials science





Au25(PET)18-
7385
7390
7395
2000
4000
6000
8000
10000
m/z



$Ag_{29}(BDT)_{12}^{3-}$
$Ag_{25}(DMBT)_{18}^{-}$
$Au_{25}(PET)_{18}^{-}$
HRMS of $Au_{25}(PET)_{18}^{-}$
7390 7392 7394 7396 7398
2000 4000 6000 8000 20000 40000 60000
m/z

Molecular materials

ACCOUNTS of chemical research

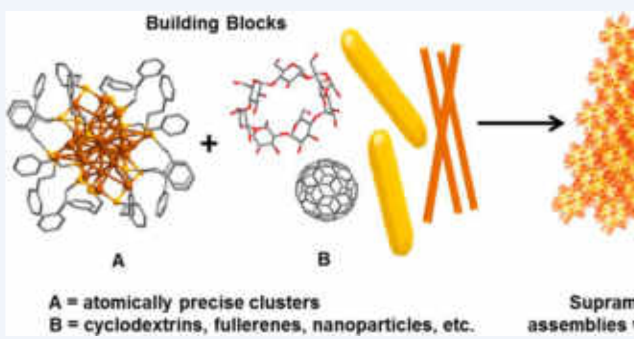
Article

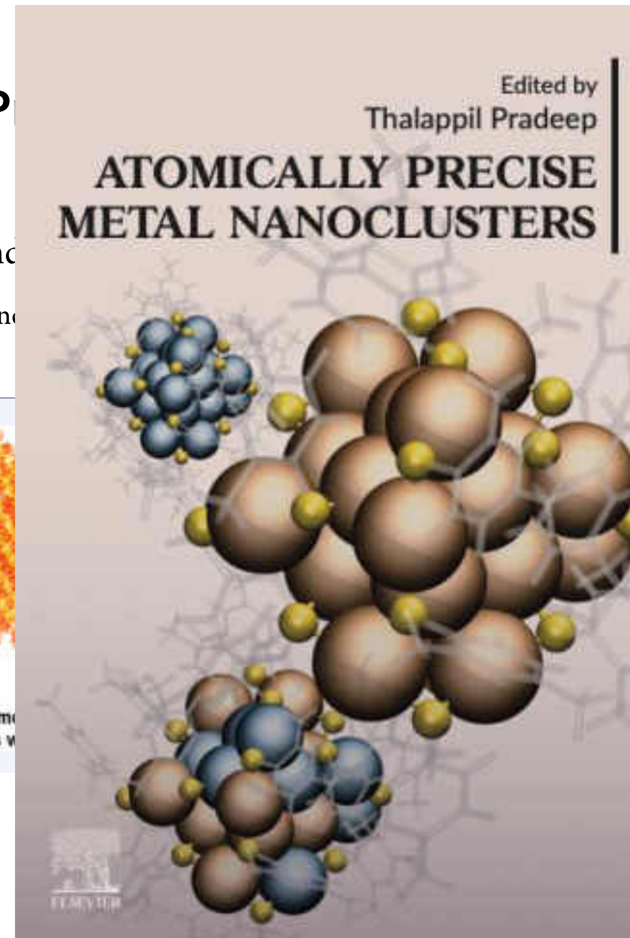
pubs.acs.org/accounts

Approaching Materials with Atomic P… lar Cluster Assemblies

Papri Chakraborty, Abhijit Nag, Amrita Chakraborty, and…

DST Unit of Nanoscience (DST UNS) and Thematic Unit of Excellen… of Technology Madras, Chennai 600 036, India





Molecules and their properties

Chemical formula	H_2O
Molecular weight	18.0148
Critical temperature	373.91°C
Critical pressure	22.05 MPa
Critical density	315.0 kg/m^3
Triple point temperature	0.01°C
Triple point pressure	615.066 Pa
Normal boiling point	100.0°C
Normal freezing point	0.0°C
Density of ice at normal melting point	918.0 kg/m^3
Maximum density, 3.98°C	999.973 kg/m^3
Viscosity, 25°C	0.889 mN s/m^2
Surface tension, 25°C	72 mN/m
Heat Capacity, 25°C	4.1796 kJ/kg.K
Enthalpy of vaporisation, 100°C	2,257.7 kJ/kg
Enthalpy of fusion, 0°C	333.8 kJ/kg
Velocity of sound, 0°C	1.403 km/s
Dielectric constant, 25°C	78.40
Electrical conductivity, 25°C	8 μS/m
Refractive index, 25°C	1.333
Liquid compressibility, 10°C	$480. \times 10^{-12}$ m^2/N
Coefficient of thermal expansion, 25°C	256.32×10^{-6} K^{-1}
Thermal Conductivity, 25°C	0.608 W/m.K

Molecular formula
Molecular weight
Molecular structure
Molecular absorption and emission
Molecular reactions
Molecular assembly
Molecular co-crystals
Ionization potential
Electron affinity

Phases - phase transitions
Physical properties
Electrical, magnetic
Mechanical properties
Electrochemical properties

Future?

Molecular reactions

Reactions on clusters

Reactions <u>between</u> clusters

Inter-cluster reactions

JACS
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Article
pubs.acs.org/JACS

Intercluster Reactions between $Au_{25}(SR)_{18}$ and $Ag_{44}(SR)_{30}$

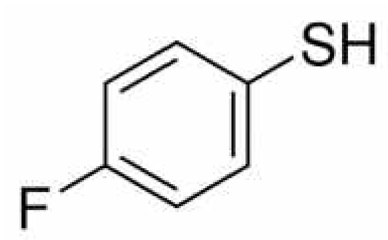
K. R. Krishnadas, Atanu Ghosh, Ananya Baksi, Indranath Chakraborty,[†] Ganapati Natarajan, and Thalappil Pradeep[*]

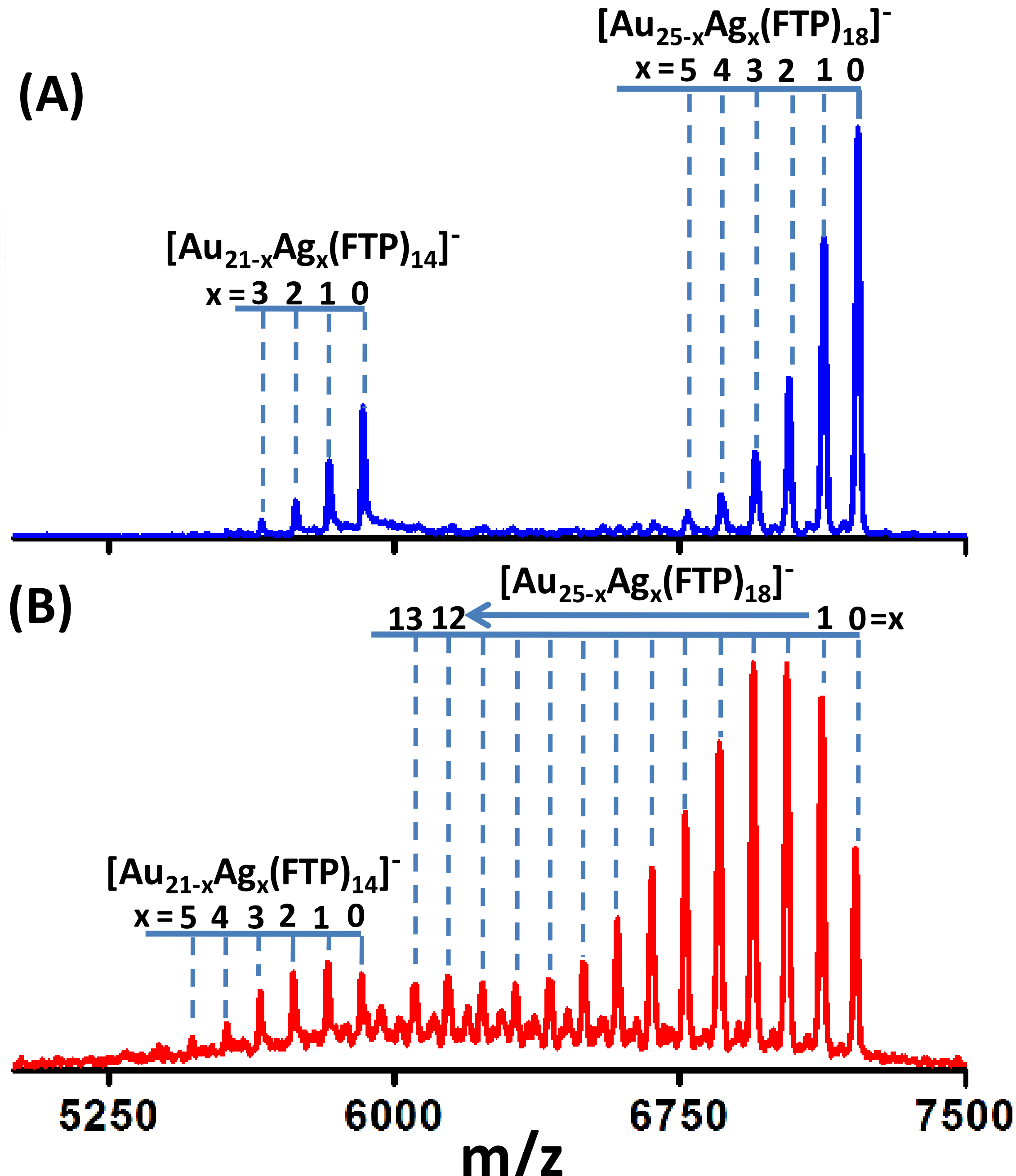
DST Unit of Nanoscience (DST UNS) and Thematic Unit of Excellence, Department of Chemistry, Indian Institute of Technology Madras, Chennai, 600 036, India

Supporting Information

$$A + B \rightarrow C + D$$

$Au_{25}(FTP)_{18}$ + $Ag_{44}(FTP)_{30}$ → $Au_{25-x}Ag_x(FTP)_{18}$



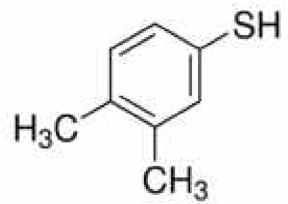


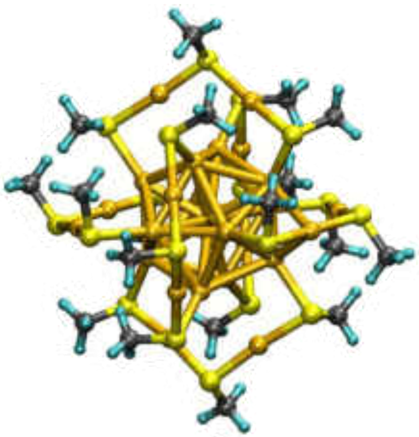
Ag_{25}-Au_{25} experiments

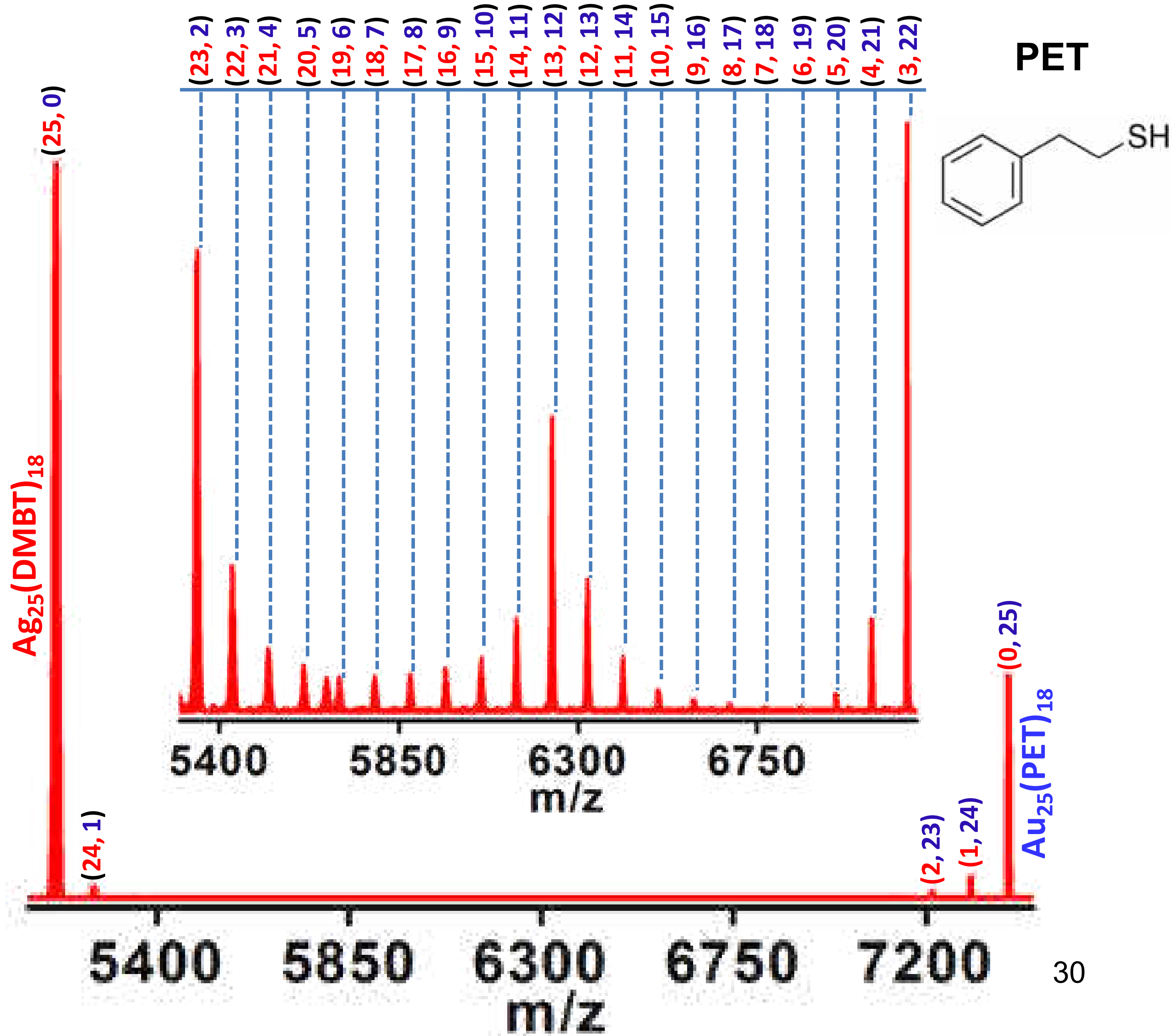
K. R. Krishnadas et al. *Nature Commun.* 2016

Reaction between $Au_{25}(PET)_{18}$ and $Ag_{25}(DMBT)_{18}$

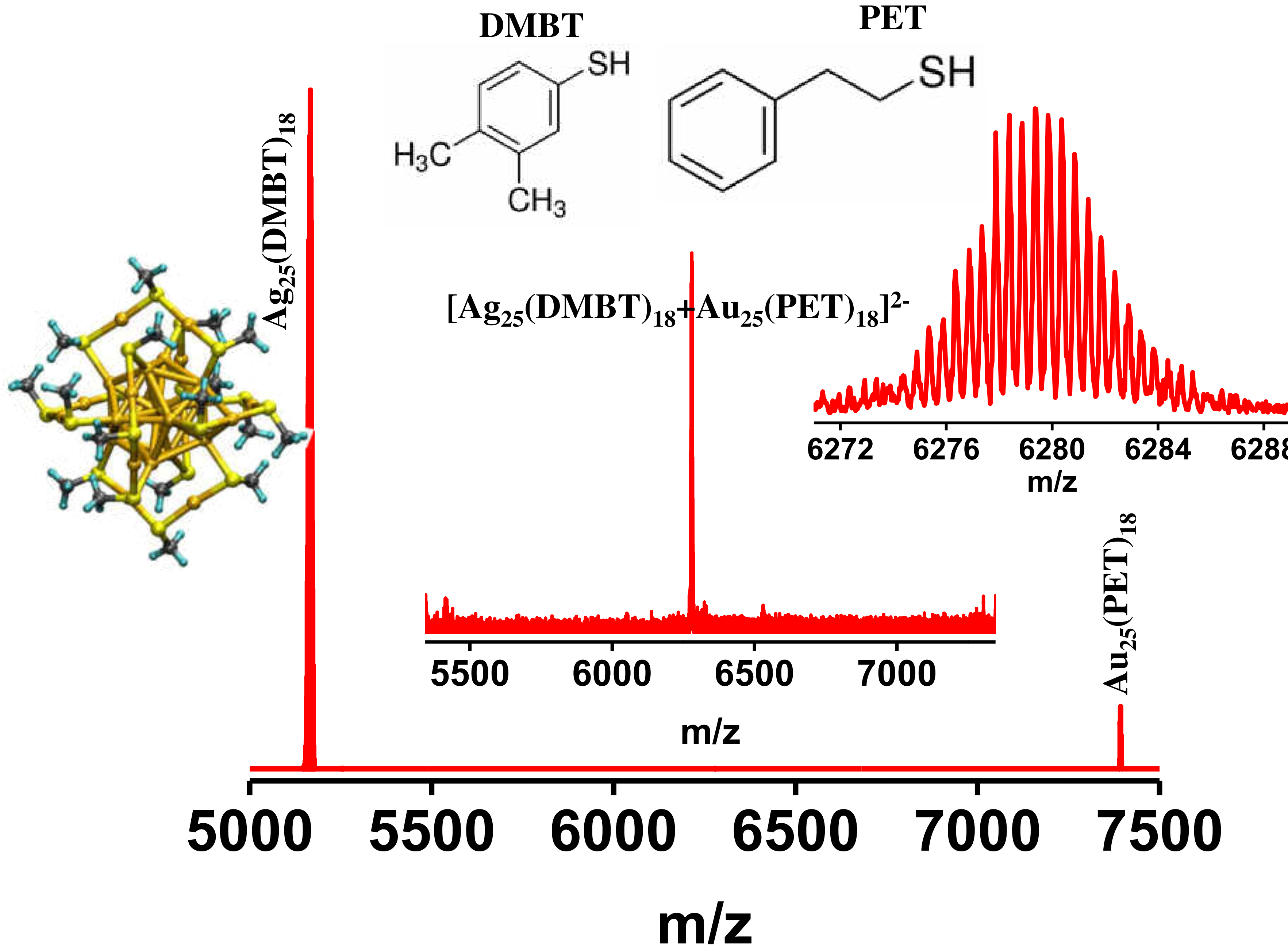
DMBT





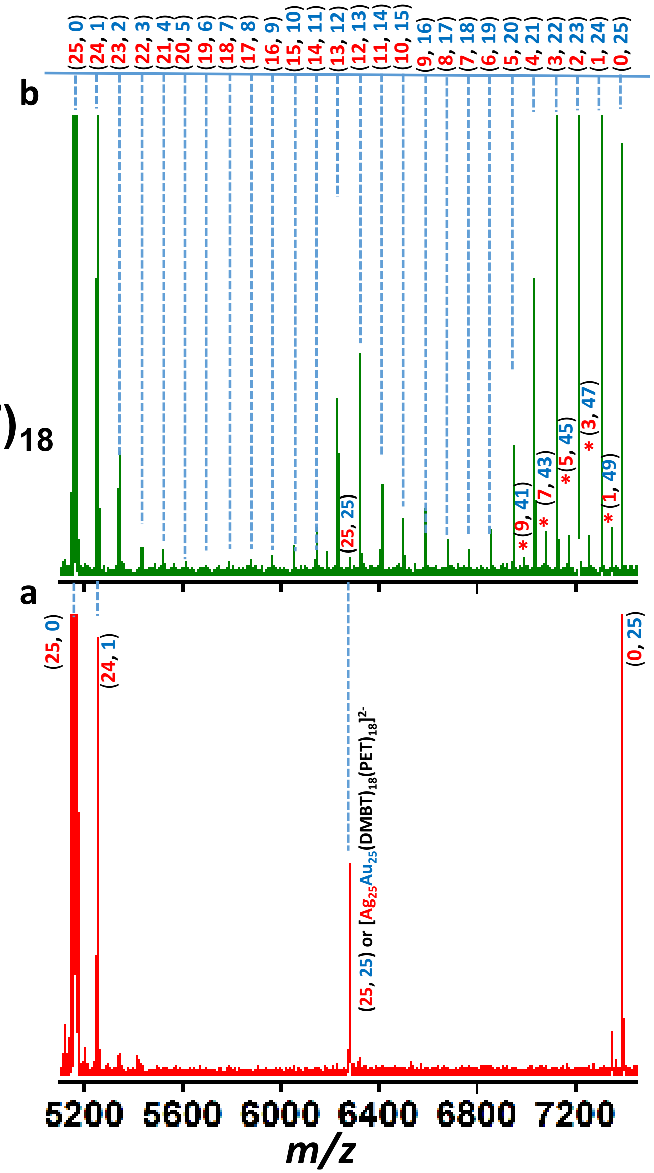


$[Ag_{25}(DMBT)_{18}+Au_{25}(PET)_{18}]^{2-}$



Evolution of alloy clusters from the dianionic adduct, $[Ag_{25}Au_{25}(DMBT)_{18}(PET)_{18}]^{2-}$

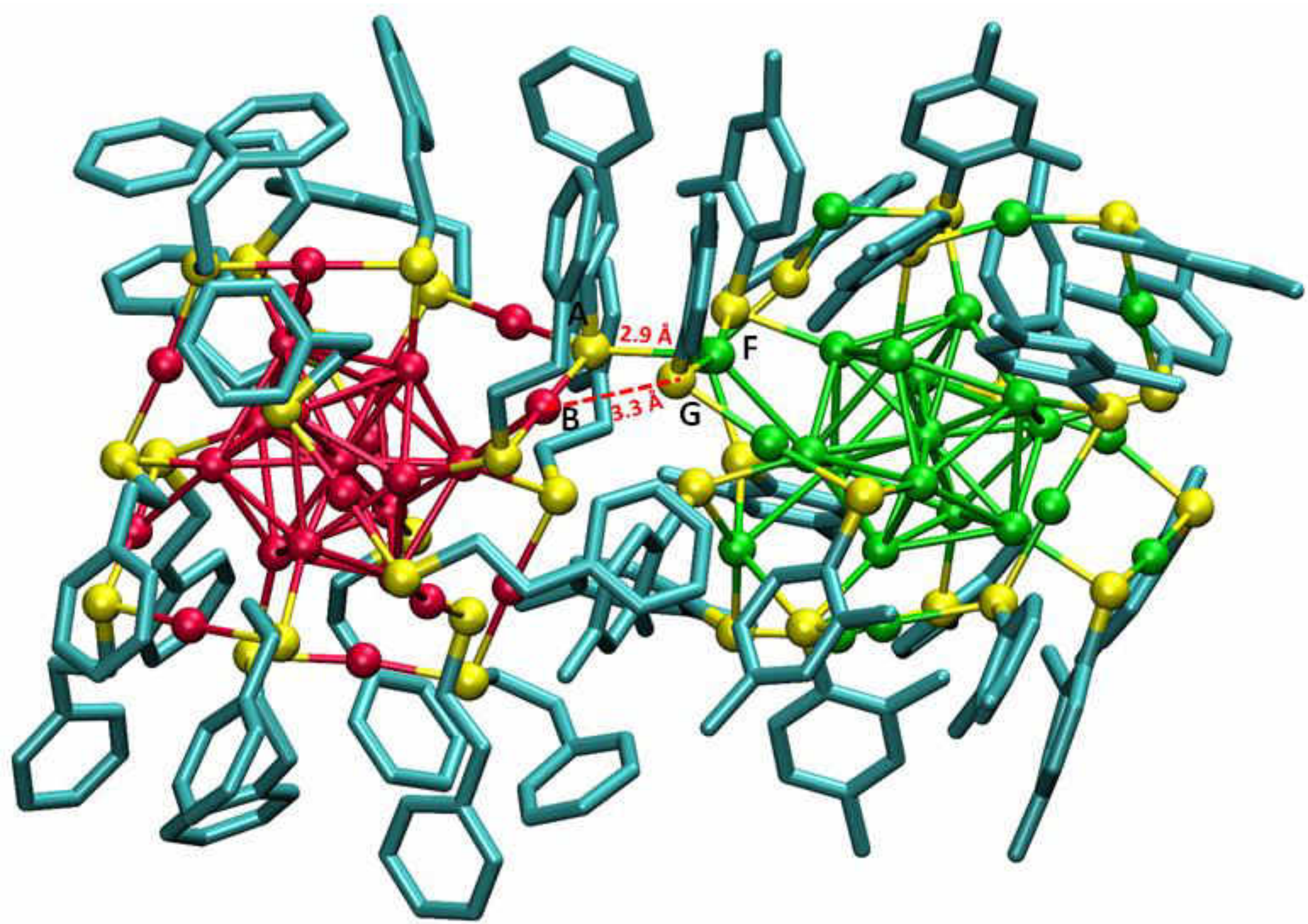
$Ag_{25}(DMBT)_{18}$:$Au_{25}(PET)_{18}$
0.3:1.0

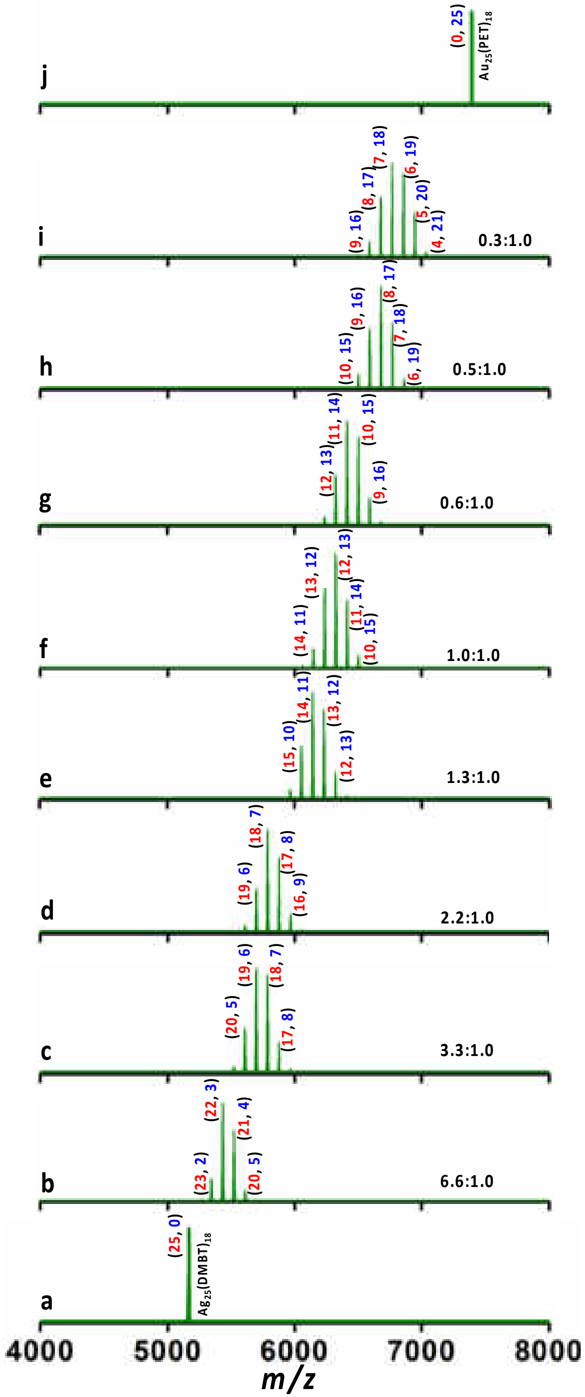


within 5 min

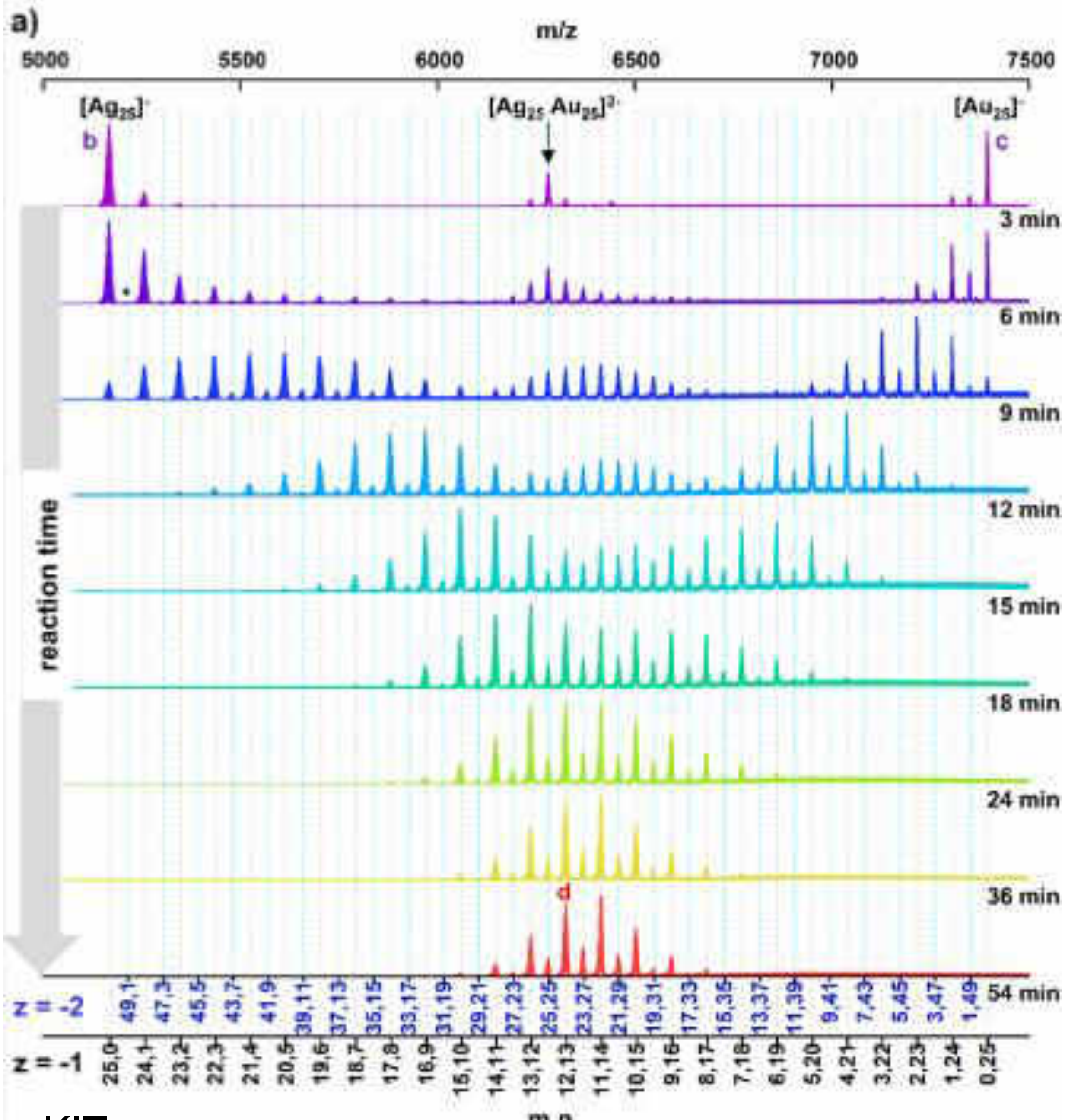
within 2 min

Optimized structure of $[Ag_{25}Au_{25}(DMBT)_{18}(PET)_{18}]^{2-}$





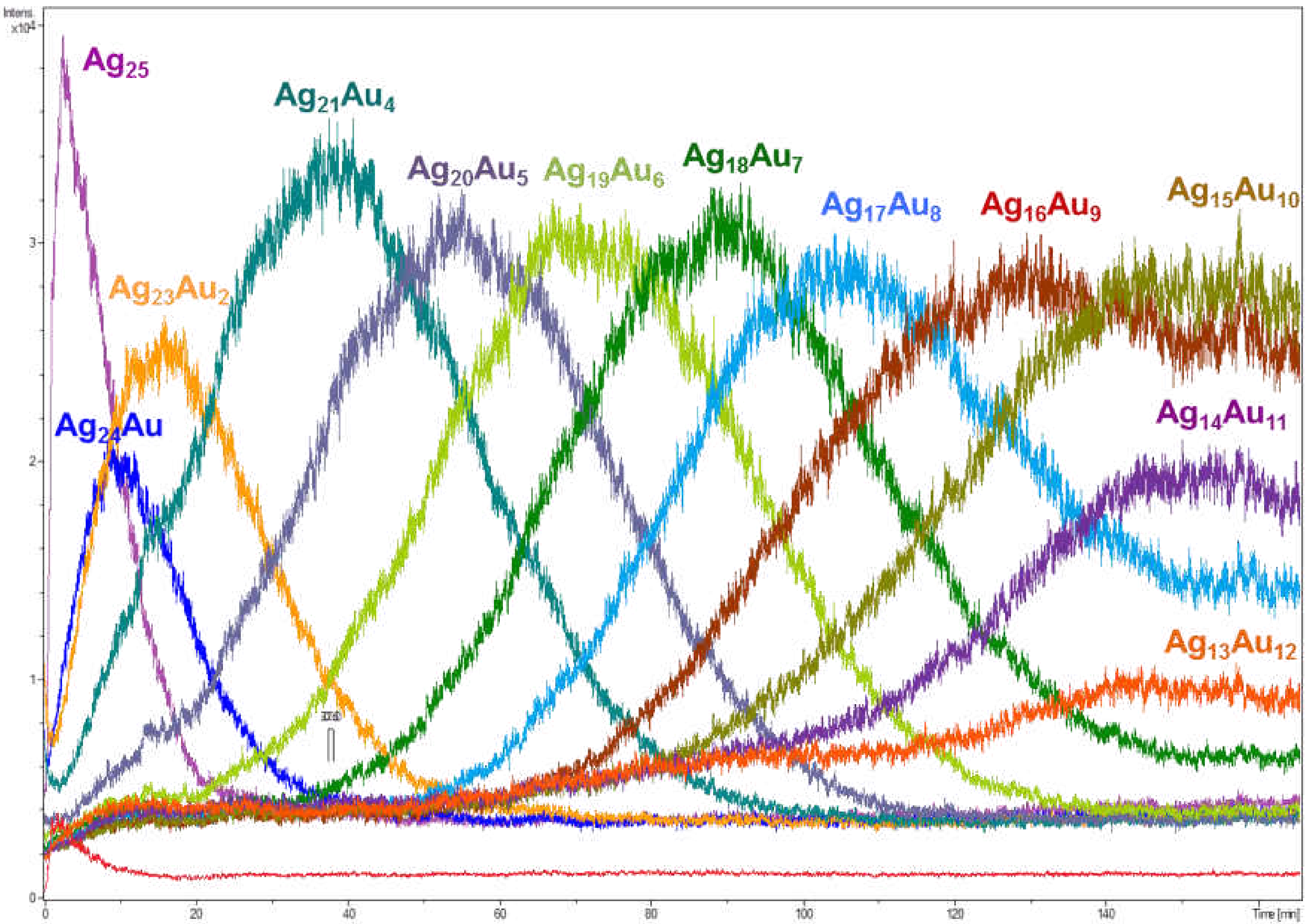
j
(0, 25)
Au25(PET)18
i
(9, 16)
(8, 17)
(7, 18)
(6, 19)
(5, 20)
(4, 21)
0.3:1.0
h
(10, 15)
(9, 16)
(8, 17)
(7, 18)
(6, 19)
0.5:1.0
g
(12, 13)
(11, 14)
(10, 15)
(9, 16)
0.6:1.0
f
(14, 11)
(13, 12)
(12, 13)
(11, 14)
(10, 15)
1.0:1.0
e
(15, 10)
(14, 11)
(13, 12)
(12, 13)
1.3:1.0
d
(19, 6)
(18, 7)
(17, 8)
(16, 9)
2.2:1.0
c
(20, 5)
(19, 6)
(18, 7)
(17, 8)
3.3:1.0
b
(23, 2)
(22, 3)
(21, 4)
(20, 5)
6.6:1.0
a
(25, 0)
Ag25(DMBT)18
4000
5000
6000
7000
8000
m/z



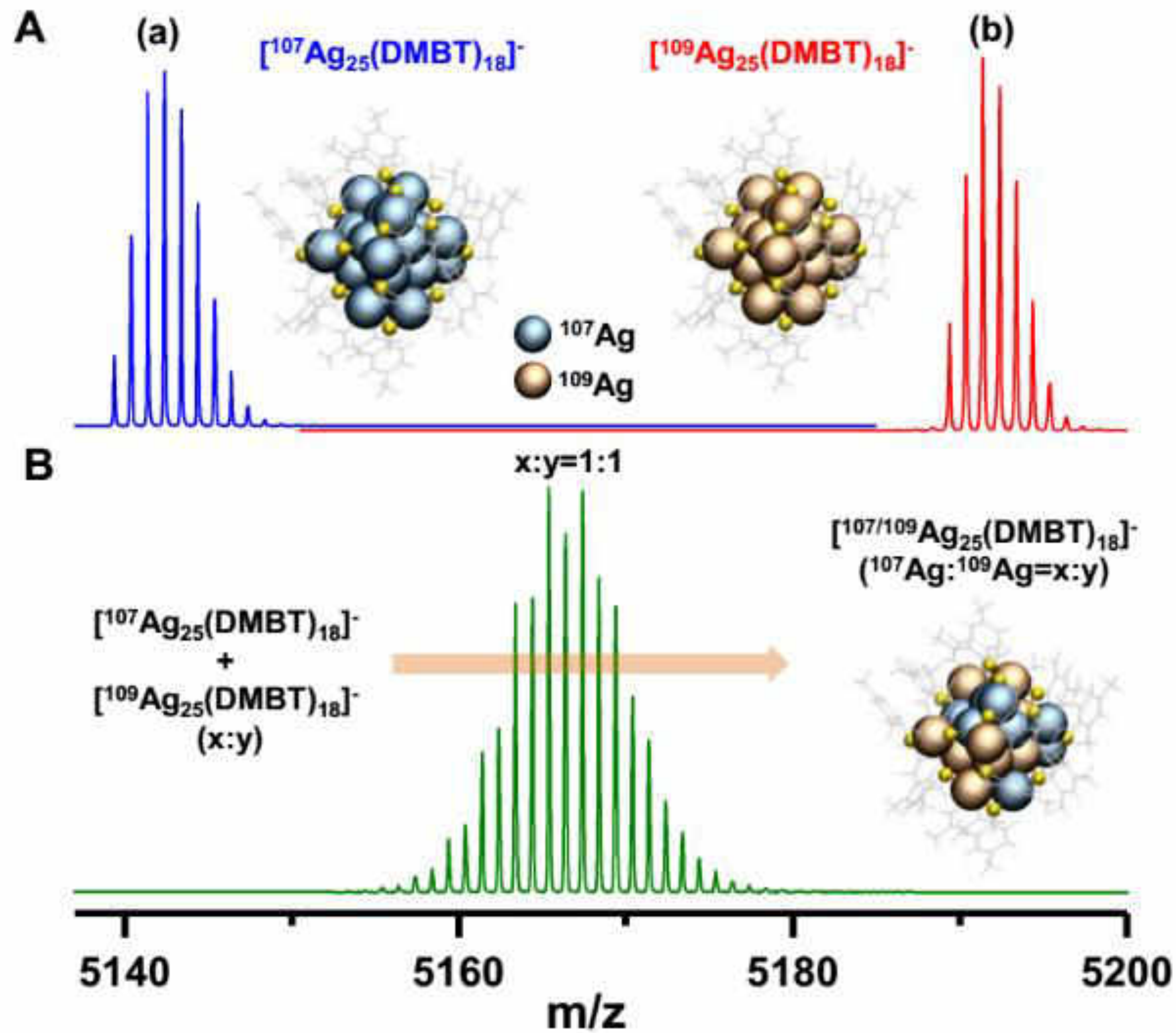
Manfred Kappes, KIT

M. Neumaier, A. Baksi, et. al., *JACS* 2021

Kinetics of the exchange (monitored on the Ag_{25} side)

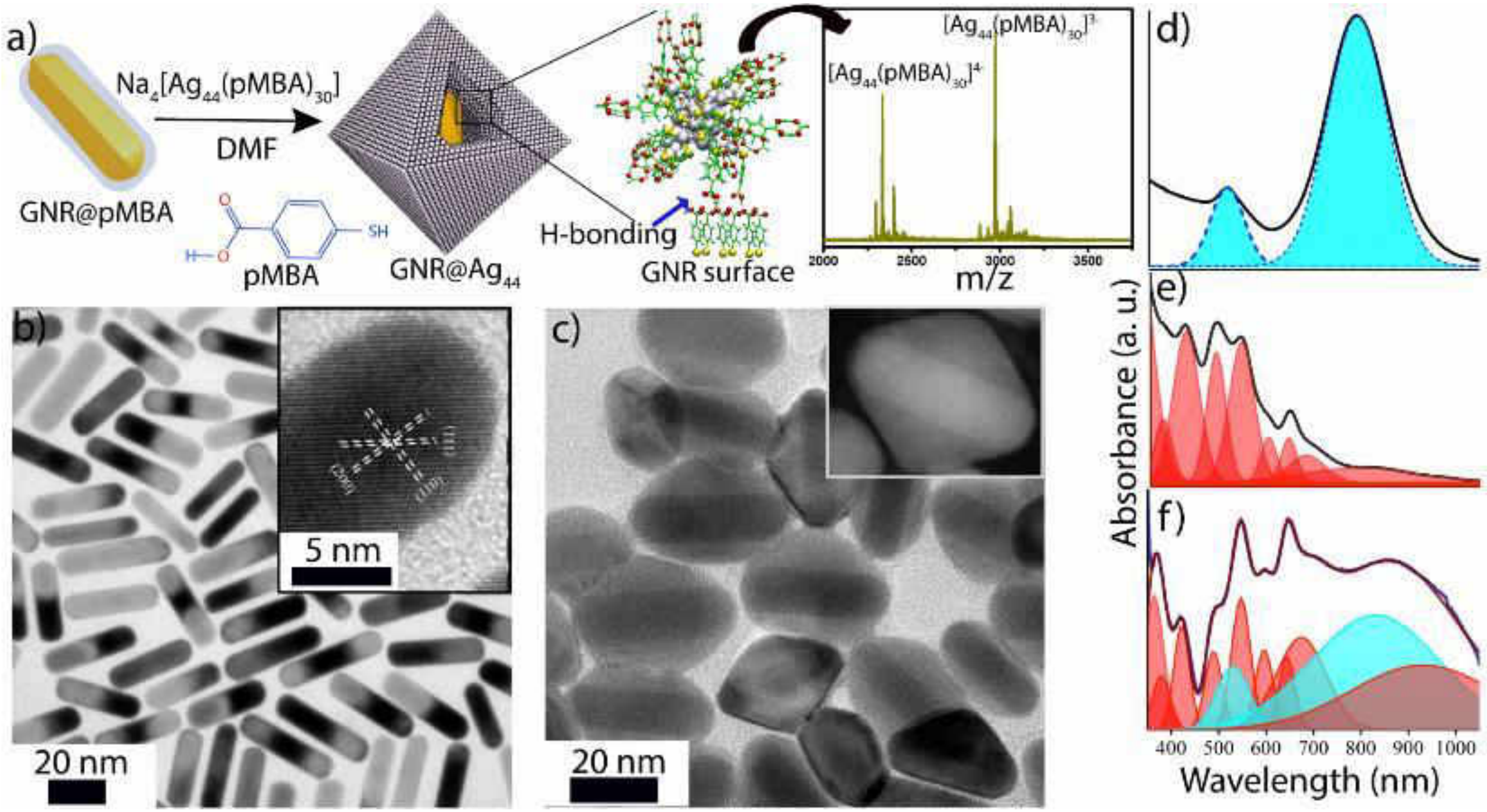


Isotopic exchange



Papri Chakraborti, et. al. Science Advances, 2019.

Atomically precise nanocluster assemblies encapsulating plasmonic gold nanorods



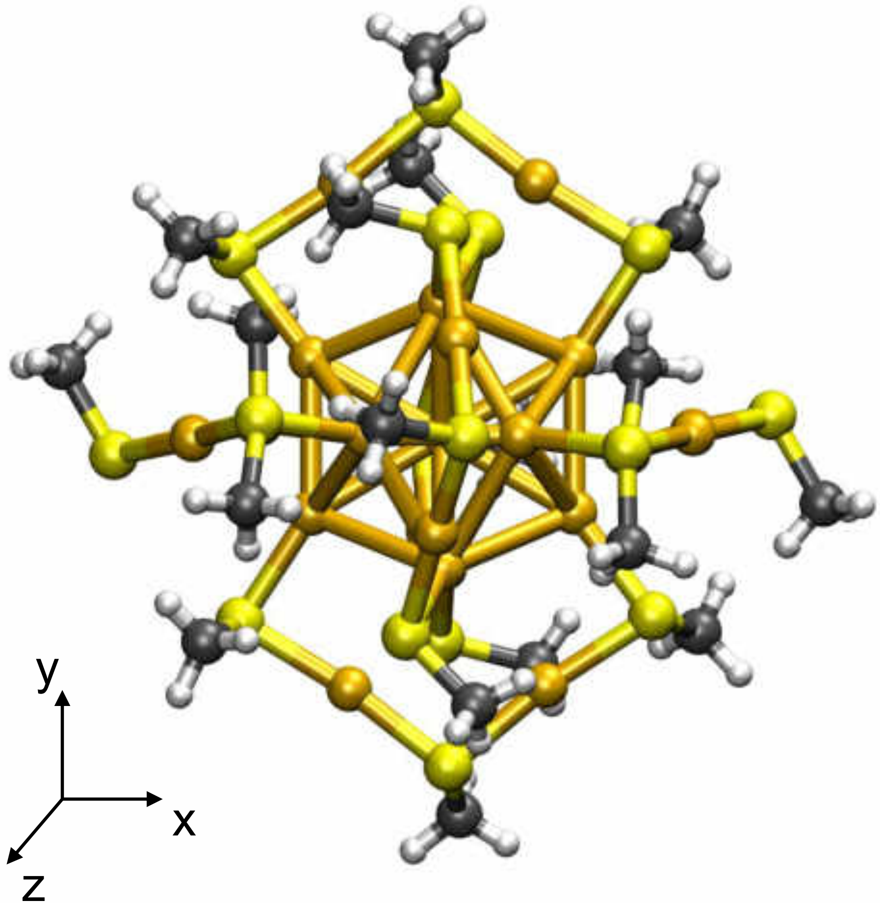
Chakraborty, A. et al., Angew. Chem. Int. Ed. **2018,** 57, 6522–6526.

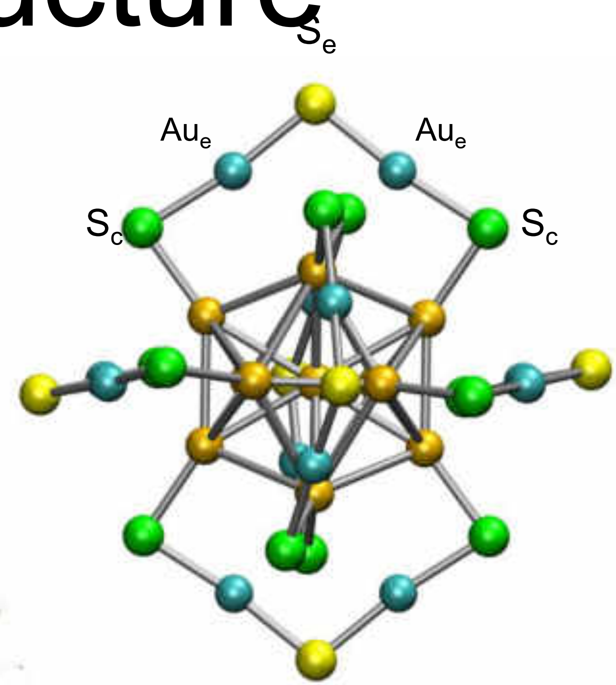
A system of nomenclature

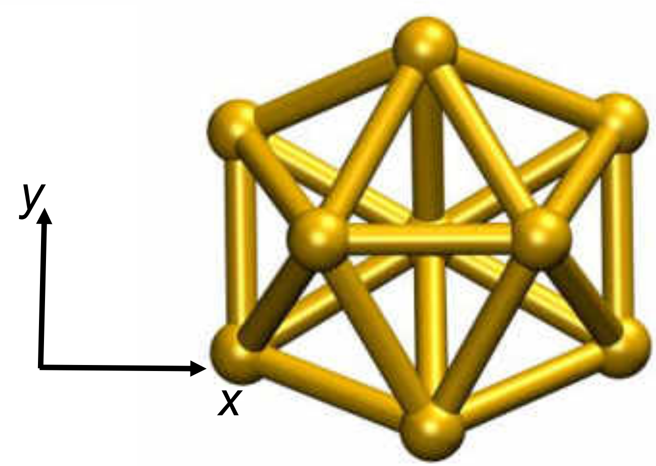
Aspicules

Gana Natarajan, et. al. JPC C. 2023

Ball and stick structure



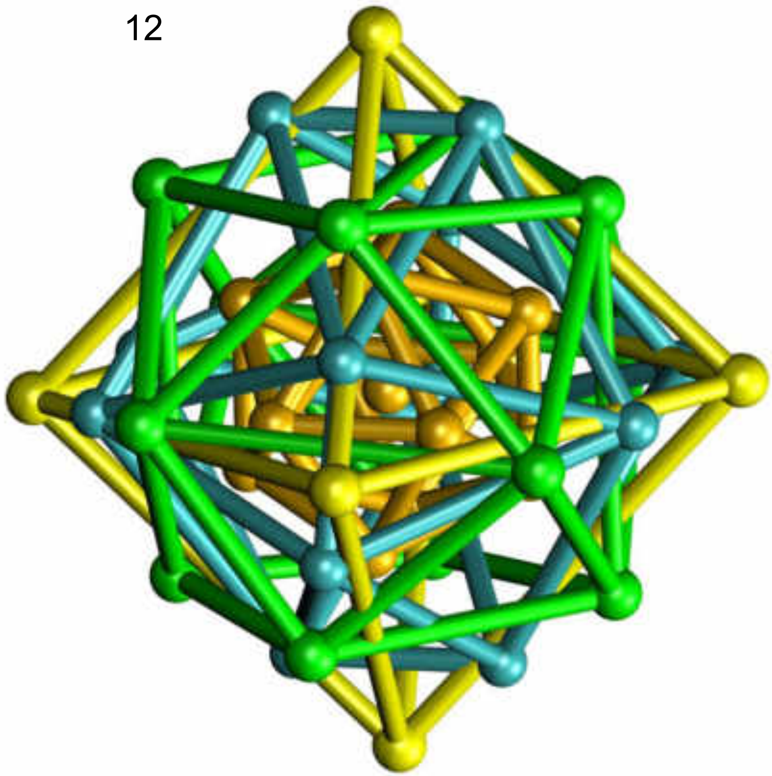




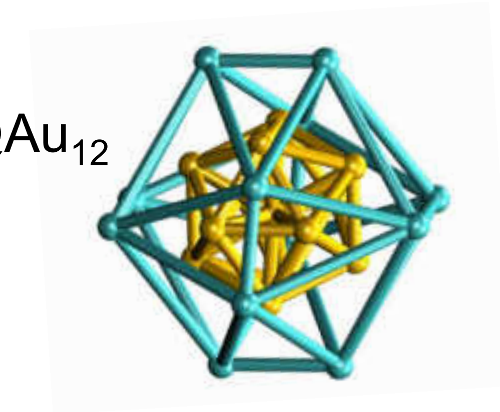
A view of gold methly thiolate [25]aspicule ($Au_{25}(SMe)_{18}$). Gold atoms colored gold, sulfur atoms by yellow, carbon dark gray, hydrogen atoms as white and (b) with the gold and sulfur atoms alone .

Shell Structure

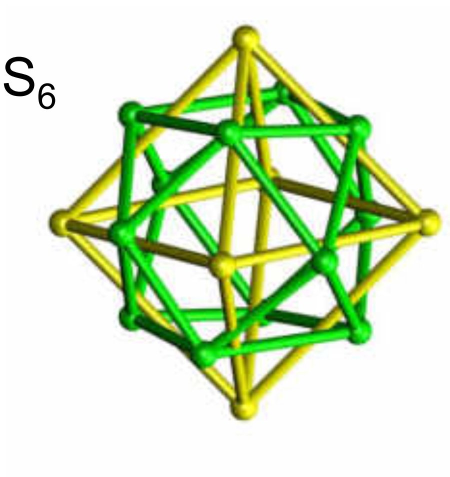
(a) $Au@Au_{12}@Au_{12}@S_6@S_{12}$



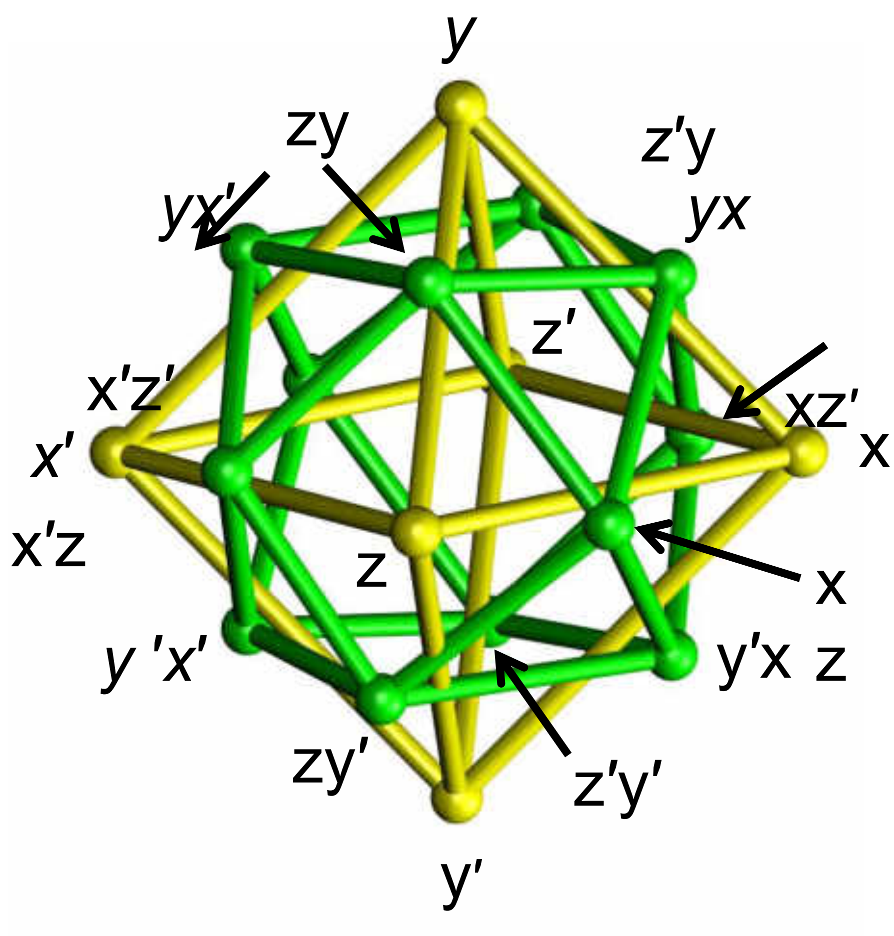
(b) $Au_{12}@Au_{12}$

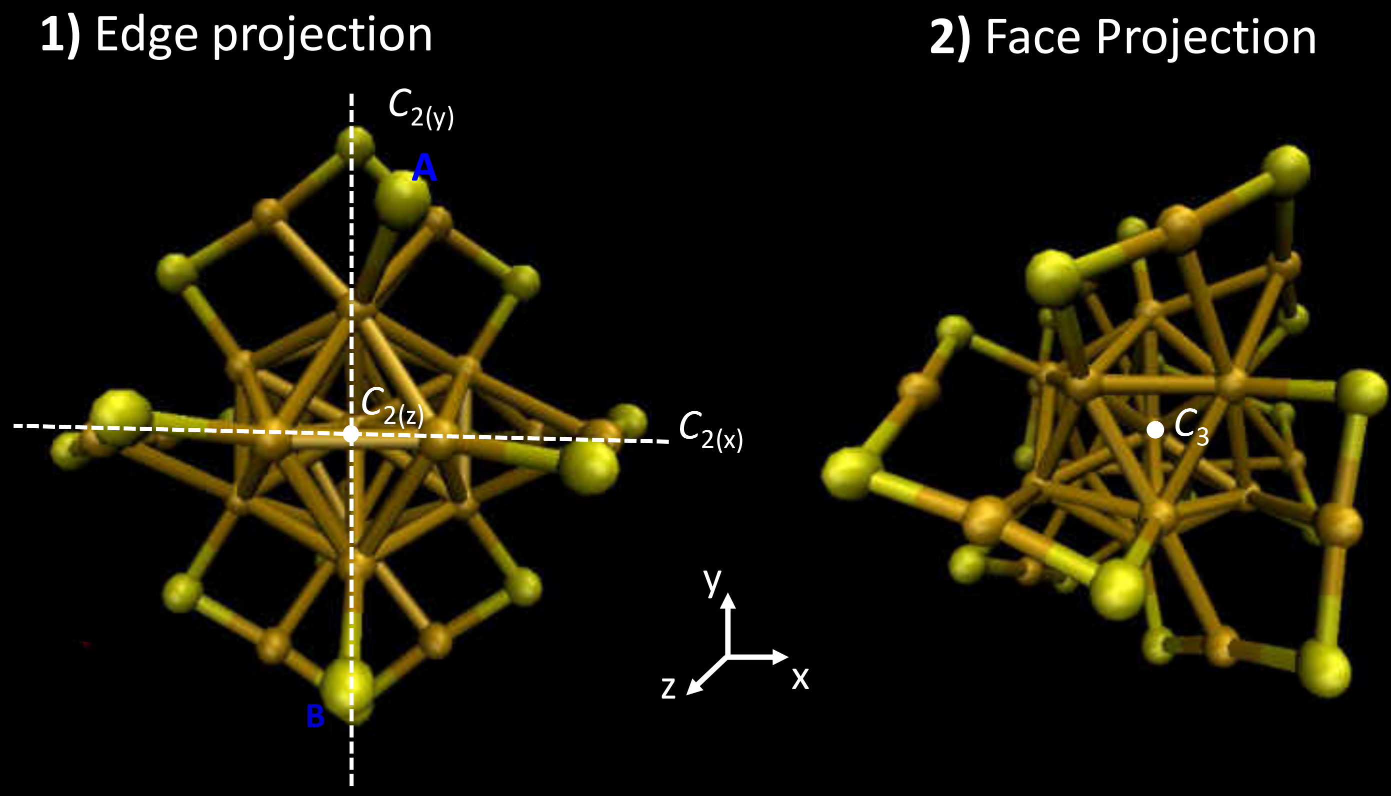


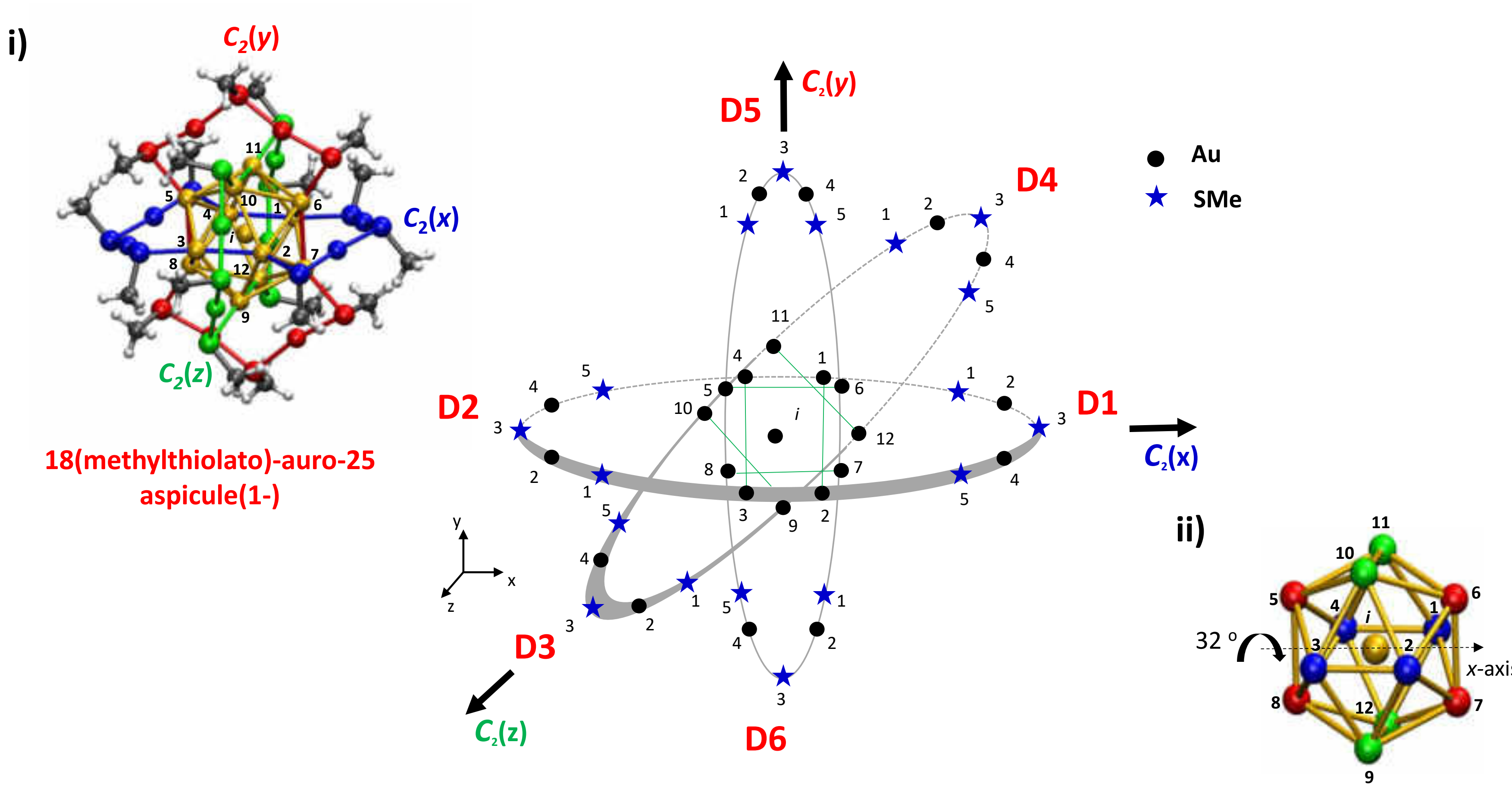
(c) $S_{12}@S_6$



Terminologies




1) Edge projection
2) Face Projection
C2(y)
A
C2(z)
C2(x)
B
C3
y
x
z



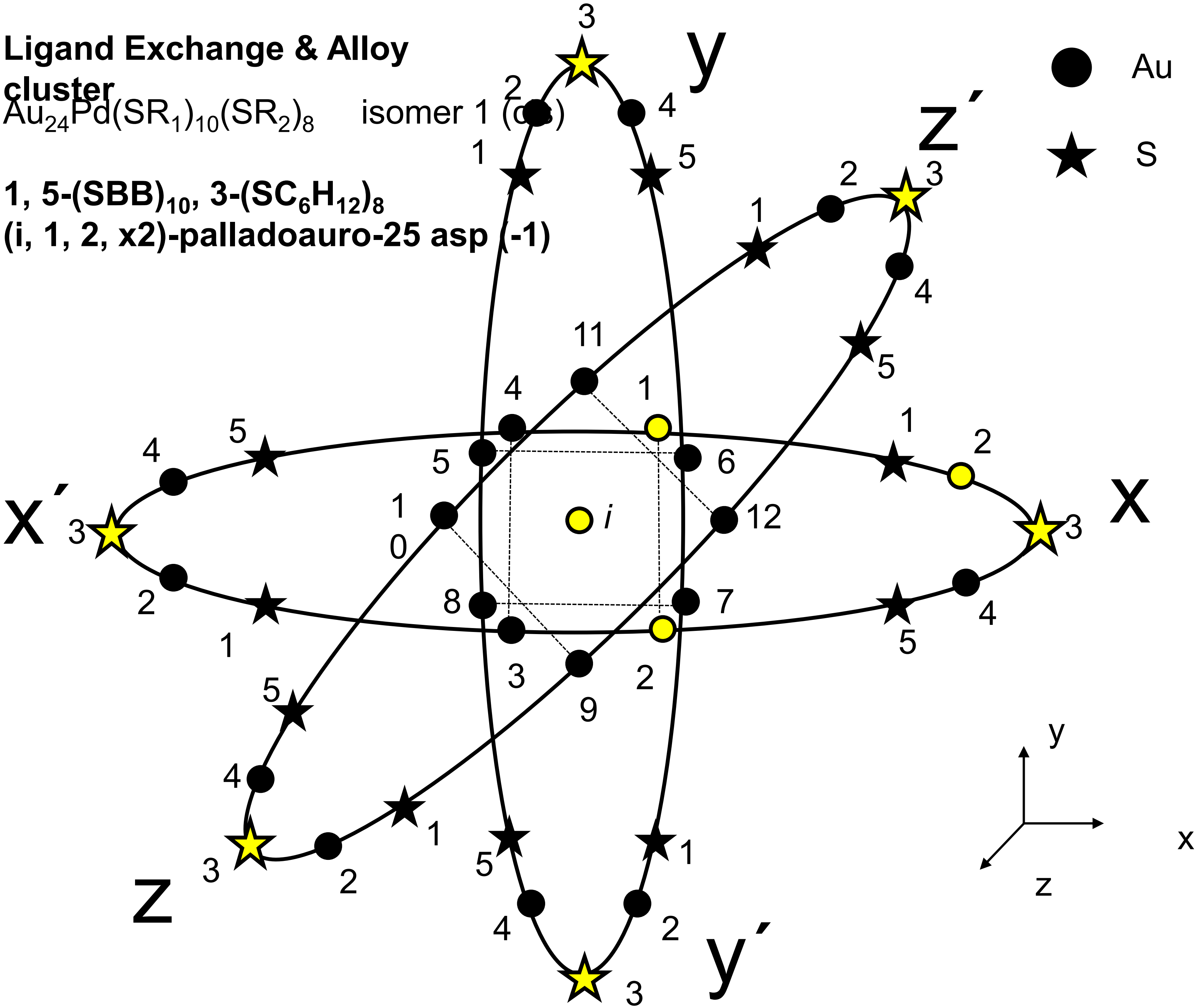
18(methylthiolato)-auro-25 aspicule(1-)

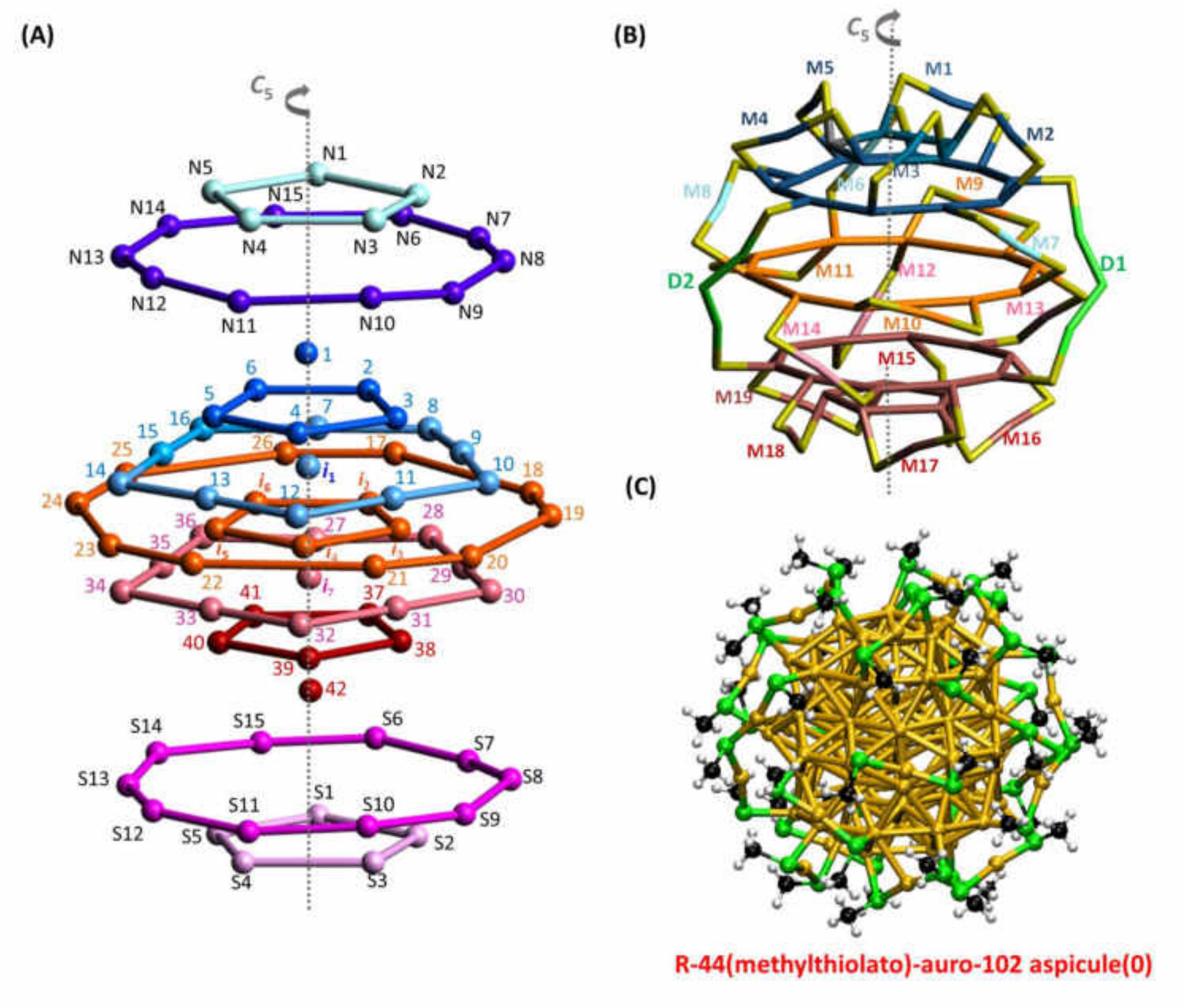
(D1-3,D2-3)-di(2-phenylethylthiolato),16(methylthiolato)-auro-25 aspicule(1-)
(D1-3,D2-3)-$(PET)_2$,$(SMe)_{16}$-auro-25 aspicule(1-)

Ligand Exchange & Alloy cluster

$Au_{24}Pd(SR_1)_{10}(SR_2)_8$ isomer 1 (cis)

1, 5-$(SBB)_{10}$, 3-$(SC_6H_{12})_8$
(i, 1, 2, x2)-palladoauro-25 asp (-1)





R-$(SMe)_{44}$-auro-102 aspicule**(**0**)** and L-$(SMe)_{44}$-auro-102 aspicule**(**0**)**

PNAS PNAS PNAS PNAS

Biopolymer-re nanocomposit water purifica

Mohan Udhaya Sankar[1], Saha
Kamalesh Chaudhari, and Tha

Unit of Nanoscience and Thematic Uni

Edited by Eric Hoek, University of Calif

Creation of affordable materials fo
water is one of the most promising
drinking water for all. Combinin
composites to scavenge toxic sp
other contaminants along with tl
affordable, all-inclusive drinking
without electricity. The critical p
synthesis of stable materials tha
uously in the presence of comp
drinking water that deposit and
surfaces. Here we show that suc
be synthesized in a simple and effe
out the use of electrical power. T
sand-like properties, such as highe
forms. These materials have beer
water purifier to deliver clean drin
ily. The ability to prepare nanos
ambient temperature has wide
water purification.

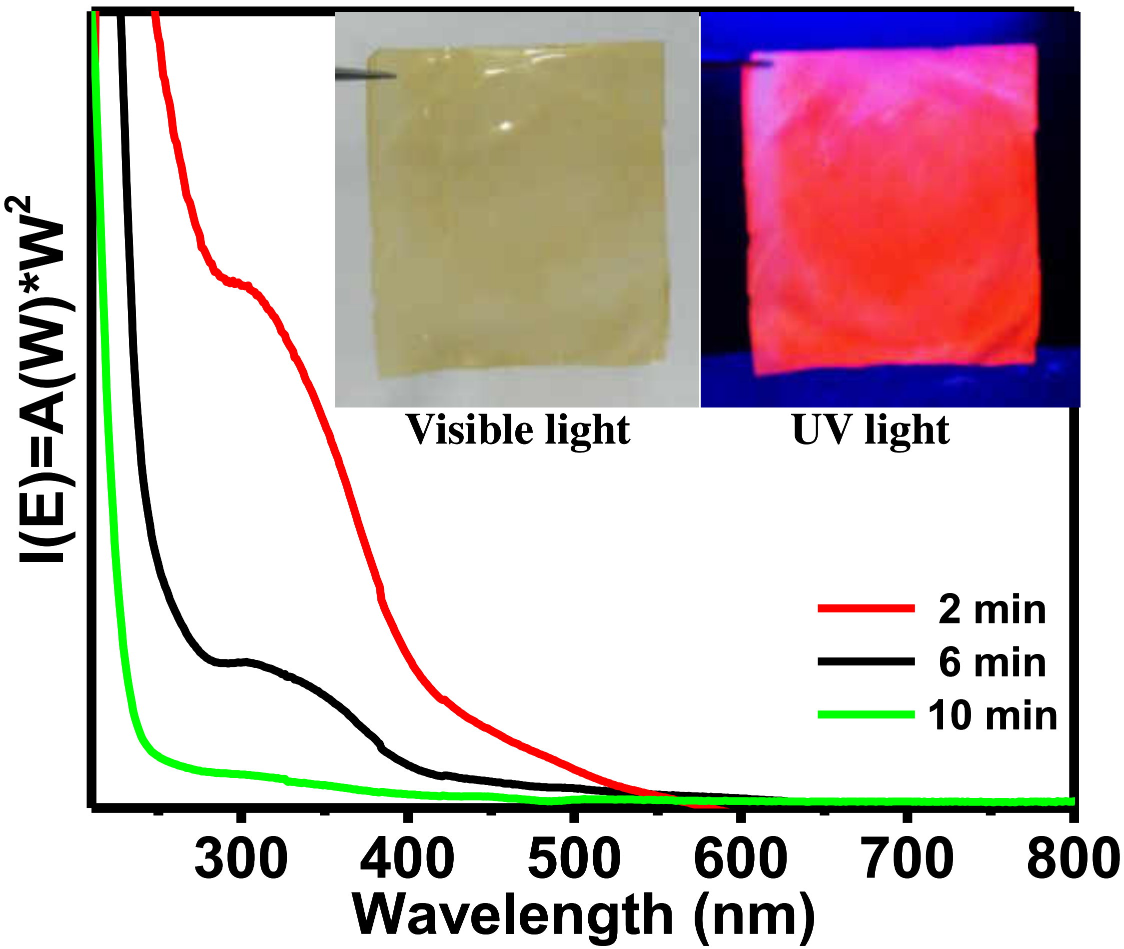
hybrid | green | appropriate technolog

Work was featured in several journals

Nature Nanotechnology, July 2014 issue


We developed environmentally friendly water positive nanoscale materials for affordable, sustainable and rapid removal of arsenic from drinking water.
There are over 1700 community installations across the country, serving 1.3 million people with arsenic and iron-free water every day.

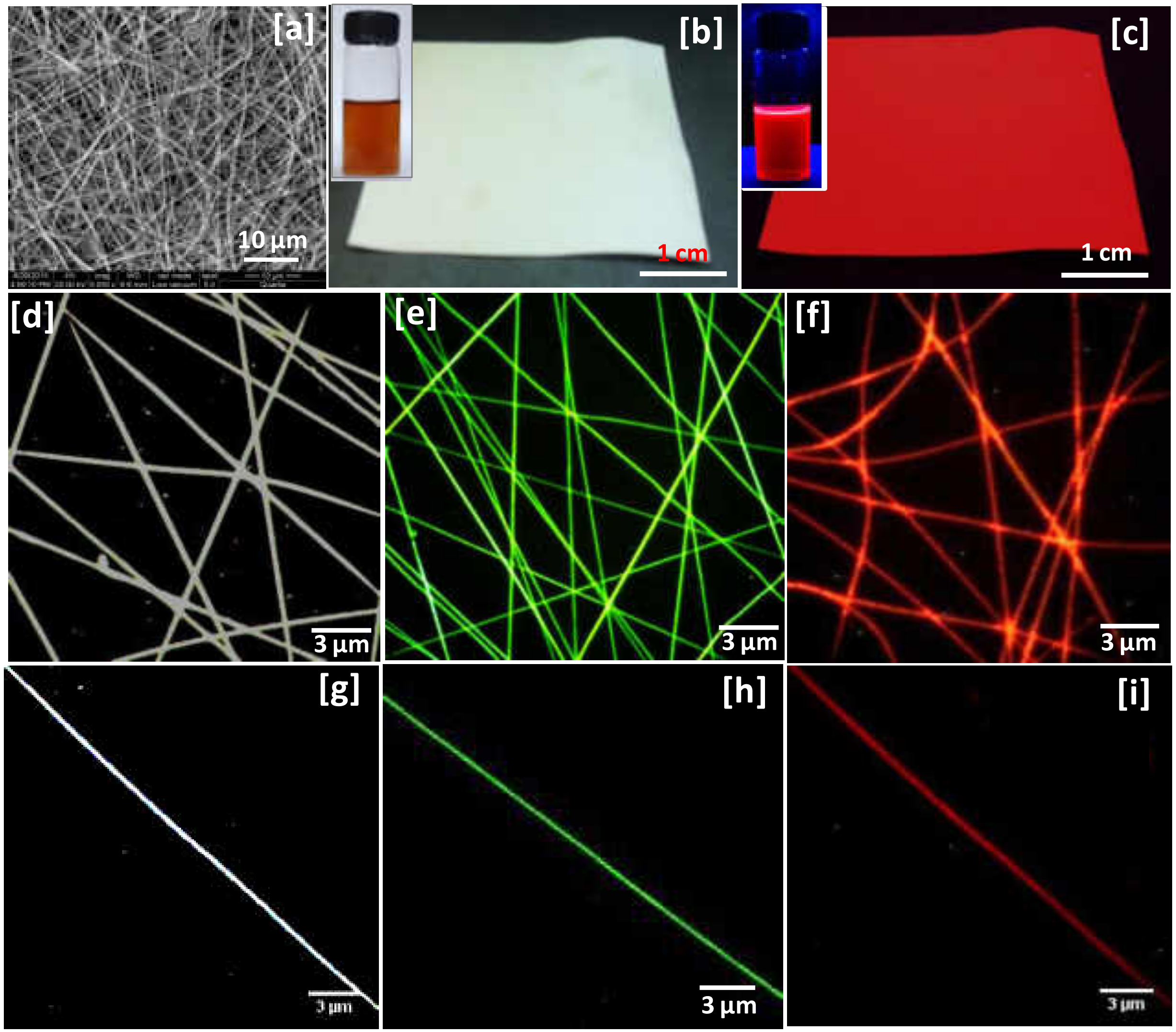
Quantum cluster based metal ion sensing paper
Large area uniform illumination using quantum cluster



Decrease in the absorption of Au_{15} as a biofilm is dipped into the cluster solution. Inset: Free standing quantum cluster loaded film in visible light and UV light.

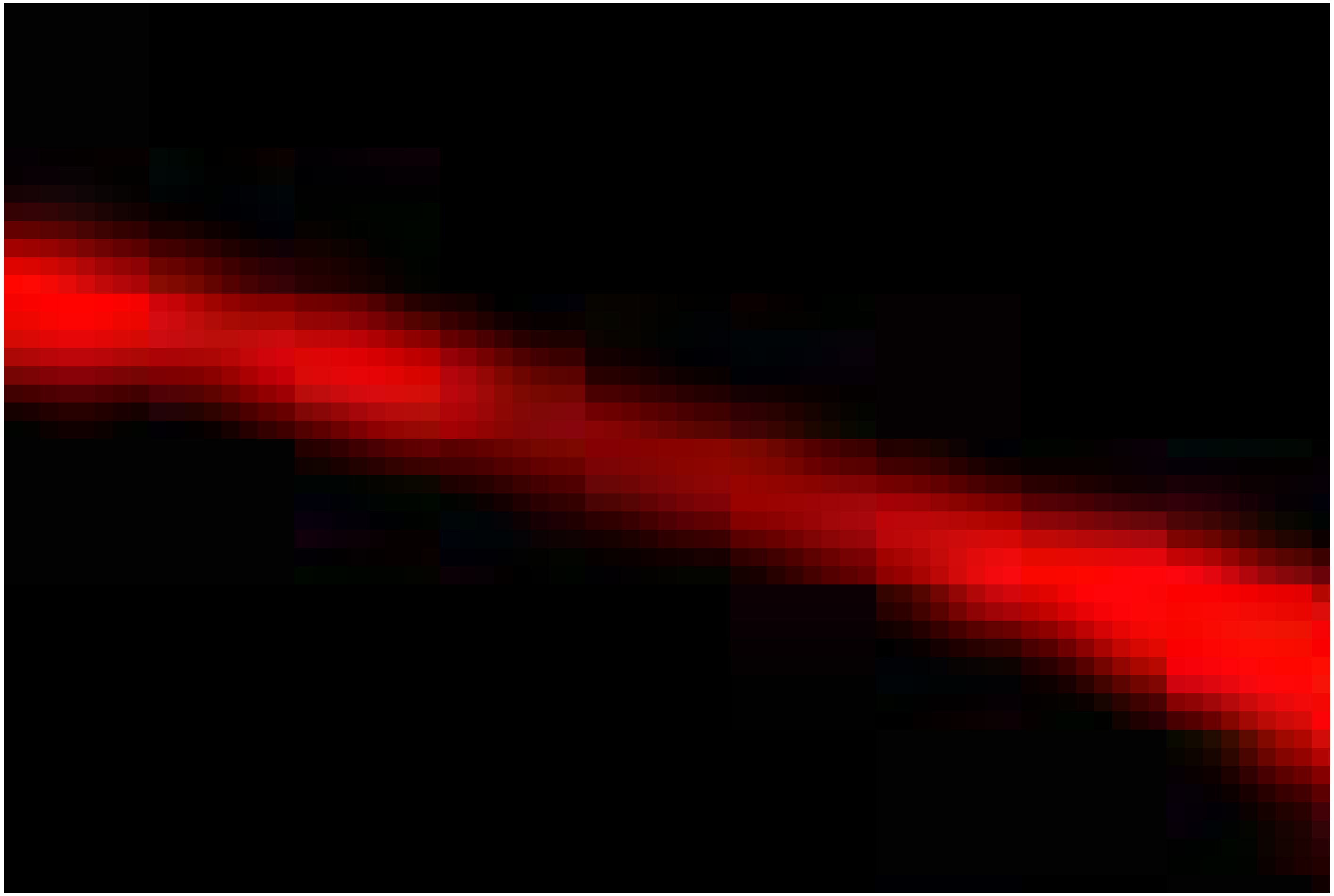
Anu George et al. *ACS Applied Materials & Interfaces*, 2012

Approaching detection limits of tens of Hg^{2+}

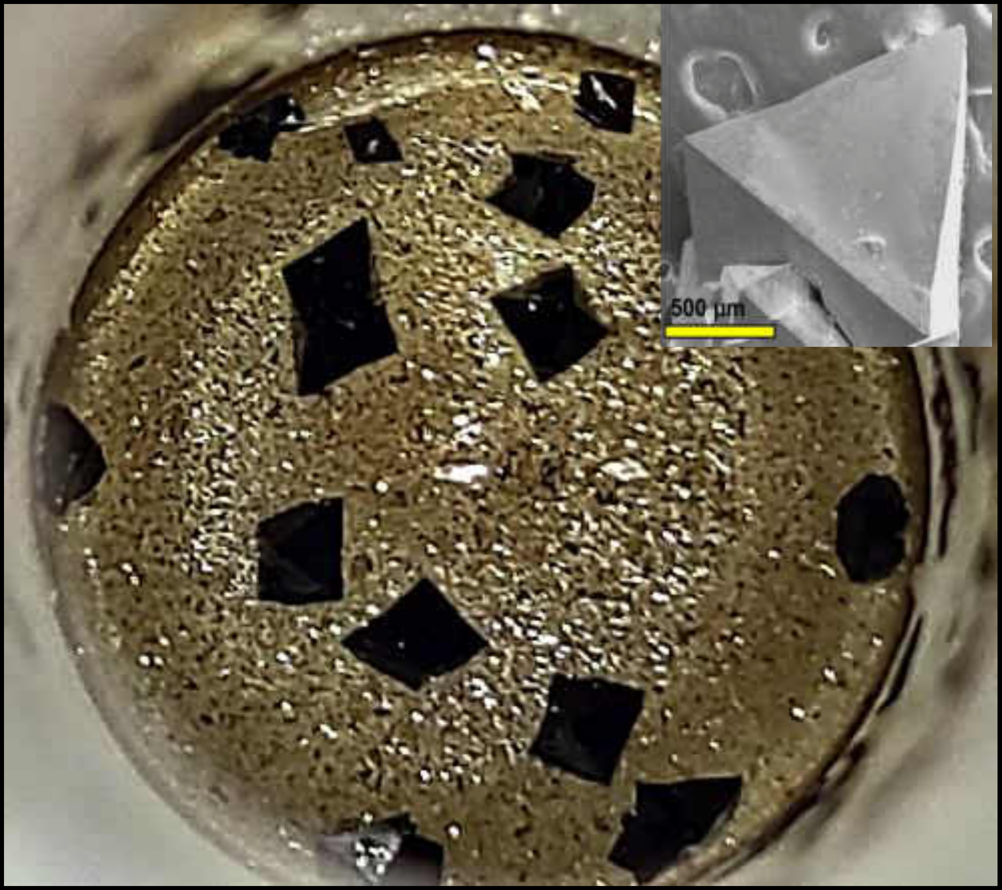


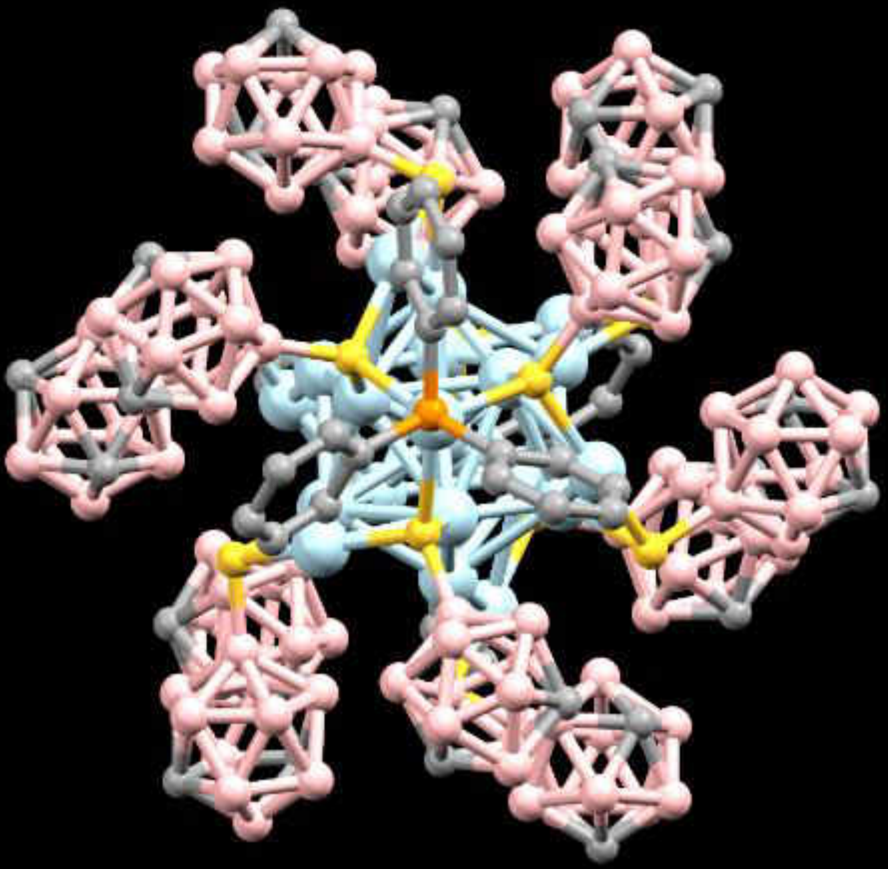
Atanu Ghosh et al. Anal. Chem.2014.

Video of mercury quenching experiment using the nanofiber



Clusters stable up to 300 °C!





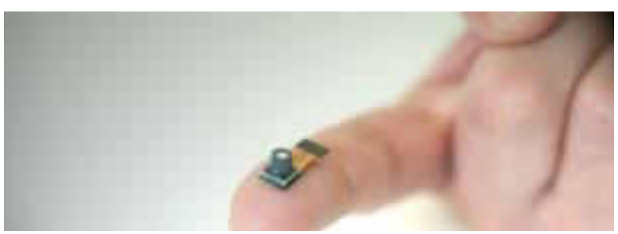
With Tomas Base

Jana *et*. *al*, Inorganic Chemistry (2022)

Sensors and new opportunities

Analog/Grating Equipment
$ 5~6 Billion (2017)
a few **100k units (2017)**

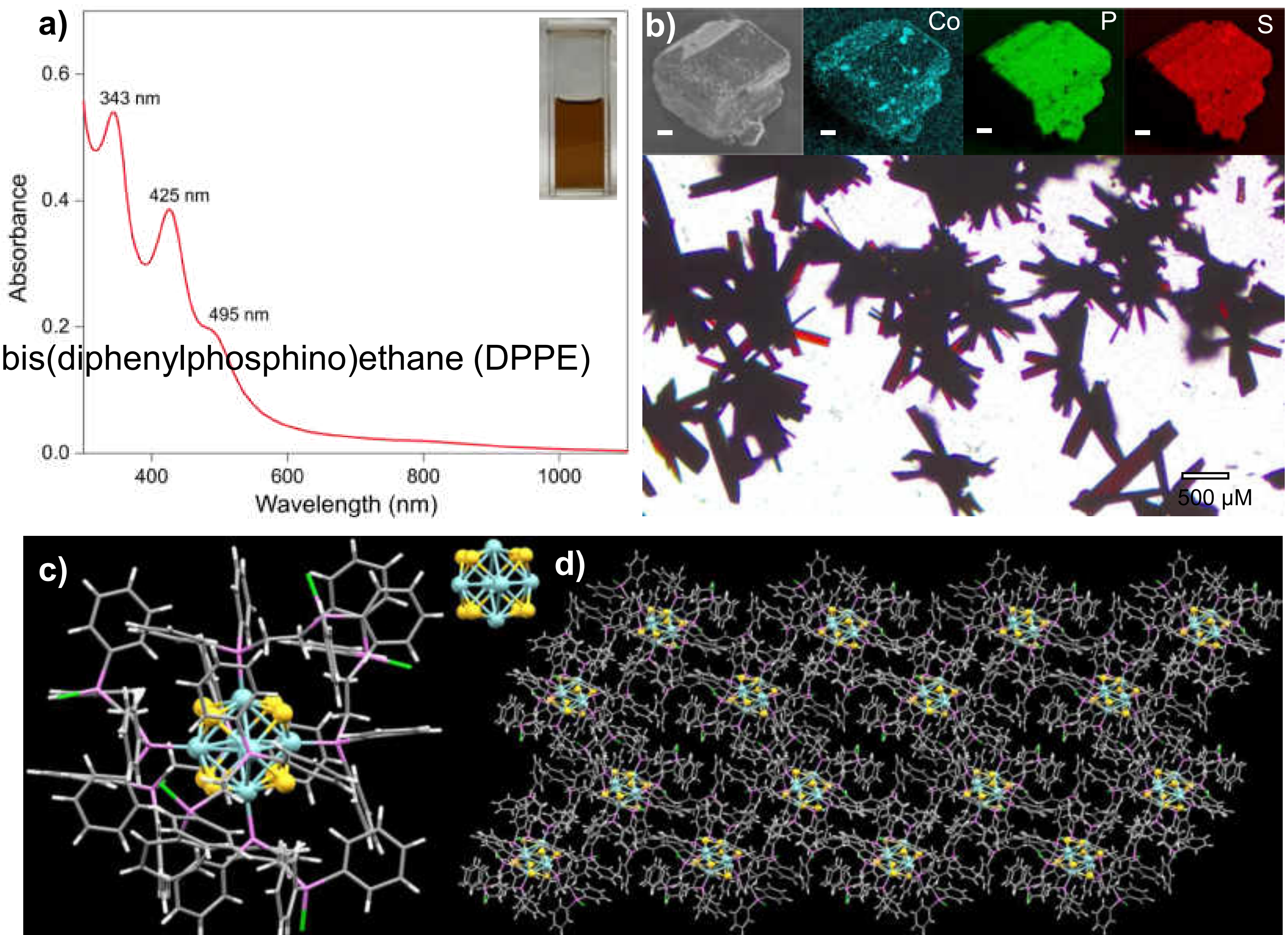
Ultra compact Low Cost Spectral Sensor Module
~ Billions units (? 2027)



Water quality measurement – In the pipeline

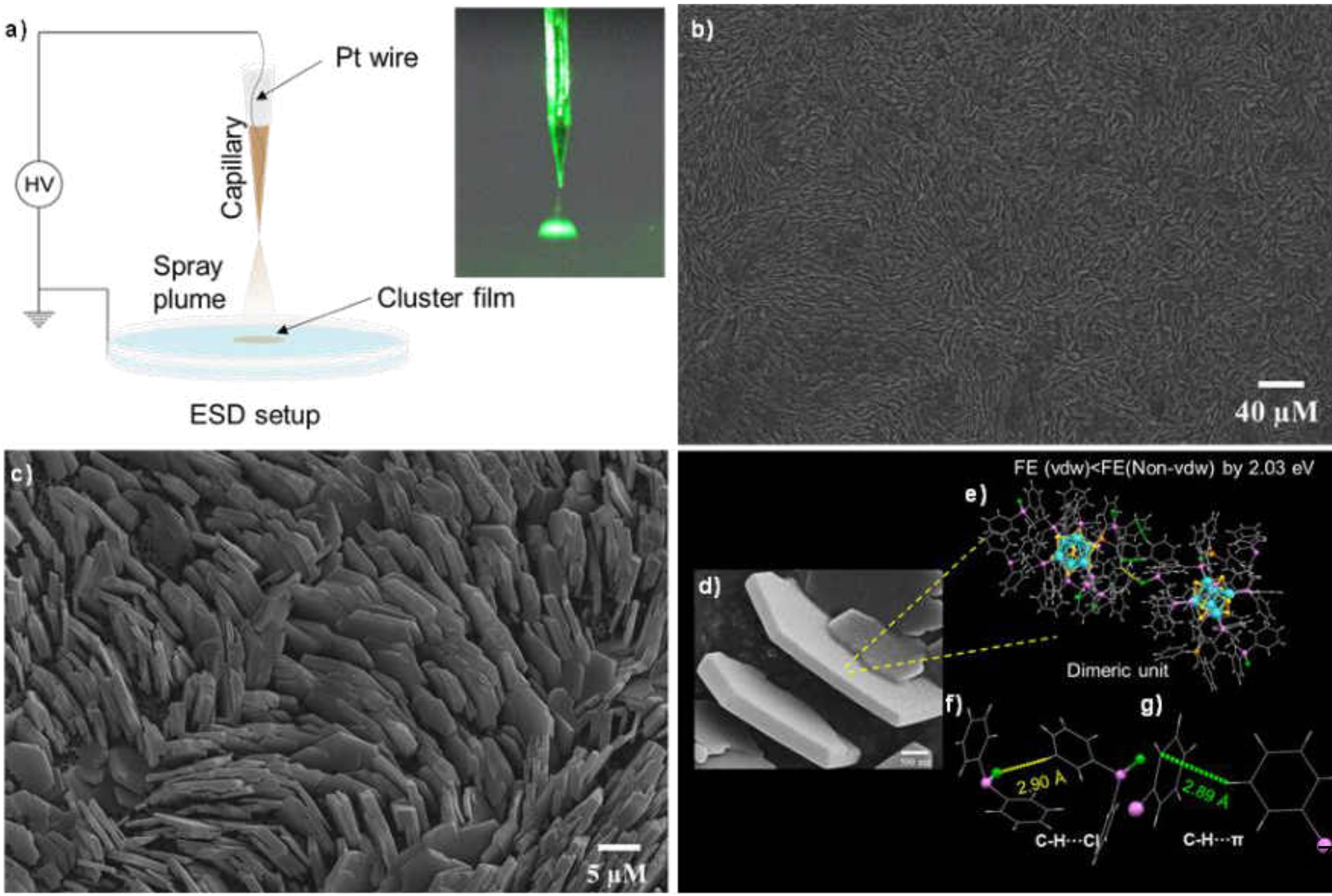
nanoλ

New electrodes - Aligned nanoplates of Co_6S_8

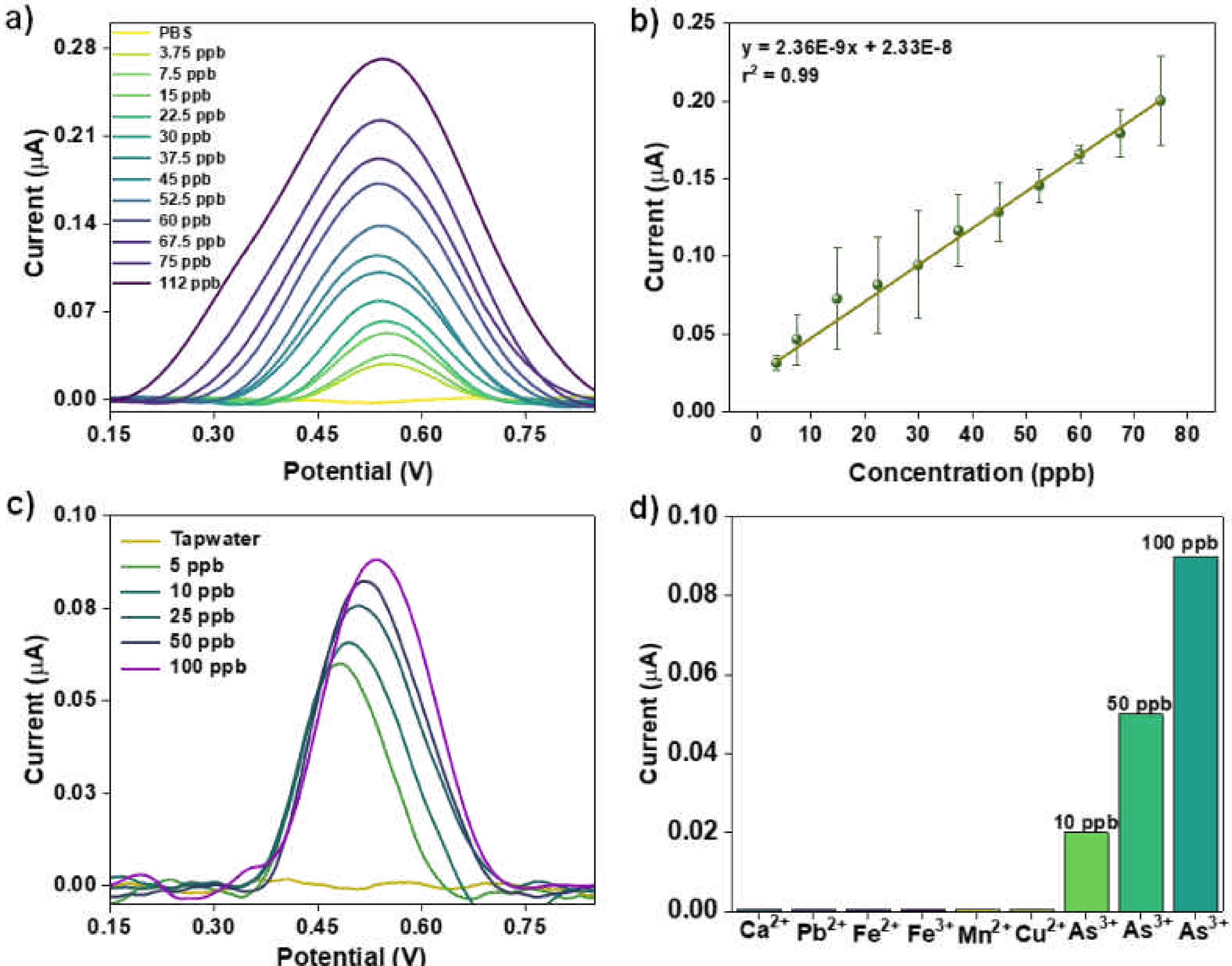


1,2-bis(diphenylphosphino)ethane (DPPE)

Electrospray deposition

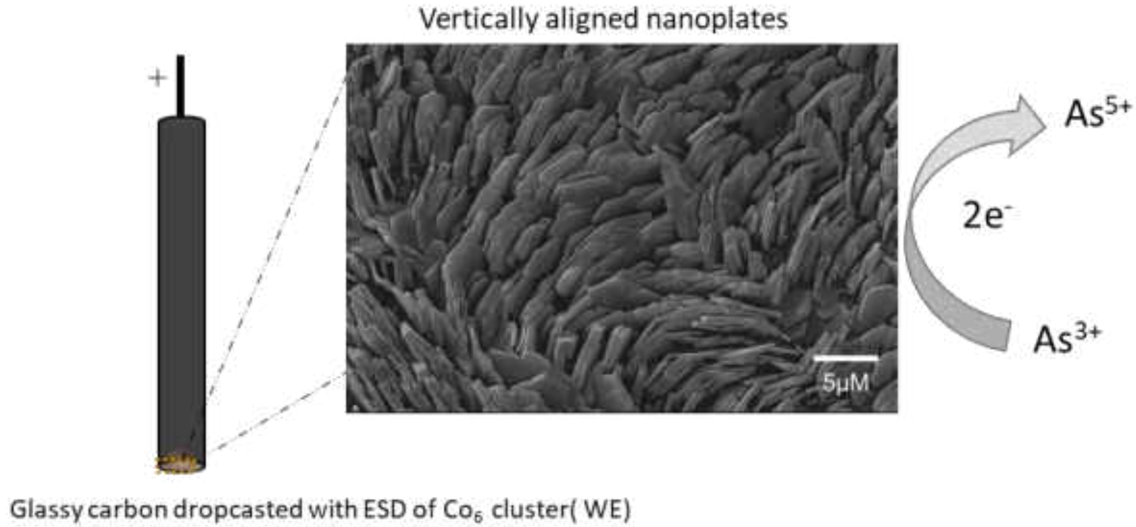


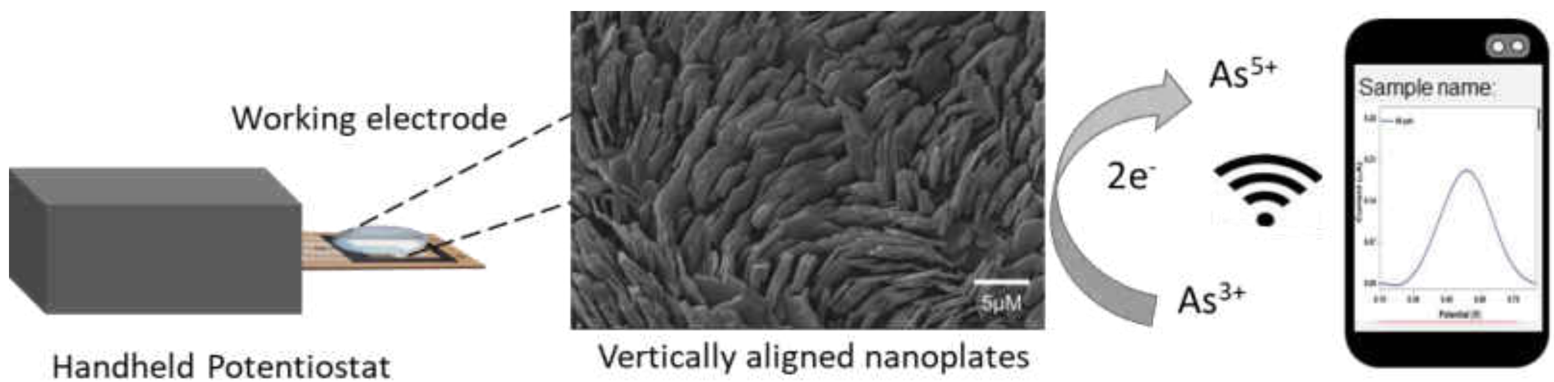
Sensing



a)
PBS
3.75 ppb
7.5 ppb
15 ppb
22.5 ppb
30 ppb
37.5 ppb
45 ppb
52.5 ppb
60 ppb
67.5 ppb
75 ppb
112 ppb
Current (μA)
Potential (V)
b)
y = 2.36E-9x + 2.33E-8
r² = 0.99
Current (μA)
Concentration (ppb)
c)
Tapwater
5 ppb
10 ppb
25 ppb
50 ppb
100 ppb
Current (μA)
Potential (V)
d)
100 ppb
50 ppb
10 ppb
Current (μA)
Ca²⁺ Pb²⁺ Fe²⁺ Fe³⁺ Mn²⁺ Cu²⁺ As³⁺ As³⁺ As³⁺

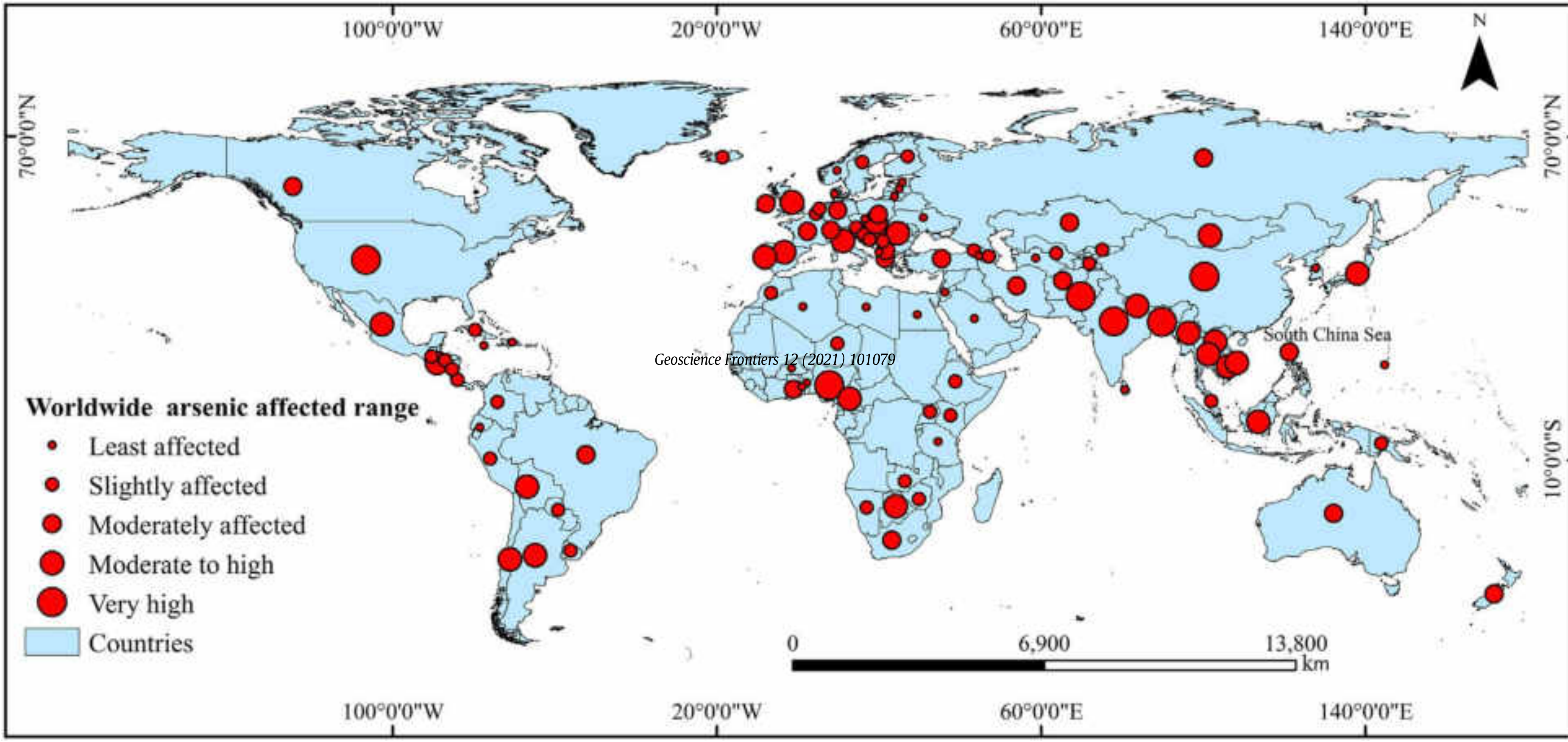
Working electrode





Anagha Jose et al. *ACS Materials Lett.*, 5 (2023) 893–899.

Arsenic poisoning across the world

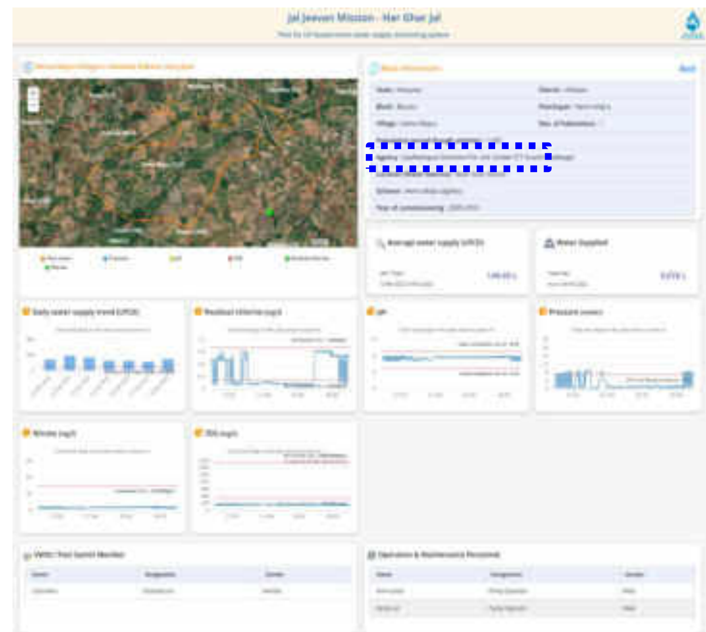


E. Shaji, M. Santosh, K. V. Sarath, Pranav Prakash, V. Deepch and B. V. Divya, Geoscience Frontiers, 12 (2021) 101079.

India's water is being monitored

IITM/IISc

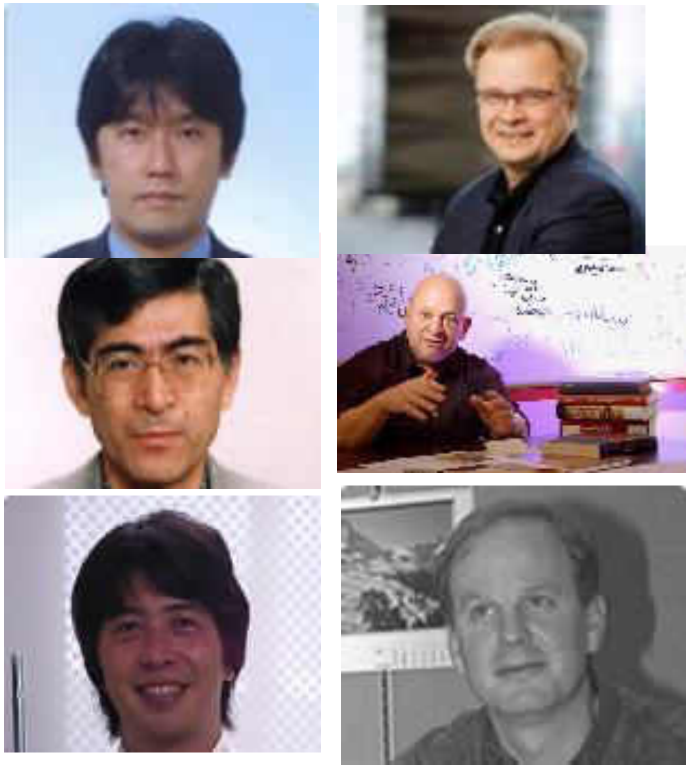
Installations made by four companies



International Centre for Clean Water


IIT Madras Research Park

Collaborators



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Rob Whetten
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Horst Hahn

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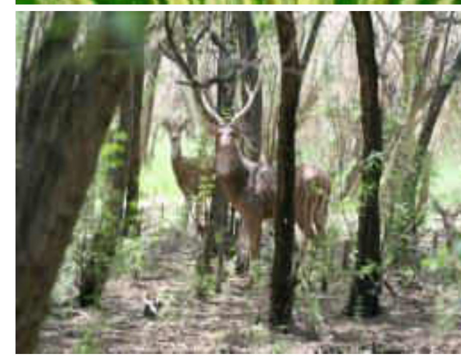
Vivek Polshettiwar

Indian Institute of Technology Madras



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