Supplemental Material

Microwave-Assisted Catalytic Acetylation of Alcohols by Gold

Nanoparticle-Supported Gadolinium Complex

Tsao-Ching Chang, and Shuchun Joyce Yu *

Department of Chemistry and Biochemistry, National Chung Cheng University,

Ming Hsiung, Chia Yi 621, TAIWAN, ROC

chejyy@ccu.edu.tw

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1. General Information

All chemicals were used as supplied without further purification. ^1H NMR spectra were recorded in CDCl_3 and methanol- d_4 on a 400 MHz spectrometer (Bruker DPX-400), and coupling constants are reported in Hz. Chemical shifts are given in ppm relative to TMS (tetramethyl silane). IR (Perkin-Elmer 682), Gas Chromatography (GC, Agilent 6890N Network), TEM (JEOL JEM-2010) and XPS (Kratos Axis Ultra DLD) were obtained in-house at National Chung Cheng. Electron paramagnetic resonance spectrometer (BRUKER, E580) and Inductively coupled plasma-atomic emission spectrometer (Kontron, S-35) were obtained at National Tsinghua University.

2. General Procedures and Characterization Data of Compounds and Au Nanoparticles

Tris-(tert-butoxycarbonylmethyl)-1,4,7,10-tetraazacyclododecane (L1)^[1]

3.5 equiv. of appropriate tert-Butyl bromoacetate (1.2 mL, 7.6 mmol) dissolved in 10.0 mL of anhydrous chloroform was added dropwise to a mixture of 1,4,7,10-tetraazacyclododecane (cyclen) (400.0 mg, 2.32 mmol) and 10.0 equiv. triethylamine (2.3 g, 23.2 mmol) in 40 mL of anhydrous chloroform under N_2 . The reaction mixture was stirred for a further 16-20 h. The resulting solution was washed

by water (40 mL $\times$ 3), and the organic phase was dried by Na₂SO₄. The solvent was removed, and the crude products were purified by column chromatography on Al₂O₃ to afford the products **3** (437 mg, 36.5%). ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.46 (s, 9H), 1.48 (s, 18H), 2.88-2.93 (m, 12H), 3.11 (br, 4H), 3.29 (s, 2H), 3.97 (s, 4H).

11-(Bromoundecyl)-thioacetate (L2)^[2]

A solution of 11-Bromo-1-undecene (5 g, 0.021 mol), thioacetic acid (10 mL, 0.12 mol) and AIBN (1.5 g, 0.09 mol) in dry toluene (50 mL) under N₂. The reaction was run and refluxed for 2 h. The result solution was washed with excess water and the crude product was purified by column chromatography on silica gel to yield product **2** (4.05 g, 81%). ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.27-1.43 (m, 14H), 1.53-1.59 (m, 2H), 1.82-1.88 (m, 2H), 2.32 (s, 3H), 2.86 (t, J = 7.3 Hz, 2H), 3.42 (t, J = 6.7 Hz, 2H).

1,4,7-tris(tert-butoxycarbonylmethyl)-10-(11-(acetylthioundecyl)-1,4,7,10-tetraazacyclododecane (L3)^[3]

To a solution of **L1** (231 mg, 0.45 mmol) in dry CH₃CN (40 mL) was added **L2** (125 mg, 0.4 mmol) and K₂CO₃ (620 mg, 4.50 mmol), and the mixture was heated at 60 °C for 24 h. The resulting mixture was filtered and the filtrate was evaporated to dryness

under reduced pressure. The residue was dissolved in hexane (20 mL) and the resulting solution was washed with H₂O (10 mL × 5) and brine. The organic layer was dried with Na₂SO₄, filtered, and concentrated in vacuo to afford the product **L3** (233 mg, 70%). ¹H NMR (400 MHz, CDCl₃), δ (ppm): 1.11-1.26 (m, 16H), 1.37-1.54 (m, 29H), 2.15 (s, 3H), 2.78-2.84 (m, 16H), 3.24-3.29 (m, 6H), 4.34 (br, 2H).

[1,4,7-tris(carboxymethyl)-10-(11-Mercaptoundecyl)-1,4,7,10-tetraazacyclododecane(DO3ASH, 1)]^[4]

Acetyl chloride (0.45 mL, 6.2 mmol) was added dropwise to the solution of dry methanol (2 mL) and **L3** (230 mg, 0.31 mmol) in an ice bath. The solution was allowed to warm to room temperature and the reaction progressed for 24 h. The solvent was removed by rotary evaporation, and the residue was dissolved in minimal CHCl₃ before the addition of a large quantity of ether to precipitate light yellow powder **1** (128 mg, 78 %). ¹H NMR (400 MHz, MeOH-*d*₄), δ (ppm): 1.32-1.48 (m, 16H), 1.58-1.60 (m, 2H), 2.49 (t, *J* = 7.2 Hz, 2H), 3.09-3.70 (m, 16H), 3.85-3.86 (m, 6H), 4.35 (br, 2H,) (see Figure S1). IR (KBr) ν (cm⁻¹): 1582 (coordinated COO⁻) (see Figures S1 and S2).

Synthesis of various thiolate-protected AuNPs and octanethiolate- protected

AuNPs (Au-SR)^[5]

To a solution of tetraoctylammonium bromide (500 mg, 0.1 mmol) in CHCl_3 (40 mL) was added a solution of tetrachloroauric acid (200 mg, 0.5 mmol) in water (15 mL). The mixture was stirred vigorously for 30 min to ensure complete transfer of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$, at which point the decoloration of the aqueous phase took place while the organic phase turned orange-brown. After this, a mixture of 1-dodecanethiol (274 mg, 1.4 mmol) in CHCl_3 (20 mL) was added to the solution. After 10 min, a freshly prepared aqueous solution of NaBH_4 (200 mg, 5.5 mmol in 20 mL of H_2O) was added dropwise to the reaction mixture. The colloids formed instantaneously as indicated by the color change of the solution from orange to red and then to black-brown. After 12 h, the organic layer was collected. The colloidal solution was then concentrated to 3 mL, and 150 mL of ethanol was added to allow sitting at room temperature for 1 h for colloid precipitation. Precipitation from ethanol was carried out continuously until no more free ligand could be detected by ^1H NMR spectroscopy (CDCl_3 , no R-CH_2 resonance at ~ 2.49 ppm) and IR spectroscopy (absence of $\nu_{\text{asym(SH)}}$ band in the region of 2569 cm^{-1}). Centrifugation gave the colloids as black solids (75 mg). ^1H NMR (400 MHz, CDCl_3), δ (ppm): 0.88 (br, 3H,), 0.85 (br, 3H), 1.30-1.68 (m, 20H) (see Figure S1).

Synthesis of dodecanethiolate and DO3ASH-protected AuNPs (RS-Au-L, **2**)

To freshly prepared dodecanethiolate-protected **Au-SR** (155 mg) were dissolved in 50 mL of CHCl_3 . To this solution was added **1** (150 mg, 0.28 mmol), and the reaction mixture was stirred for 24 h at room temperature. After 24 h, the crude precipitates were subsequently centrifuged and washed with CHCl_3 for many times to remove unreacted ligand **1**. Finally, the AuNPs was dried under vacuo to afford the pure mixed dodecanethiolate- and DO3ASH-protected AuNPs **2** (200 mg). ^1H NMR (400 MHz, $\text{MeOH-}d_4$), δ (ppm): 0.93 (br, 3H), 1.30-1.68 (m, 56H), 3.09-3.70 (m, 16H), 3.85-3.86 (m, 6H), 4.35 (br, 2H) (see Figure S1). IR (KBr), ν (cm^{-1}): 1734 (non-coordinated COO^-) (see Figure S2). TEM: $3.09 \text{ nm} \pm 0.52$ (see Figure S3).

Synthesis of AuNP-supported Gd-complex (RS-Au-L-Gd, **3**)^[6]

A solution of AuNPs **2** (167 mg) and $\text{GdCl}_3 \cdot 6\text{H}_2\text{O}$ (154 mg, 0.20 mmol) in H_2O was maintained the pH value at 6-8 with NaOH. After stirring for 24 h at room temperature, the reaction mixture was filtered and the filtrate evaporated to dryness under reduced pressure. The residue was dissolved in minimal H_2O and added large quantity acetone to precipitate gadolinium(III)-Functionalized AuNPs **3** (130 mg). IR (KBr) ν (cm^{-1}): 1582 (coordinated COO^-), 1734 (non-coordinated COO^-) (see Figure S2). TEM: $2.99 \text{ nm} \pm 0.45$ (see Figure S4).

Synthesis of 1,4,7,10-tetraazacyclododecane-1,4,7-triacetic acid (**DO3A**)^[7]

To a solution of **L1** (300 mg, 0.87 mmol) in CH₂Cl₂ (3 mL) was added TFA (3 mL, 38.8 mmol) dropwise. The mixture was stirred in an ice bath progressed for ~ 24 h, the solvent was removed by rotary evaporation. Finally, the residue was dissolved in minimal H₂O and added large quantity acetone to precipitate white powder **DO3A** (150 mg, 74 %). ¹H NMR (400 MHz, D₂O), δ (ppm): 3.21 (br, 12H), 3.39 (br, 4H), 3.60 (s, 6H).

Synthesis of **DO3A**-ligated gadolinium(III) complex (**Gd-DO3A**, **4**)^[8]

To a solution of **DO3A** (220 mg, 0.63 mmol) and GdCl₃·6H₂O (142 mg, 0.38 mmol) in H₂O (3 mL) was maintained the pH value at 6-8 with NaOH. After stirring for 24 h at 70 °C, the reaction mixture was filtered and the filtrate evaporated to dryness under reduced pressure. The residue was dissolved in minimal H₂O and added large quantity acetone to precipitate white Gd complex **4** (150 mg, 50%). IR (KBr), ν (cm⁻¹): 1582 (coordinated COO⁻) (see Figure S2). MS-ESI: m/z (M + Na⁺) calcd. for GdC₁₄H₂₇N₄O₈Na, 560.1, found 560.2 (see Figure S5).

3. Copies of Spectra and TEM Images for All Compounds and Au Nanoparticles

Figure S1. The 400 MHz ¹H NMR (CDCl₃ and MeOH-*d*₄) spectra of

(a) dodecanethiolate-covered **Au-SR**, (b) DO3ASH (**1**), and (c) RS-Au-L (**2**)

(# is CHCl₃ and * is MeOH in the system).

Figure S2. IR spectra of (a) DO3A, (b) Gd-DO3A (**4**), (c) RS-Au-L (**2**), and (d) RS-Au-L-Gd (**3**).

RS-Au-L (2)

$$D = 3.09 \text{ nm} \pm 0.52$$

Figure S3. The TEM image of RS-Au-L (2).

RS-Au-L-Gd (3)

$$D = 2.99 \text{ nm} \pm 0.45$$

Figure S4. The TEM image and EDX spectrum of RS-Au-L-Gd NPs (3).

Figure S5. ESI-Mass spectrum of Gd-DO3A (4).

Figure S6. XPS spectra of (a) GdCl₃, (b) Gd-DO3A (**4**), and (c) RS-Au-L-Gd NPs (**3**).

Figure S7. The EPR spectrum of RS-Au-L-Gd NPs (**3**).

4. Spectral data of acetate products

The acetate products obtained from the **3**- or **4**-catalyzed acetylation of alcohols were analyzed by ^1H - and ^{13}C -NMR. The analytical yields obtained from both ^1H NMR and GC are comparable. For a specific reaction of phenol (0.55 mmol) with acetic anhydride (5 mmol) in the presence of catalyst **3** (0.4 mol%) was carried out under 300 W MW for 60 sec. The resulting mixture was subjected for both NMR and GC analyses. Integration of the ^1H NMR signals of phenyl acetate (iodoanisole as internal standard) gave 97.9% NMR yields (0.538 mmol), while GC analysis on the same sample gave 99% GC yields (0.544 mmol). The experimental details of MW-assisted

RS-Au-L-Gd (**3**)-catalyzed acetylation of (+)-menthol (Table 2, entry 12) and the isolation and purification procedures of the final product, (1S)-(+)-menthyl acetate were given at the end of the Supplemental Material section.

The ^1H and ^{13}C NMR spectra of the following acetate products were also attached at the end of the Supplemental Material section.

Heptyl acetate^[9a]

^1H NMR (400 MHz, CDCl_3), δ (ppm): 0.89 (t, $J = 7.4$ Hz, 3H), 1.24-1.29 (m, 8H), 1.67 (q, $J = 6.4$ Hz, 2H), 2.04 (s, 3H), 4.05 (t, $J = 6.4$ Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 14.08, 21.00, 22.59, 25.89, 28.60, 28.92, 31.71, 64.55, 170.89.

Dodecyl acetate^[9b]

^1H NMR (400 MHz, CDCl_3), δ (ppm): 0.88 (t, $J = 7.2$ Hz, 3H), 1.22-1.37 (m, 16H), 1.59-1.65 (m, 2H), 2.05 (s, 3H), 4.05 (t, $J = 6.9$ Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 14.3, 21.2, 22.8, 26.1, 28.8, 29.4, 29.5, 29.7,

29.7, 29.8, 29.8, 32.1, 64.8, 171.4.

2-Ethylhexyl acetate^[9c]

¹H NMR (400 MHz, CDCl₃), δ (ppm): 0.86 (t, $J = 7.5$ Hz, 3H), 0.89 (t, $J = 7.5$ Hz, 3H), 1.28-1.35 (m, 8H), 1.55 (septet, $J = 6$ Hz, 1H), 2.04 (s, 3H), 3.98 (dd, $J = 6$, 1.5 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃), δ (ppm): 10.9, 14.0, 21.0, 22.9, 23.7, 28.9, 30.4, 38.7, 66.9, 171.4.

Phenyl acetate^[9d]

¹H NMR (400 MHz, CDCl₃), δ (ppm): 2.23 (s, 3H), 7.06 (d, $J = 8.56$ Hz, 2H), 7.18 (t, $J = 7.44$ Hz, 1H), 7.34 (t, $J = 7.40$ Hz, 2H).

¹³C NMR (100 MHz, CDCl₃), δ (ppm): 21.1, 121.6, 125.8, 129.5, 150.8, 169.5.

Benzyl acetate^[9d]

^1H NMR (400 MHz, CDCl_3), δ (ppm): 2.06 (s, 3H), 5.08 (s, 2H), 7.29-7.35 (m, 5H).

^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 21.0, 66.3, 128.3, 128.6, 136.0, 170.8.

4-Methoxybenzyl acetate^[9e]

^1H NMR (400 MHz, CDCl_3), δ (ppm): 2.08 (s, 3H), 3.81 (s, 3H), 5.04 (s, 2H), 6.88 (d,

$J = 8.80$ Hz, 2H), 7.26 (d, $J = 8.4$ Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 21.1, 55.3, 66.1, 113.9, 128.0, 130.1, 159.5,

170.2.

4-Phenyl benzyl acetate^[9f]

^1H NMR (400 MHz, CDCl_3), δ (ppm): 2.14 (s, 3H), 5.13 (s, 2H), 7.33-7.35 (m, 1 H),

7.41-7.44 (m, 4 H), 7.56-7.59 (m, 4H).

^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 20.7, 65.8, 126.9, 127.1, 127.3, 128.7, 128.7,

134.9, 140.4, 140.9, 170.6.

4-Methylbenzyl acetate^[9g]

¹H NMR (400 MHz, CDCl₃), δ (ppm): 2.08 (s, 3H) , 2.35 (s, 3H), 5.06 (s, 2H), 7.17 (d, J = 7.9 Hz, 2H), 7.25 (d, J = 8.0 Hz, 2H).

¹³C NMR (100MHz, CDCl₃), δ (ppm): 21.1, 21.0, 21.1, 66.2, 128.3, 129.1, 132.8, 138.0, 170.8.

4-Nitrobenzyl acetate^[9h]

¹H NMR (400 MHz, CDCl₃), δ (ppm): 2.16 (s, 3H) 5.21 (s, 2H), 7.52 (d, J = 8.0 Hz, 2H), 8.23 (d, J = 8.0 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃), δ (ppm): 20.78, 64.73, 123.73, 128.35, 143.30, 147.63, 170.50.

2-Iodobenzyl acetate^[9i]

^1H NMR (400 MHz, CDCl_3), δ (ppm): 2.14 (s, 3 H), 5.11 (s, 2 H), 6.97-7.06 (m, 1 H), 7.30-7.41 (m, 2 H), 7.84 (d, $J = 7.8$ Hz, 1 H).

^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 20.8, 69.9, 98.3, 128.2, 129.3, 129.8, 138.2, 139.4, 170.4.

Cyclohexyl acetate^[9e]

^1H NMR (400MHz, CDCl_3), δ (ppm): 1.22-1.46 (m, 5H), 1.52-1.58 (m, 1H), 1.71-1.74 (m, 2H), 1.86-1.88 (m, 2H), 2.06 (s, 3H), 4.71-4.77 (m, 1H).

^{13}C NMR (100MHz, CDCl_3), δ (ppm): 21.4, 23.8, 25.4, 31.7, 73.0, 171.0.

(1S)-(+)-Menthyl acetate^[9j]

A mixture of colloidal AuNP-surface-bound Gd catalyst **RS-Au-L-Gd (3)** (2.0×10^{-3} mmol Gd), (+)-menthol (0.5 mmol) and acetic anhydride (5 mmol) in DMF (80 μ L) were placed in the reaction vessel under 300 W MW (CEM single-mode Discover Labmate System) for 60 sec. After the reaction, the resulting crude mixture was added 4-iodoanisole as the internal standard (61.0 mg, 0.26 mmol). Integration of the ^1H NMR signals of the starting (+)-menthol, the designated product, (1S)-(+)-menthyl acetate and 4-iodoanisole gave 0.26 mmol of product (yield = 52%). Further purification of the acetate product was done by column chromatography on silica gel (EA/hexane, 1/70) to give (1S)-(+)-menthyl acetate (47.6 mg, 48% isolated yield). ^1H NMR (400 MHz, CDCl_3), δ (ppm): 0.70 (d, $J = 7.0$, 3H), 0.74-0.80 (m, 1H), 0.84 (d, $J = 7.4$, 6H), 0.90-1.11 (m, 2H), 1.13-1.52 (m, 2H), 1.54-1.70 (m, 2H), 1.71-1.86 (m, 1H), 1.86-1.93 (m, 1H), 1.97 (s, 3H), 4.62 (dt, $J = 11.0, 4.4$, 1H). ^{13}C NMR (100 MHz, CDCl_3), δ (ppm): 16.28, 20.64, 21.23, 21.91, 23.40, 26.22, 31.29, 34.17, 46.93, 74.13, 170.80.

Figure S8. The ^1H NMR spectrum of the reaction crude product mixtures containing 4-iodoanisole (as the internal standard), remaining (+)-menthol and (1S)-(+)-menthyl acetate product. (* is CHCl_3 and # is DMF.)

Figure S9. ^1H NMR spectrum of (1S)-(+)-menthyl acetate. (* is CHCl_3).

Figure S10. ^{13}C NMR spectrum of (1S)-(+)-menthyl acetate.

Figure S11. ^1H NMR spectrum of heptyl acetate (* is CHCl_3).

Figure S12. ^{13}C NMR spectrum of heptyl acetate (* is acetic anhydride).

Figure S13. ^1H NMR spectrum of dodecyl acetate (* is CHCl_3).

Figure S14. ^{13}C NMR spectrum of dodecyl acetate.

Figure S15. ^1H NMR spectrum of 2-ethylhexyl acetate (* is CHCl_3 , # is acetic anhydride).

Figure S16. ^{13}C NMR spectrum of 2-ethylhexyl acetate.

Figure S17. ¹H NMR spectrum of phenyl acetate (* is CHCl₃).

Figure S18. ¹³C NMR spectrum of phenyl acetate (* is acetic anhydride).

Figure S19. ^1H NMR spectrum of benzyl acetate (* is CHCl_3 , # is acetic anhydride).

Figure S20. ^{13}C NMR spectrum of benzyl acetate.

Figure S21. ¹H NMR spectrum of 4-methoxybenzyl acetate (* is CHCl₃).

Chemical structure: O=C1NCCC1Cc2ccc(cc2)-c3ccccc3

¹H NMR spectrum (CDCl₃) showing peaks at 7.6090, 7.6022, 7.5987, 7.5929, 7.5908, 7.5818, 7.4668, 7.4492, 7.4297, 7.3772, 7.3593, 7.2598, 5.1564 (H_b), 2.2265, 2.1279 (H_a), and 1.5888 ppm.

Integration values: 4.1229, 4.0665, 0.9777 (aromatic), 2.0000 (H_b), 2.9163 (H_a).

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Figure S24. ^{13}C NMR spectrum of 4-phenyl benzyl acetate.

Figure S25. ^1H NMR spectrum of 4-methylbenzyl acetate.

Figure S26. ^{13}C NMR spectrum of 4-methylbenzyl acetate.

Figure S27. ^1H NMR spectrum of 4-nitrobenzyl acetate (* is CHCl_3 , # is acetic anhydride).

Figure S28. ^{13}C NMR spectrum of 4-nitrobenzyl acetate.

Figure S29. ^1H NMR spectrum of 2-iodobenzyl acetate (* is CHCl_3).

Figure S30. ^{13}C NMR spectrum of 2-iodobenzyl acetate.

Figure S31. ^1H NMR spectrum of cyclohexyl acetate (* is CHCl_3 , # is acetic

anhydride).

Figure S32. ^{13}C NMR spectrum of cyclohexyl acetate (* is acetic anhydride).

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