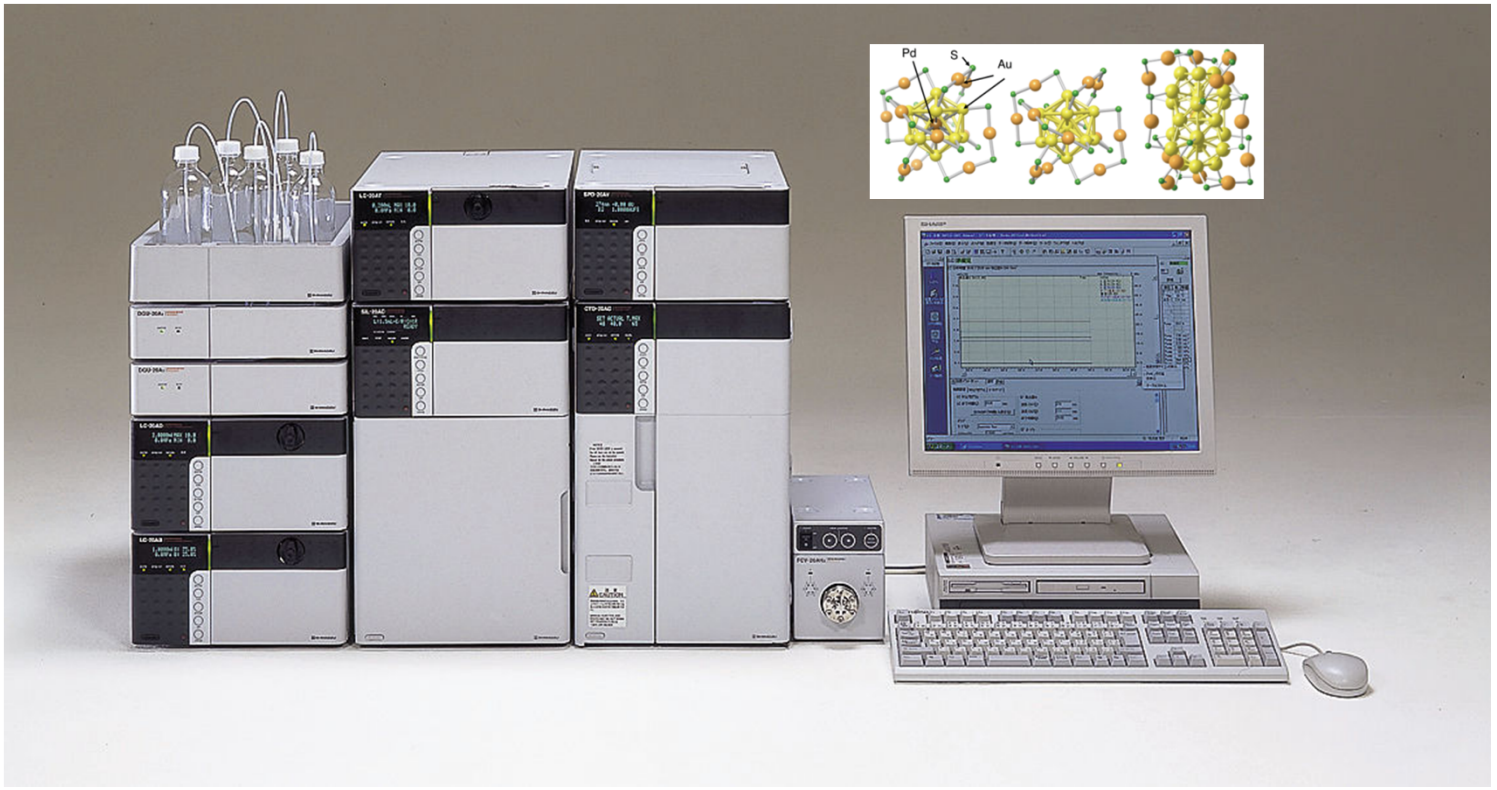
HPLC OF CLUSTERS



SHRIDEVI S BHAT

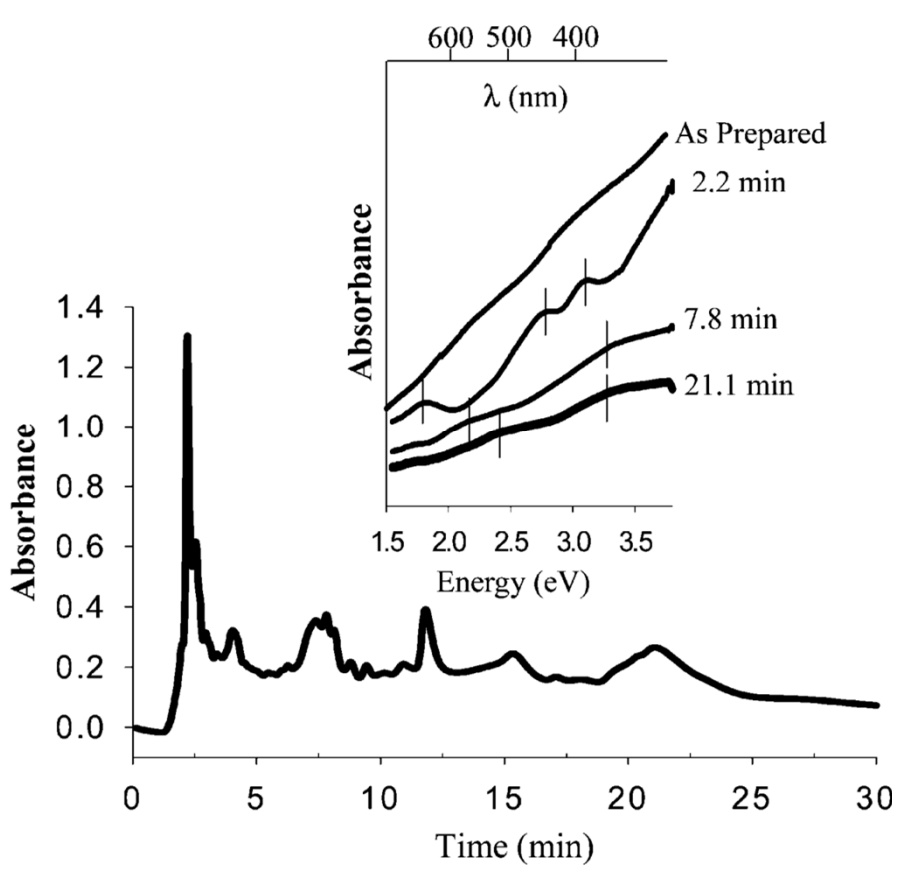
27/09/2014

Anal. Chem. **2003**, *75*, 199–206

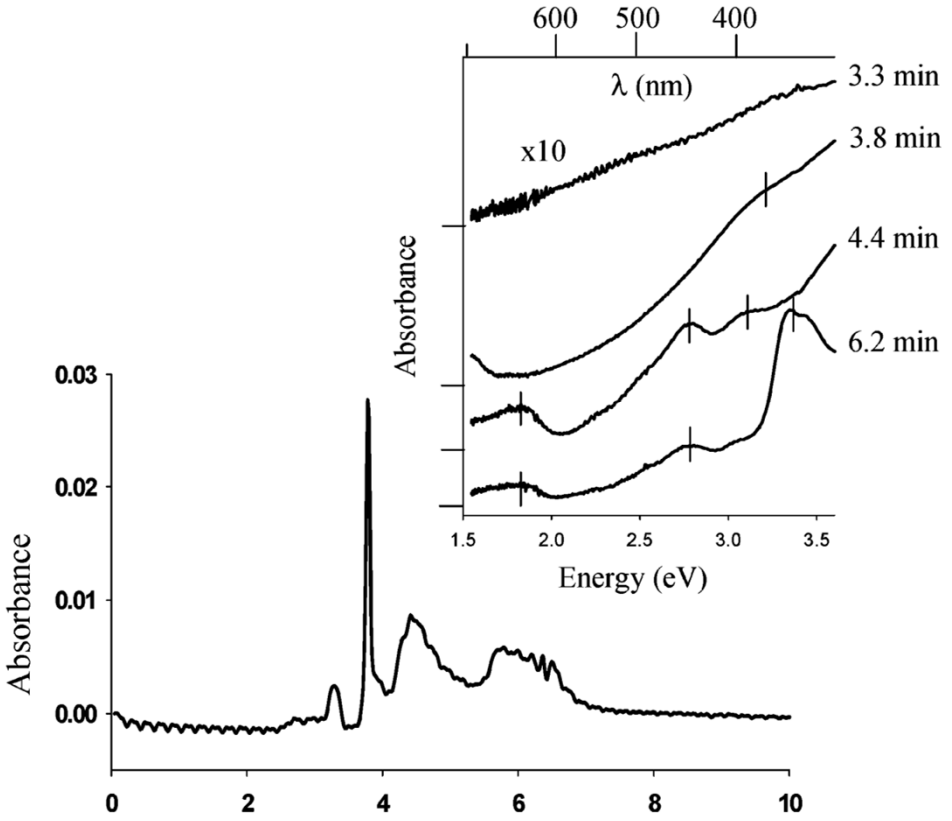
HPLC of Monolayer-Protected Gold Nanoclusters

Victoria L. Jimenez, Michael C. Leopold,† Carolyn Mazzitelli, James W. Jorgenson, and Royce W. Murray*

- **Au nanoparticles protected with monolayers of hexanethiolate ligands and with mixed monolayers of hexanethiolate and mercaptoundecanoicacid have been chromatographically**
- **The stainless steel silica bonded C8 columns**
- **Acetone/toluene mobile phase at a flow rate of 1 mL/min (or 0.6 mL/min)**
- **PDA and UV – vis detectors**



Chromatogram (absorbance detected at 400 nm) of C6MPC. The mobile phase was 66% acetone/34% toluene at a flow-rate of 1 mL/min.

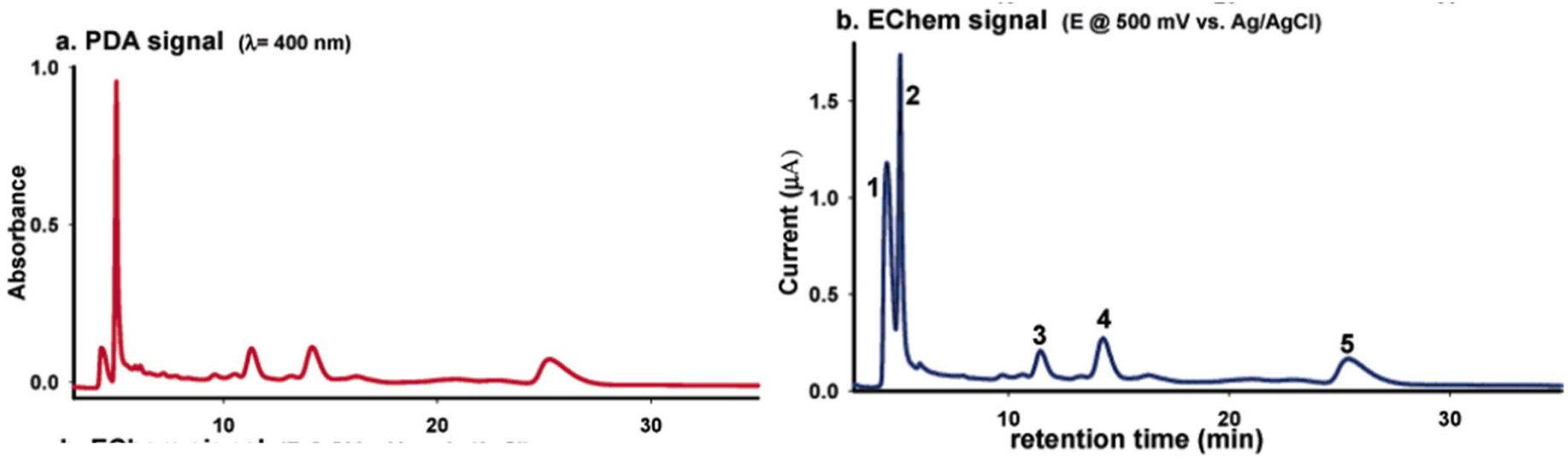


Chromatogram (absorbance detected at 400 nm) mixed monolayer MPCs. The mobile phase was 85% acetone/15% toluene at a flow-rate of 1 mL/min.

Anal. Chem. **2003,** *75,* 5088−5096

Estimation of Size for 1−2 nm Nanoparticles Using an HPLC Electrochemical Detector of Double Layer Charging

Yang Song, Victoria Jimenez, Collin McKinney, Robert Donkers, and Royce W. Murray*



Chromatograms detected by photodiode array (PDA) detector (a) and thin-layer flow cell electrochemical detector (EC) (b) for a 100 µL injection of 10-5 M hexanethiolate MPCs (C6 MPCs) with 10 mM tetrabutylammonium perchlorate. The mobile phase was 66% acetone and 34% toluene. Part 1a was taken at 400 nm wavelength for the best resolution; part b was taken at 500 mV vs Ag/AgCl cell potential.

Anal. Chem. 2009, *81*, 1676–1685

Application of HPLC and MALDI-TOF MS for Studying As-Synthesized Ligand-Protected Gold Nanoclusters Products

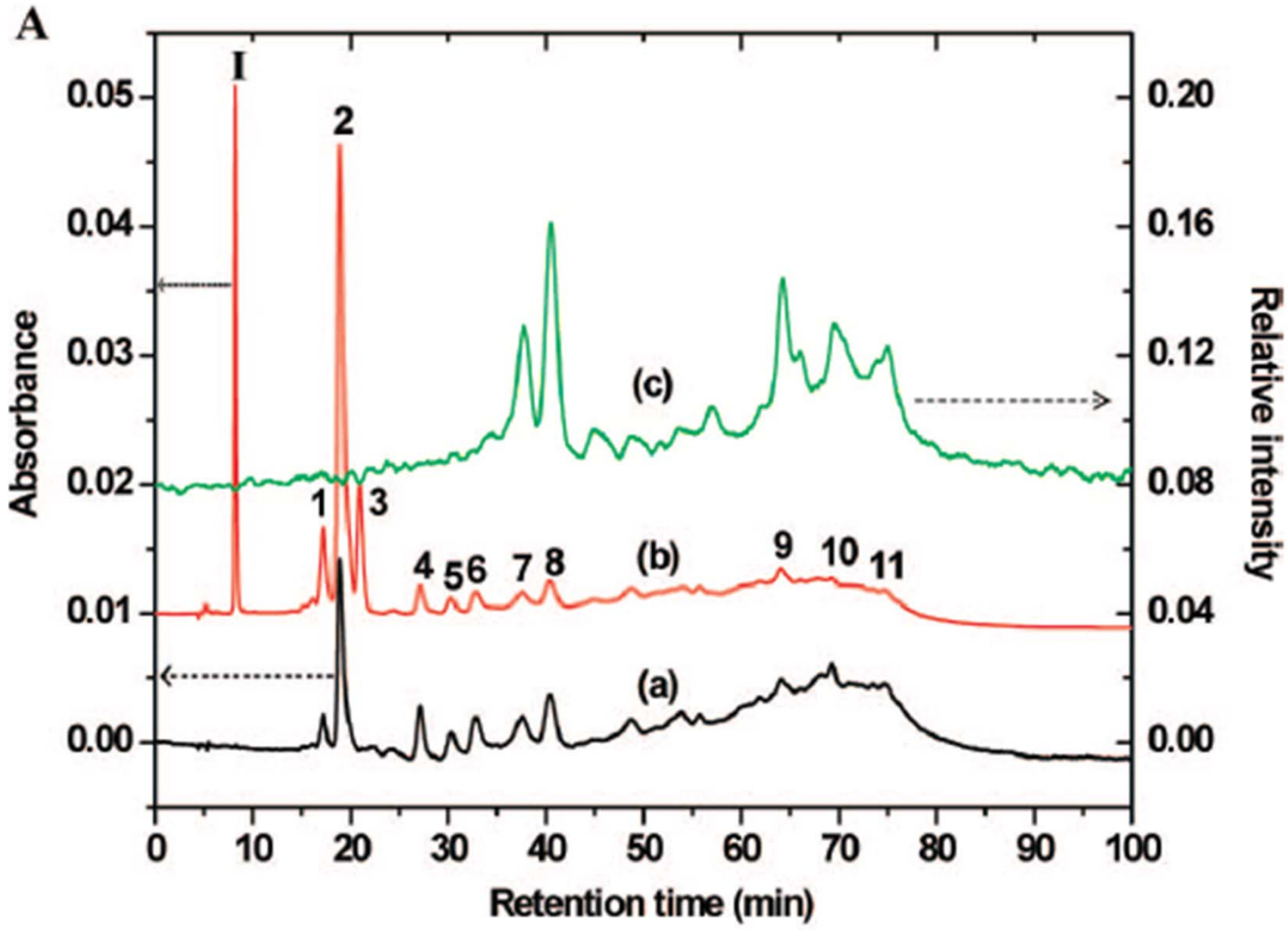
Yan Zhang,[†,§] Shaomin Shuang,[*,†] Chuan Dong,[†] Chung Keung Lo,[‡] Man Chin Paau,[‡] and Martin M. F. Choi[*,‡]

- Samples of polydisperse gold nanoclusters (AuNCs) protected with monolayers of N-acetyl-L-cysteine (NAC) have been chromatographically separated

- C18 column (250 mm × 4.6 mm i.d. stainless steel, with 5 μm C18 bonded silica 130 Å pore size)

- A gradient elution program was used at a flow rate of 0.70 mL/min, in which eluent I was 50 mM Bu4N+F- and 2.0 mM NaCl in 65% v/v MeOH and eluent II was pure MeOH. The elution program was applied as follows: 100% I (0% II) for 40 min, linearly decreased to 80% I from 40 to 70 min, maintained at 80% I from 70 to 90 min, linearly decreased to 50% I from 90 to 120 min, and kept at 50% I from120 to 150 min.

- PDA, fluorescence and electrochemical detectors have been used



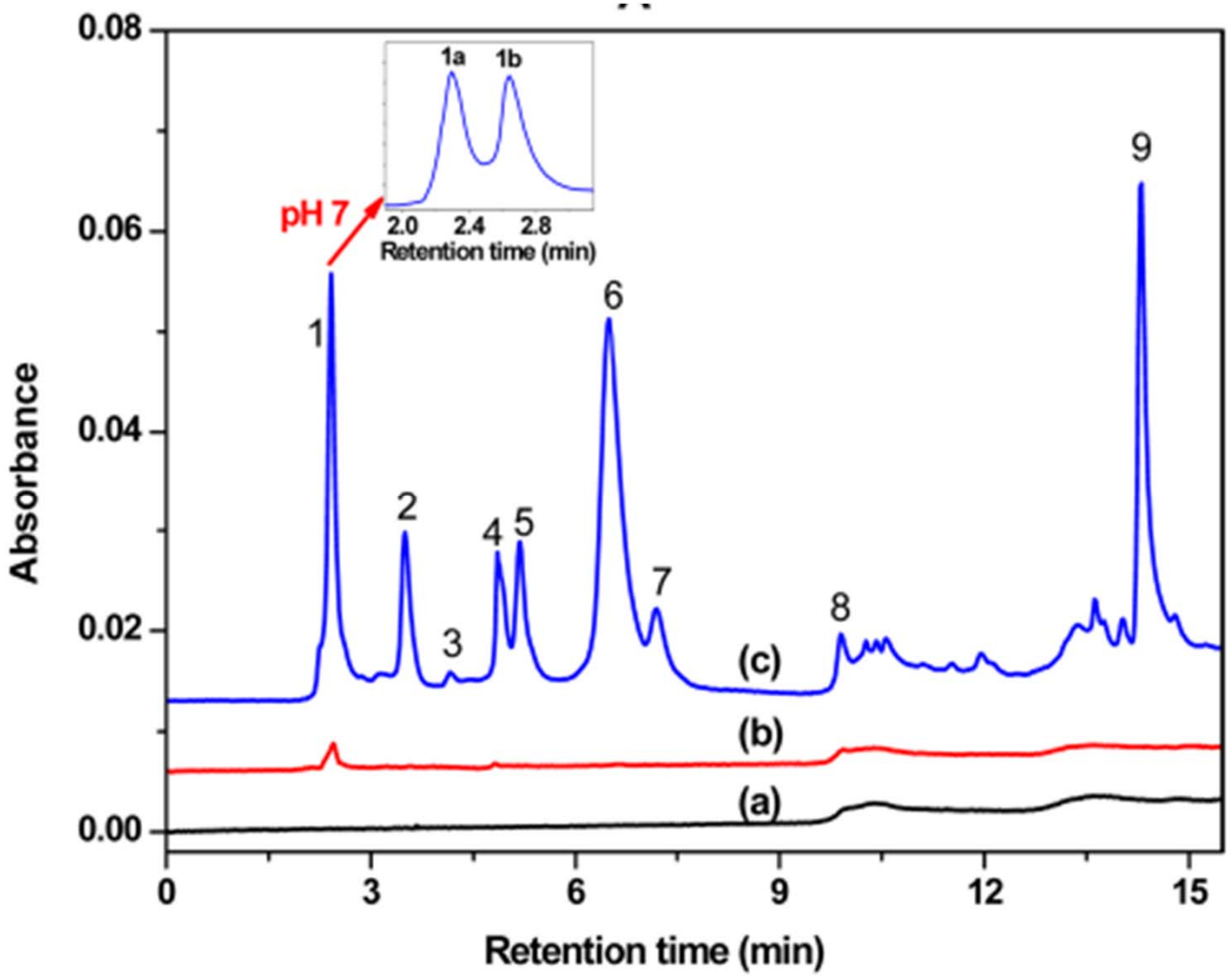
Chromatographic separation of a 3000 mg/L polydisperse AuNC sample. Curves a (black) and b (red) are absorbance chromatograms monitored by a PDA at 400 and 250 nm, respectively, and curve c (green) is a fluorescence chromatogram monitored by a fluorescence detector at excitation/ emission 400/680 nm.

Probing Histidine-Stabilized Gold Nanoclusters Product by High-Performance Liquid Chromatography and Mass Spectrometry

Yan Zhang,[†,∥] Qin Hu,[‡] Man Chin Paau,[‡] Shunping Xie,[‡,⊥] Pengfei Gao,[†] Wan Chan,[*,§] and Martin M. F. Choi[*,‡]

- **Gold nanoclusters stabilized with histidine (His-AuNCs) with core diameter smaller than 1 nm are separated by HPLC**
- **C18 column (150 mm × 4.6 mm i.d. stainless steel, with 5 μm C18 bonded silica 300 Å pore size)**
- **The mobile phase consisted of ammonium acetate in water (pH 5) and MeOH. Flow rate 0.7 mL/min. A solvent program was applied as follows: 100% A for 5.5 min, linear decreased to 90% A from 5.5 to 6.5 min, maintained at 90% A from 6.5 to 9.0 min, linear decreased to 80% A from 9.0 to 10 min, maintained at 80% A from 10 to 12 min, linear decreased to 60% A from 12 to 13 min, and kept at 60% A from 13 to 16 min.**
- **PDA detector**

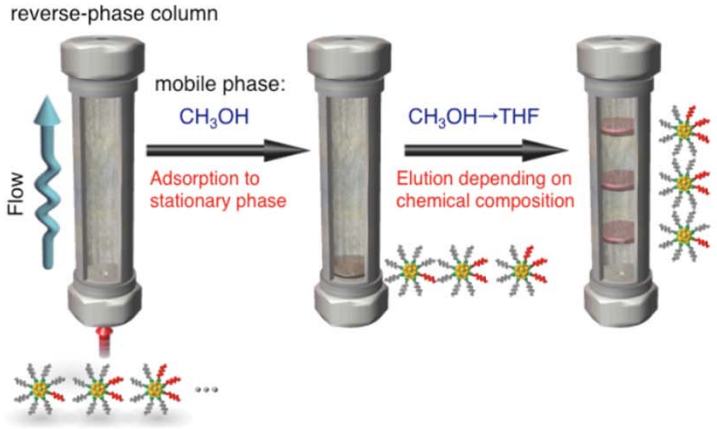
dx.doi.org/10.1021/jp405013m | *J. Phys. Chem. C* 2013, 117, 18697−18708

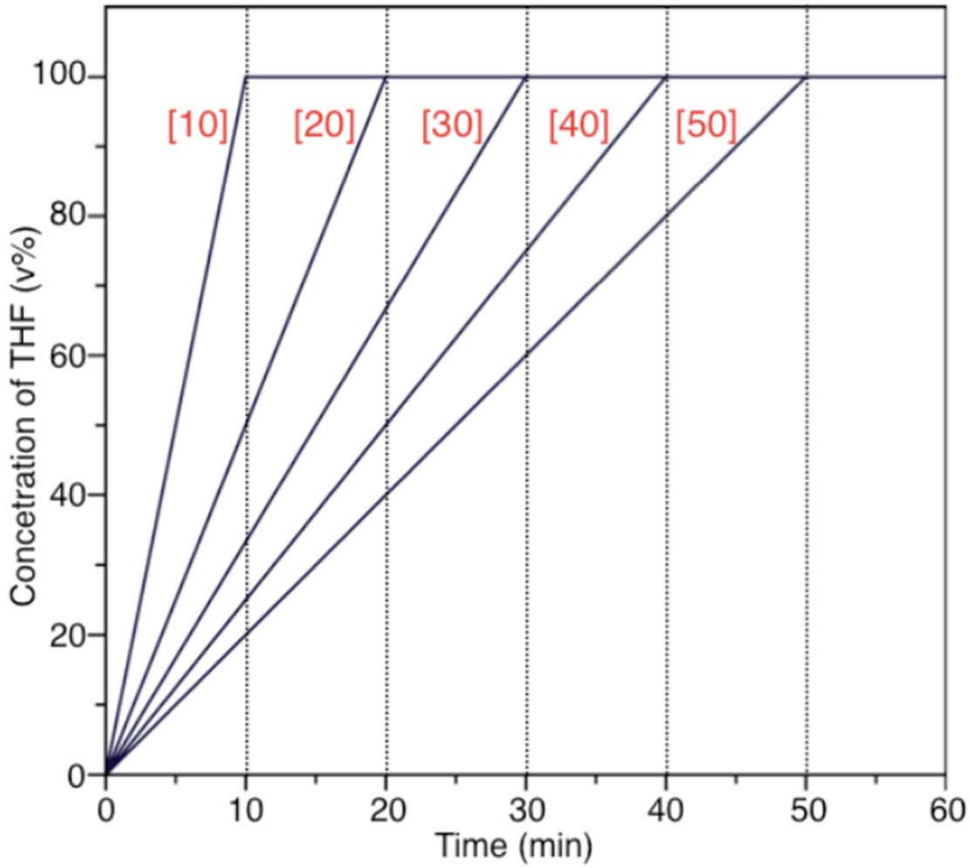


Chromatographic separation of 15.0 mg/mL His- AuNCs sample under gradient elution. Curve a (black) is water injection chromatogram and curves b (red) and c (blue) are 15.0 mg/mL histidine and His-AuNCs chromatograms monitored at 250 nm, respectively. Chromatograms are offset for clarity. The inset displays the separation of Peak 1 into two peaks (labeled as 1a and 1b) at pH 7.0.

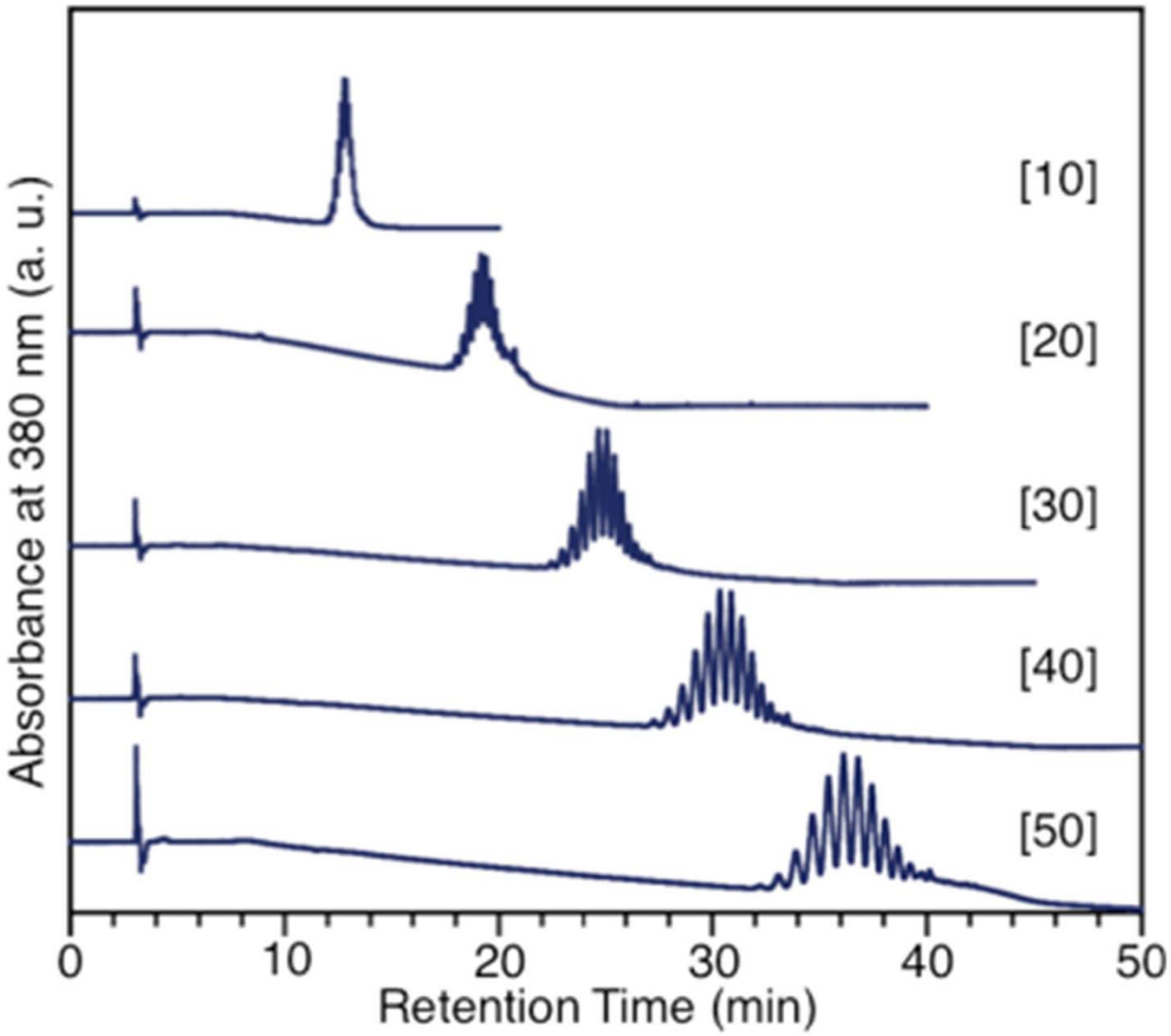
Separation of Precise Compositions of Noble Metal Clusters Protected with Mixed Ligands

Yoshiki Niihori,[†] Miku Matsuzaki,[†] Thalappil Pradeep,*[,‡] and Yuichi Negishi*[,†]

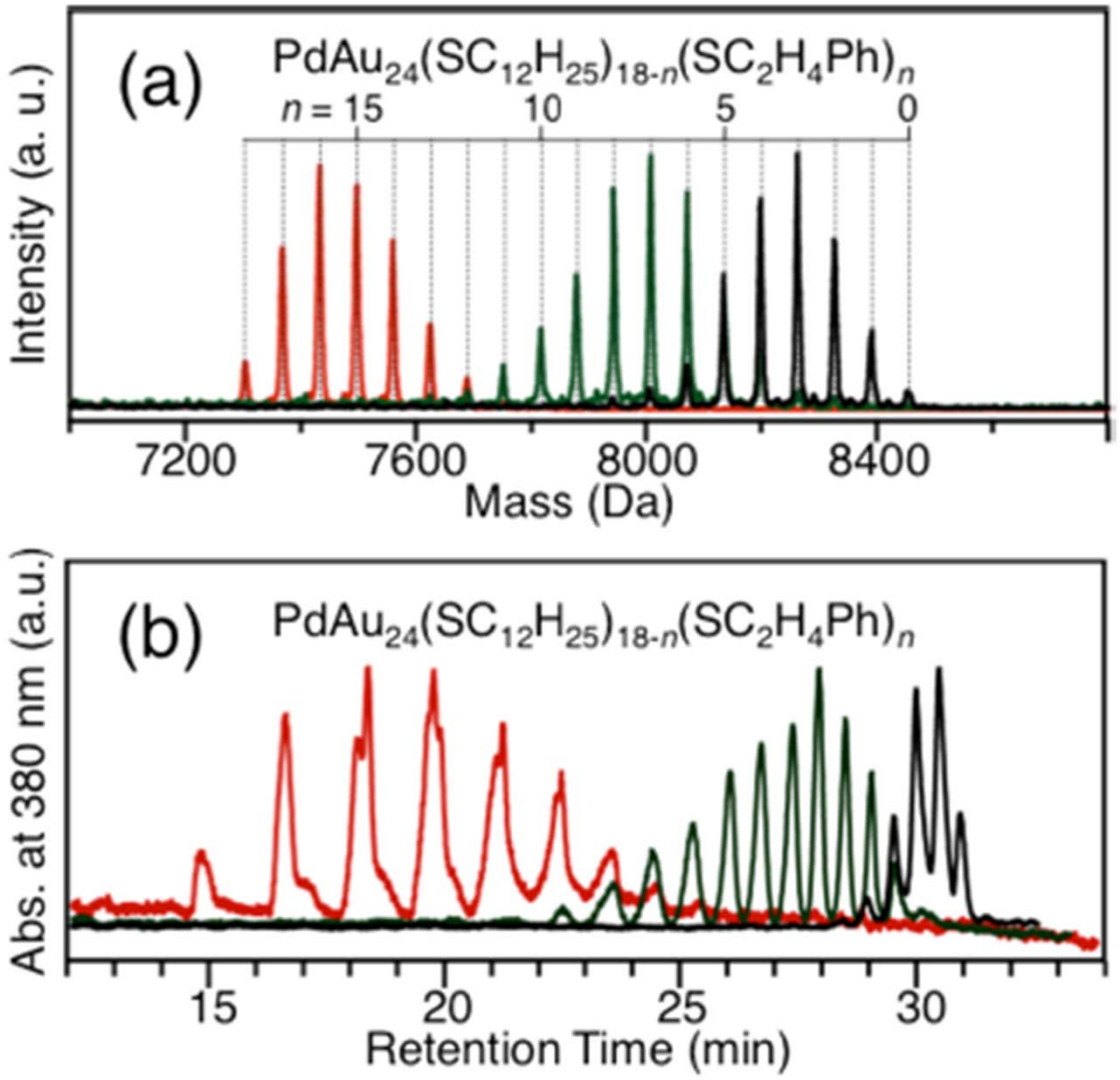




dx.doi.org/10.1021/ja4009369 | J. Am. Chem. Soc. 2013, 135, 4946−4949



Chromatogr ams of $PdAu_{24}(SC_{12}H_{25})_{18-n}(SBB)_n$ (n = 6–16) at each gradient program. The peak observed at 3.0 min was confirmed to be due to the solvent, THF.



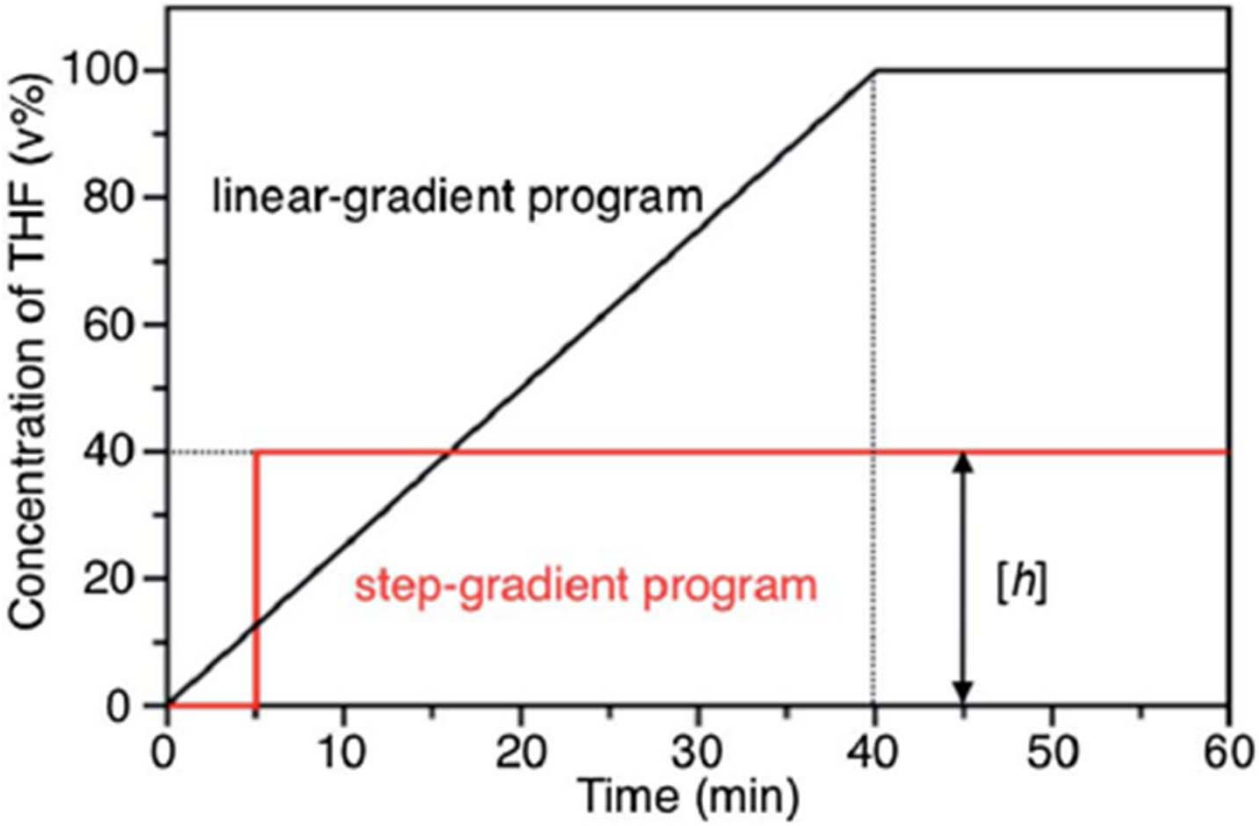
Comparison between the MALDI mass spectrum and chromatogram for $PdAu_{24}(SC_{12}H_{25})_{18-n}(SC_2H_4Ph)_n$.

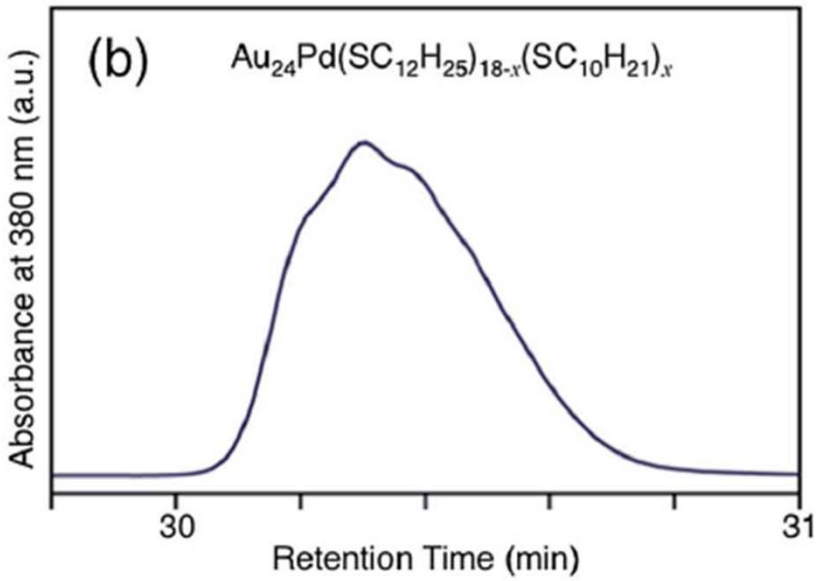
Advanced use of high-performance liquid chromatography for synthesis of controlled metal clusters†

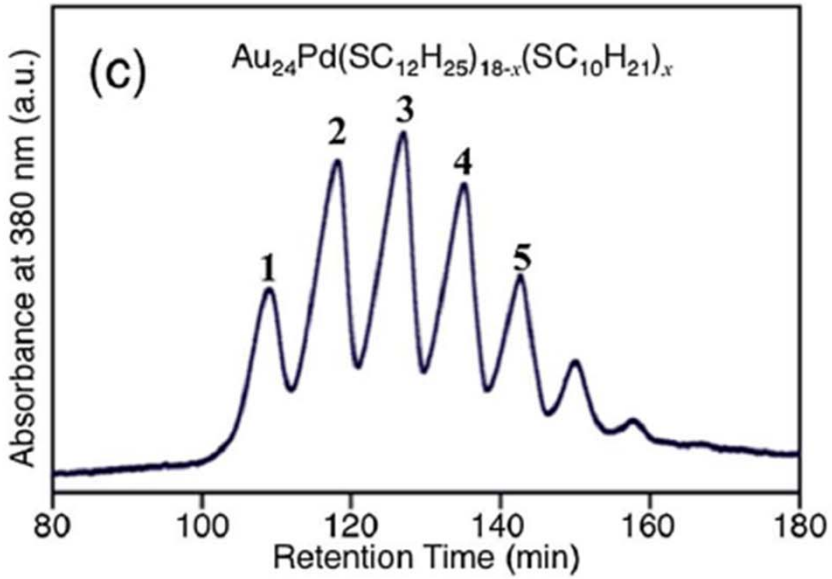
Yoshiki Niihori,[a] Miku Matsuzaki,[a] Chihiro Uchida[a] and Yuichi Negishi*[ab]

Table 1 Ligand combinations applied in this study

Entry	SR_1	MW_{av}[a]	SR_2/SeR_2	MW_{av}[a]
1	$SC_{12}H_{25}$	201.39	SC_8H_{17}	145.29
2	$SC_{12}H_{25}$	201.39	SC_6H_{13}	117.23
3	$SC_{12}H_{25}$	201.39	SC_4H_9	89.18
4	$SC_{12}H_{25}$	201.39	SCH_2Ph^tBu[b]	179.30
5	$SC_{12}H_{25}$	201.39	SCH_2PhBr[c]	202.09
6	$SC_{12}H_{25}$	201.39	SC_2H_4Ph	137.22
7	SC_2H_4Ph	137.22	$SC_{14}H_{29}$	229.45
8	SC_2H_4Ph	137.22	$SC_{10}H_{21}$	173.34
9	SC_2H_4Ph	137.22	SC_6H_{13}	117.23
10	SC_2H_4Ph	137.22	$SeC_{12}H_{25}$	248.29
11	$SC_{12}H_{25}$	201.39	$SeC_{12}H_{25}$	248.29


Concentration of THF (v%)
100
80
60
40
20
0
linear-gradient program
step-gradient program
[h]
0
10
20
30
40
50
60
Time (min)





Chromatogram obtained with a linear gradient and chromatogram obtained with a step gradient for $Au_{24}Pd(SC_{12}H_{25})_{18-x}(SC_{10}H_{21})_x$ (x = 12–18)

THANK YOU