Hybrid Multivalent Jack Bean α-Mannosidase Inhibitors: The First Example of Gold Nanoparticles Decorated with Deoxynojirimycin Inhitopes

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Supplementary Materials

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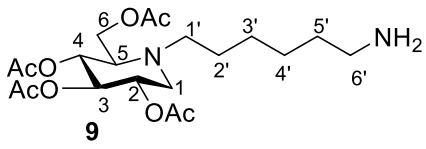

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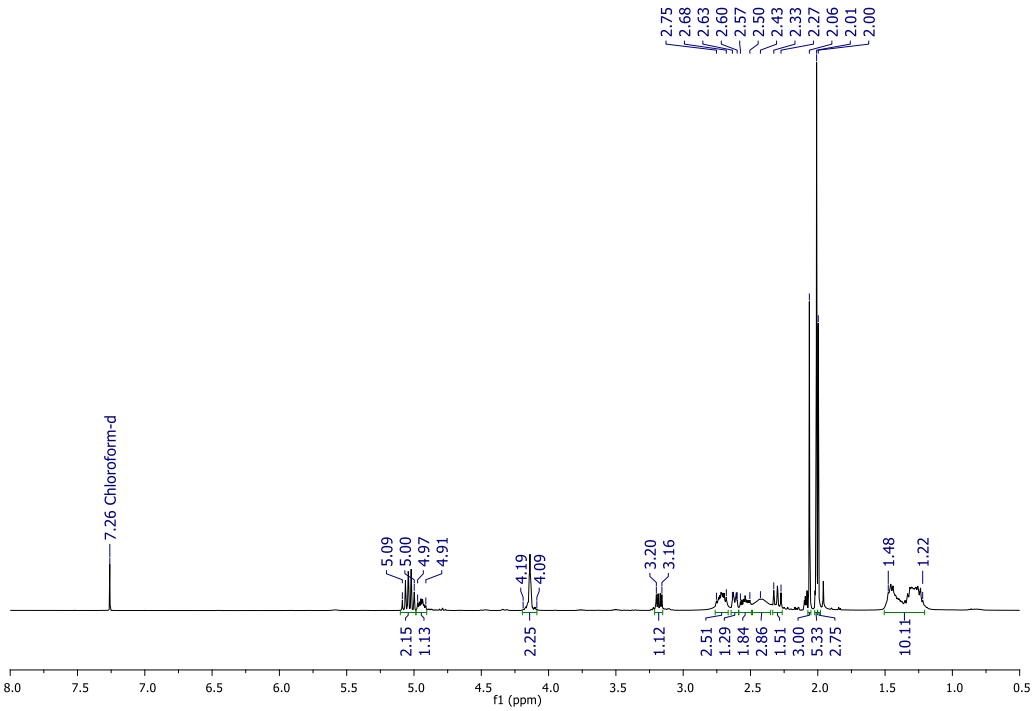


Figure S1. ^{1}H-NMR spectrum of compound **9** (400 MHz, $CDCl_3$).

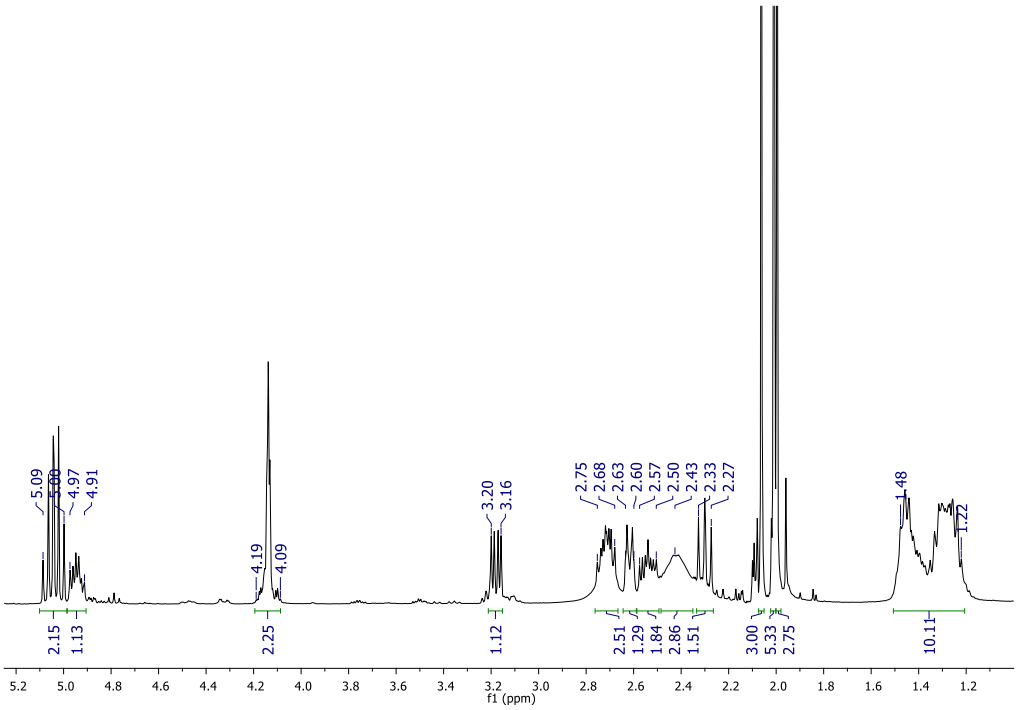


Figure S2. Expansion of ^{1}H-NMR spectrum of compound **9** (400 MHz, $CDCl_3$).

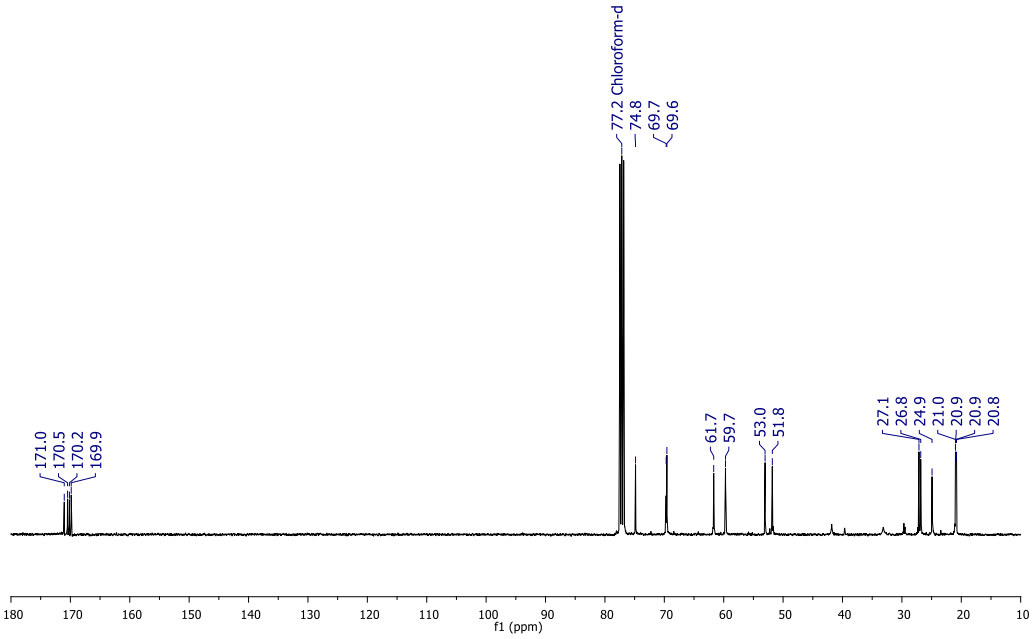
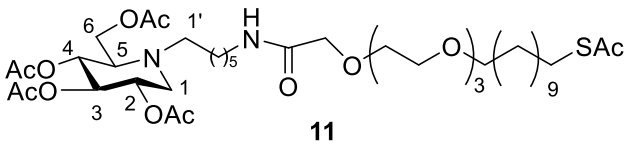


Figure S3. ^{13}C-NMR spectrum of compound **9** (100 MHz, $CDCl_3$).



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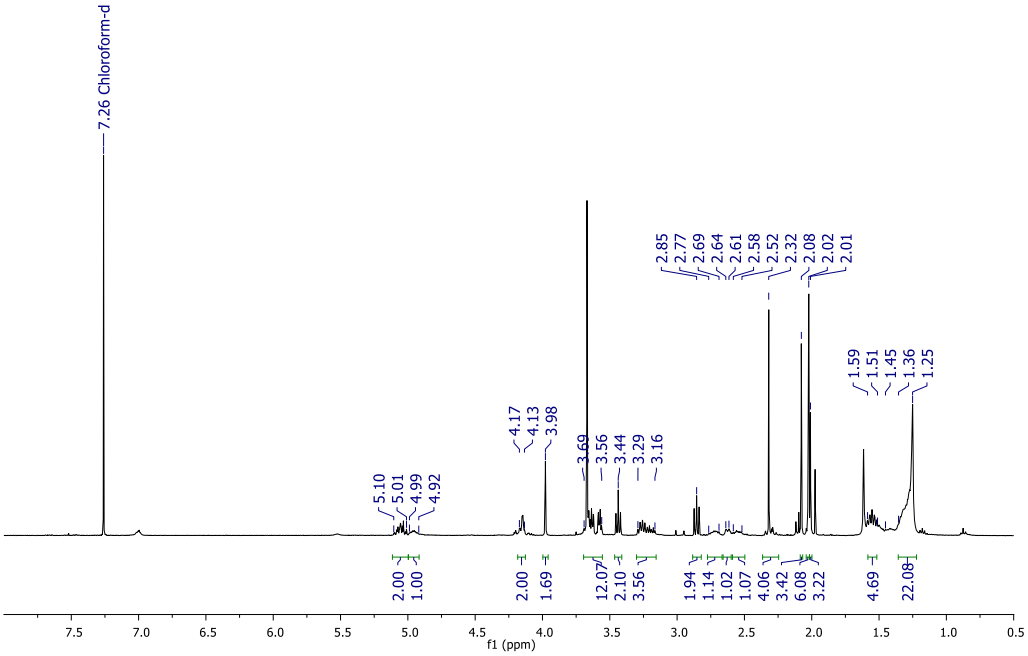


Figure S4. ^{1}H-NMR spectrum of compound **11** (400 MHz, $CDCl_3$).

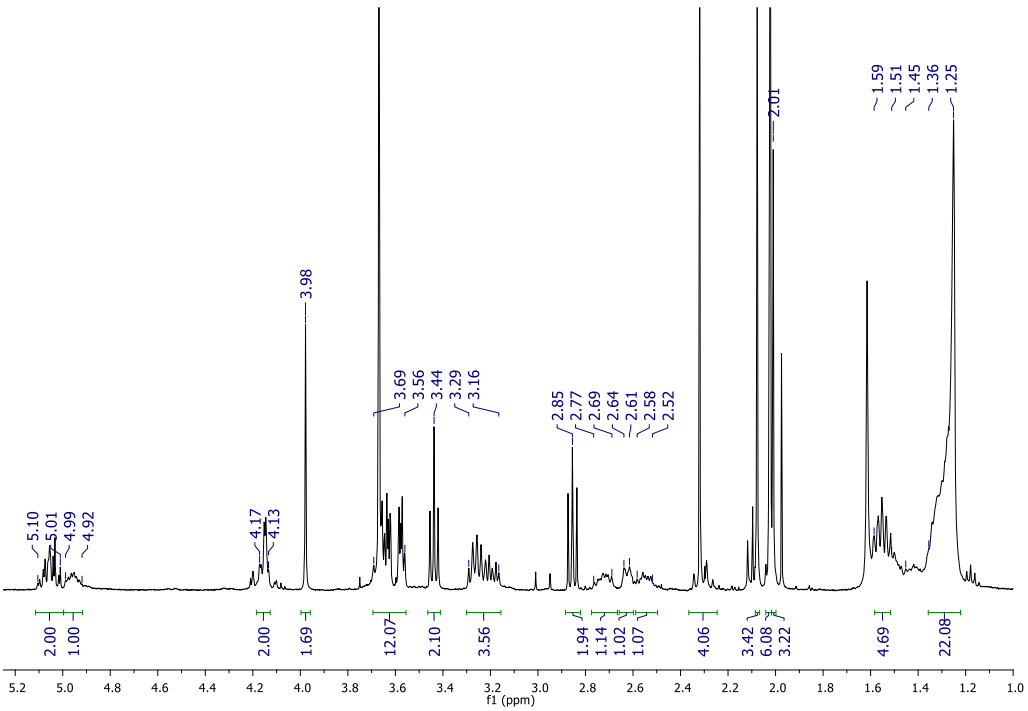


Figure S5. Expansion of ^{1}H-NMR spectrum of compound **11** (400 MHz, $CDCl_3$).

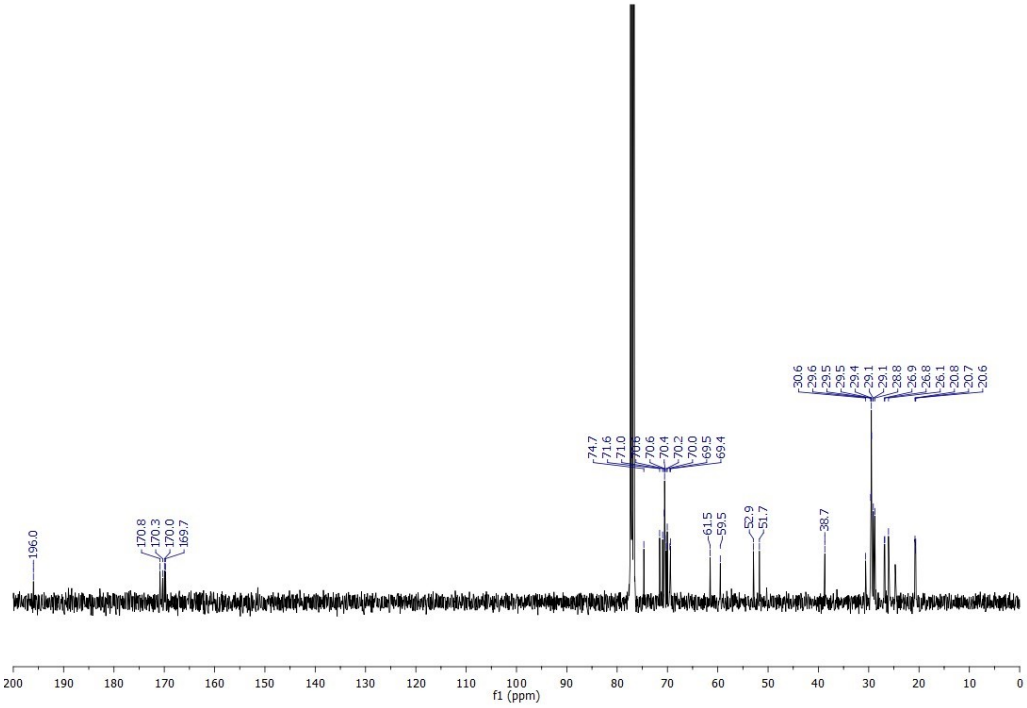
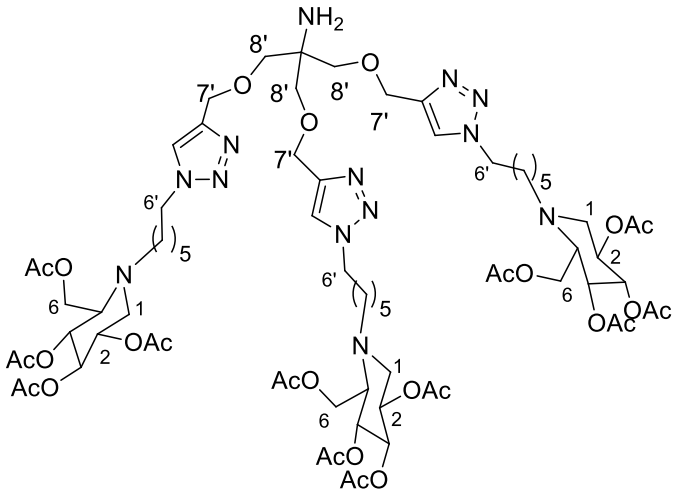


Figure S6. ^{13}C-NMR spectrum of compound **11** (100 MHz, $CDCl_3$).



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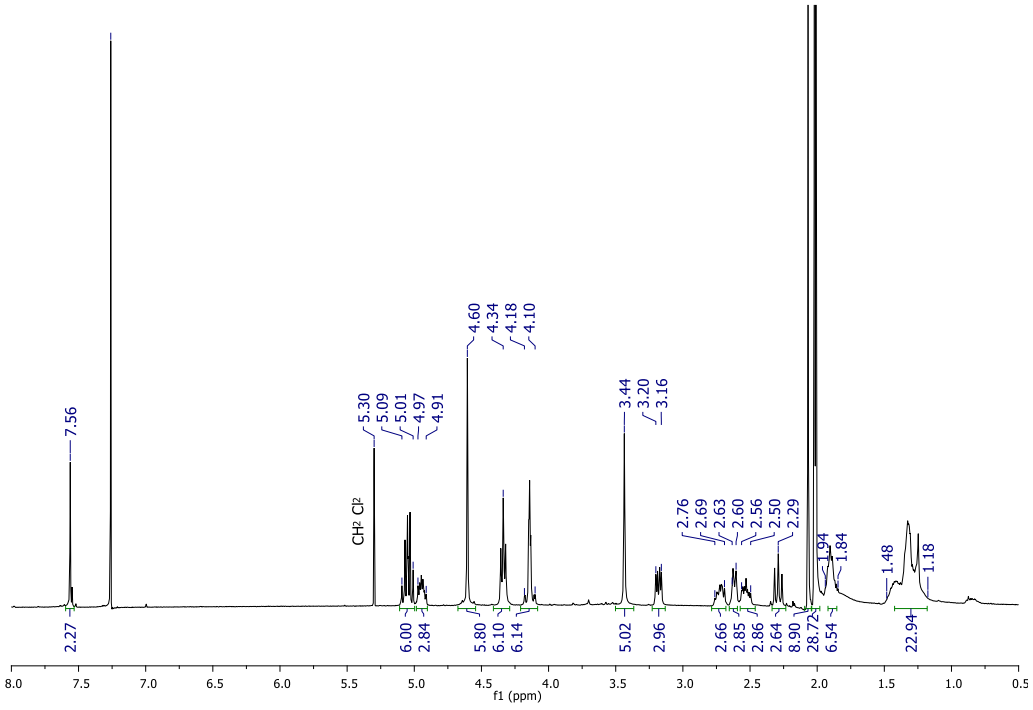


Figure S7. ^{1}H-NMR spectrum of compound **16** (400 MHz, $CDCl_3$).

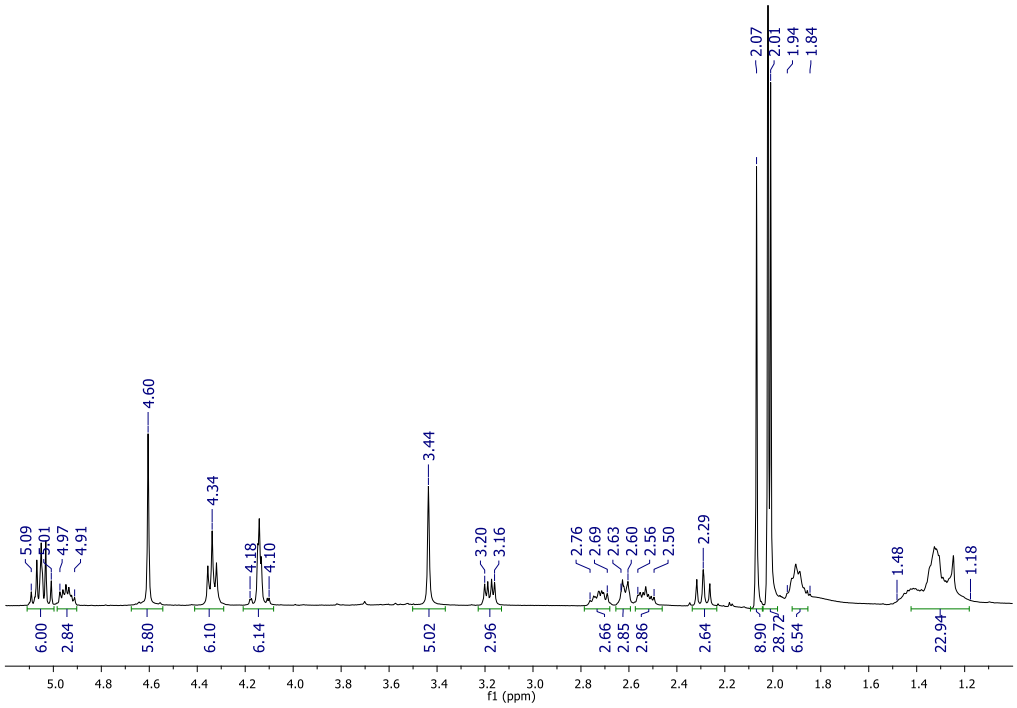


Figure S8. Expansion of ^{1}H-NMR spectrum of compound **16** (400 MHz, $CDCl_3$).

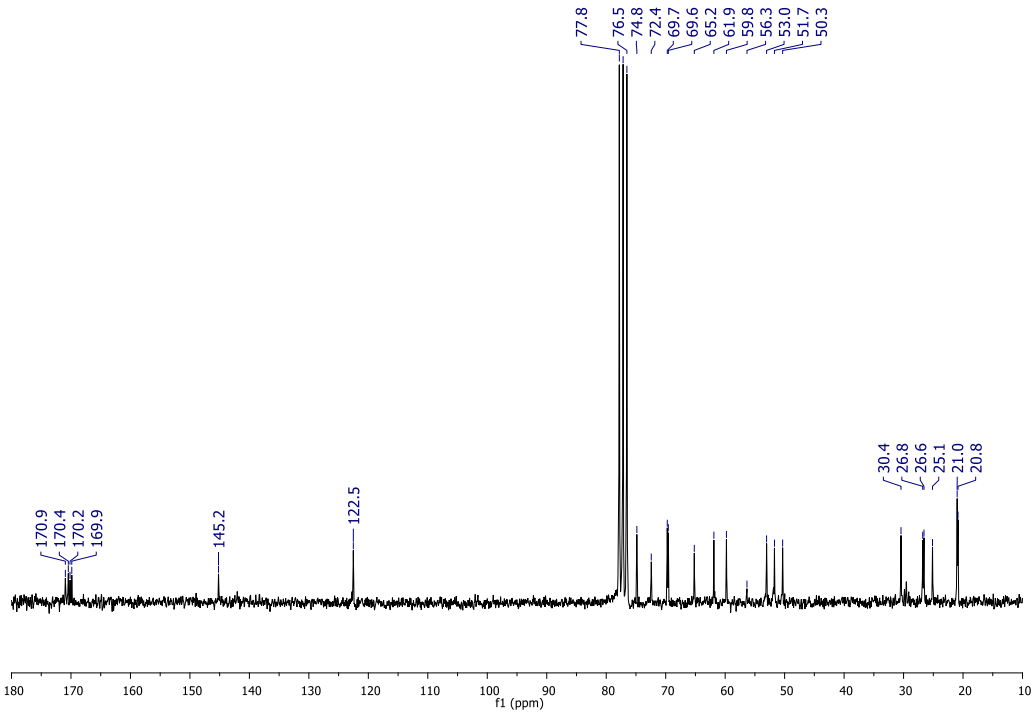
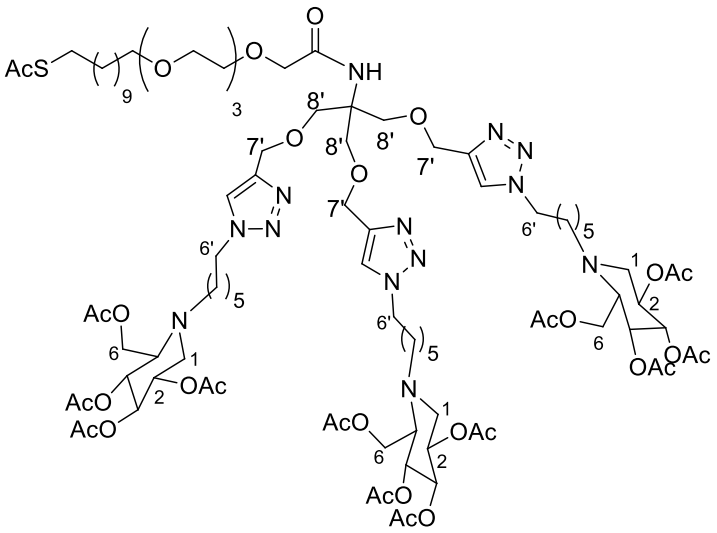


Figure S9. ^{13}C-NMR spectrum of compound **16** (100 MHz, $CDCl_3$).



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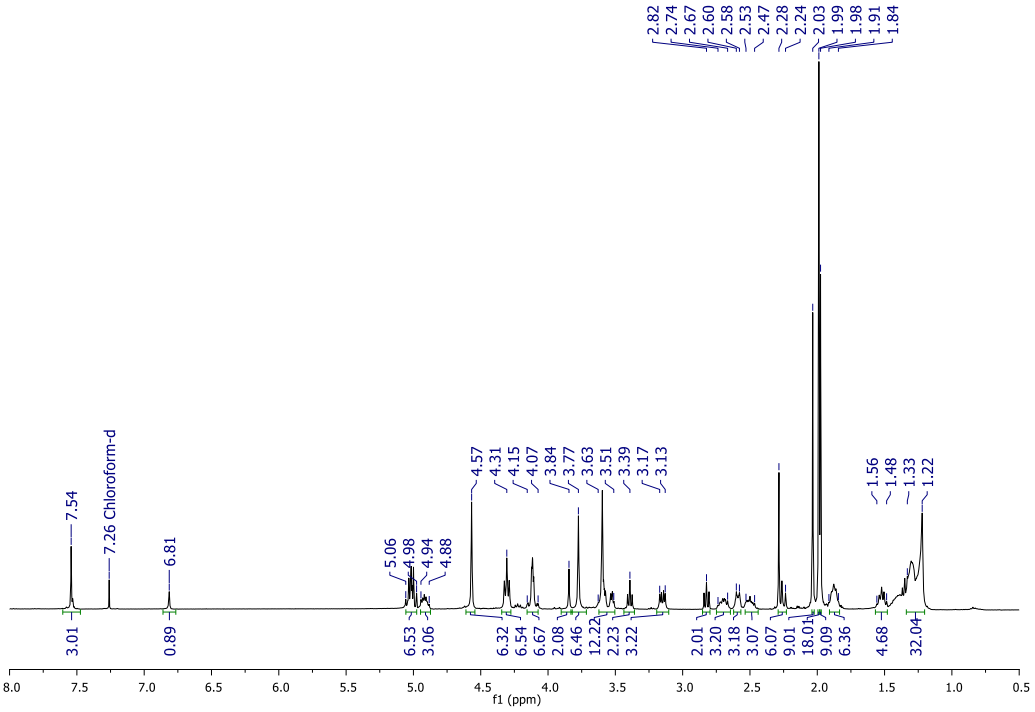


Figure S10. ^{1}H-NMR spectrum of compound **17** (400 MHz, $CDCl_3$).

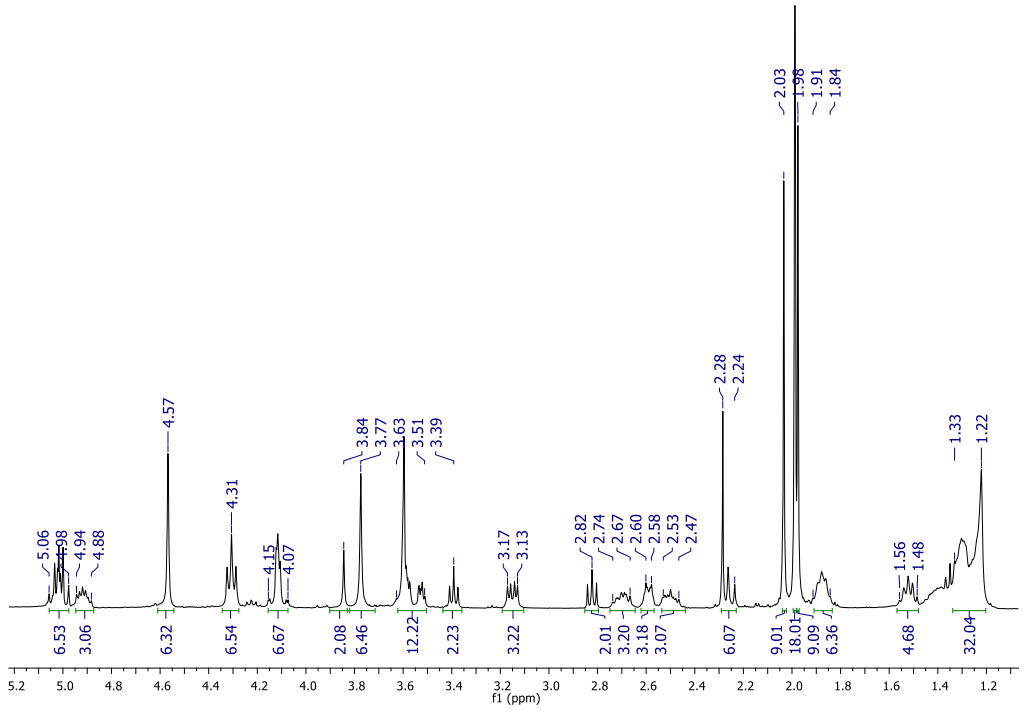


Figure S11. Expansion of ^{1}H-NMR spectrum of compound **17** (400 MHz, $CDCl_3$).

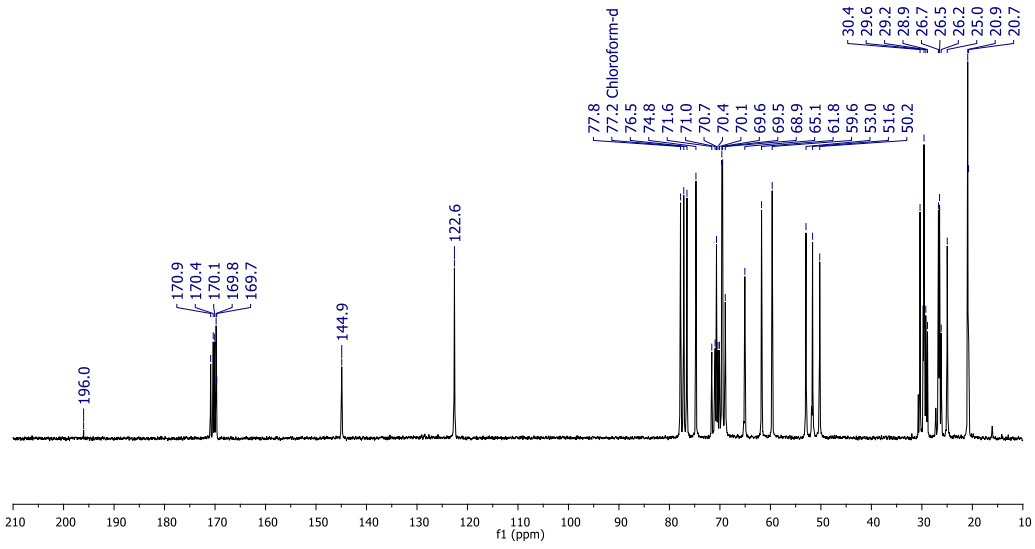


Figure S12. ^{13}C-NMR spectrum of compound **17** (100 MHz, $CDCl_3$).

*Characterization of AuGNP **1***

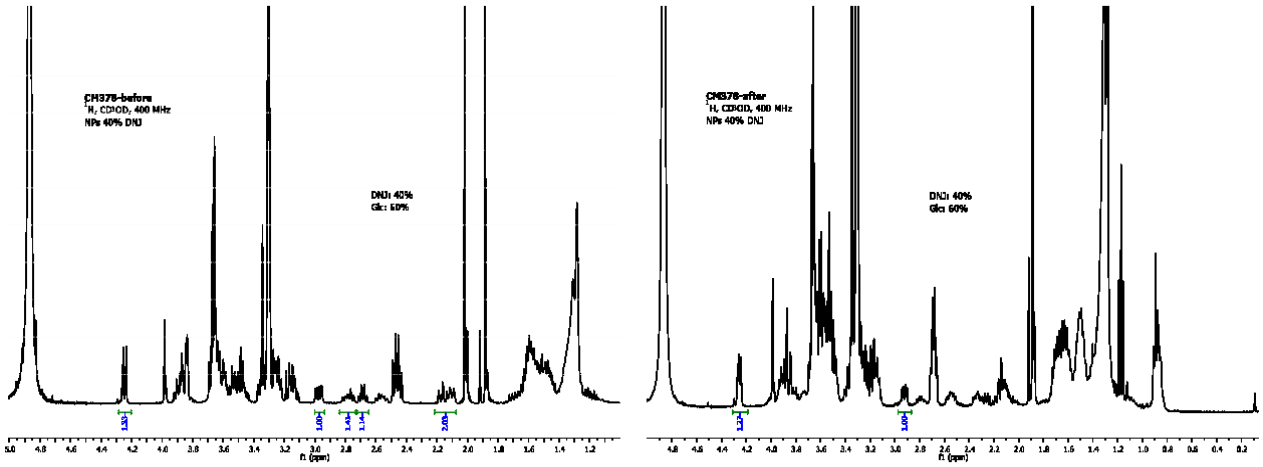


Figure S13. ^{1}H NMR of sugar/iminosugar ligands mixture before (left) and after (right) formation of AuGNP **1** (400 MHz, CD_3OD).

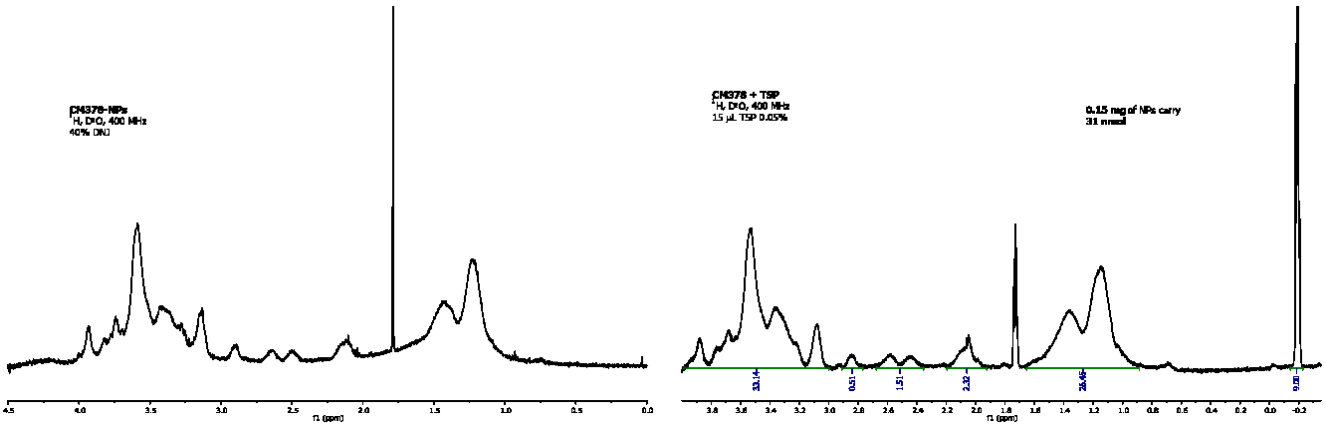


Figure S14. ^{1}H NMR and ^{1}H qNMR with TSP-d_4 of AuGNP **1** (400 MHz, D_2O).

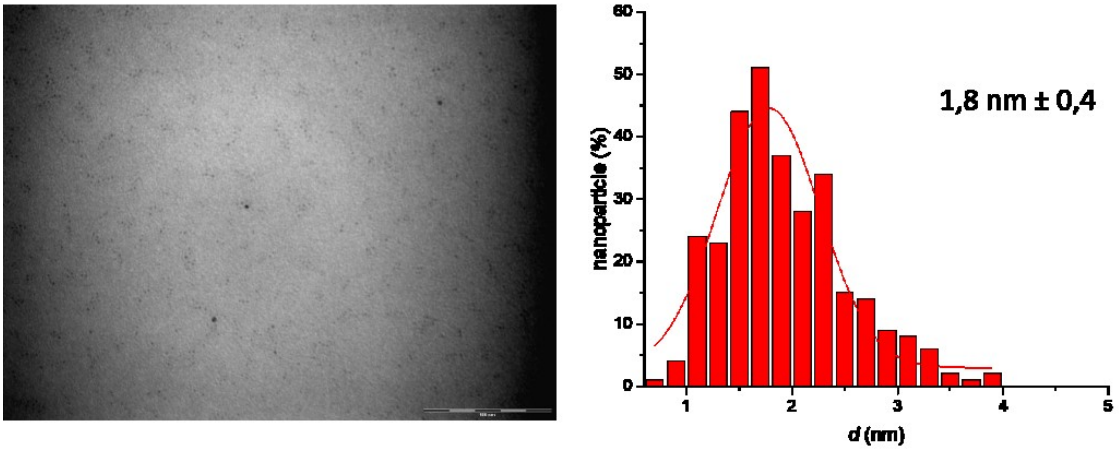


Figure S15. TEM micrograph (scale = 100 nm) in H_2O and size-distribution histogram obtained by measuring 300 nanoparticles of AuGNP **1** (average diameter: 1.8 ±0.4 nm).

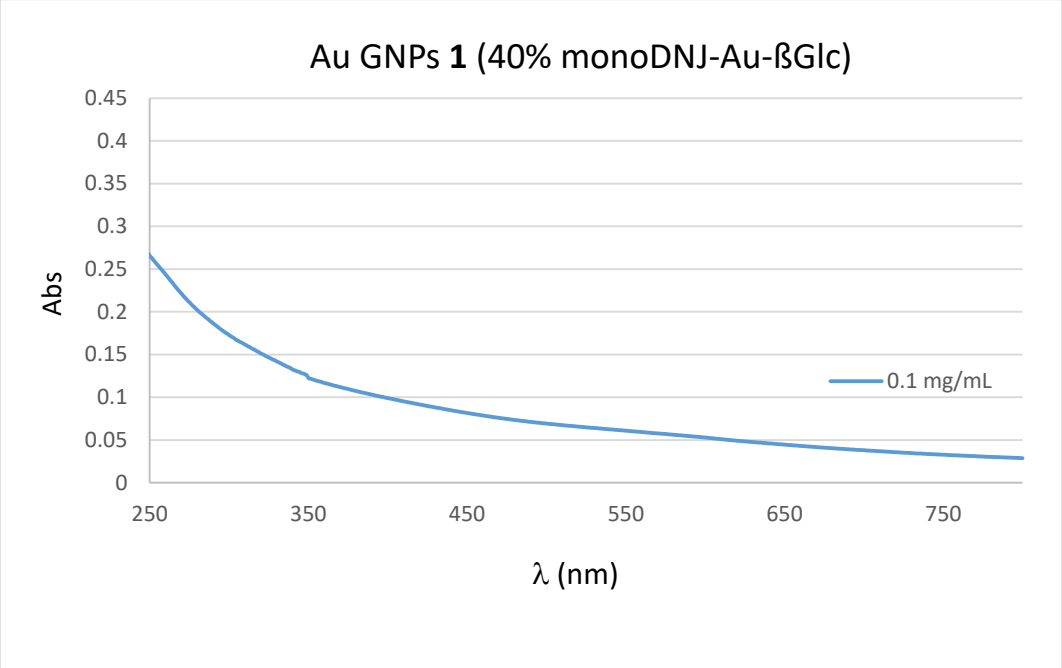


Figure S16. UV/vis spectrum of H_2O solution of AuGNP **1** recorded at concentration of 0.1 mg/mL.

*Characterization of AuGNP **2***

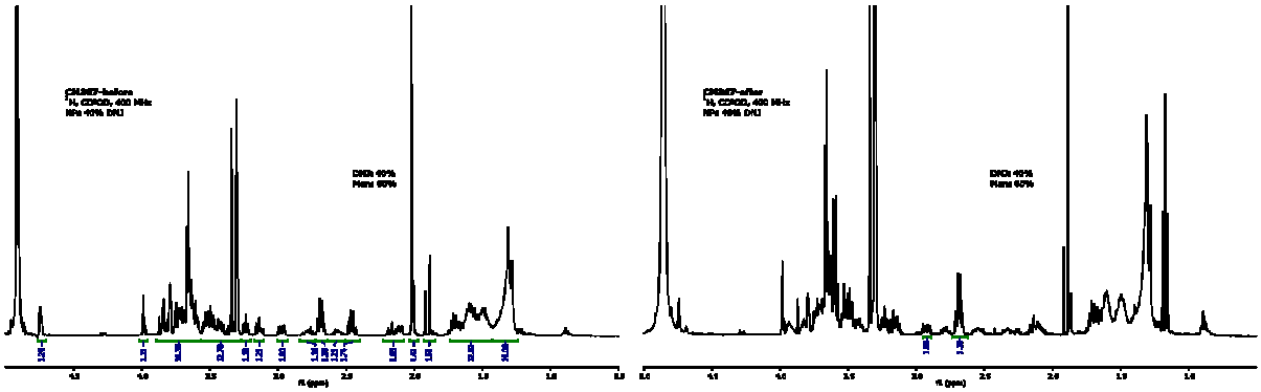


Figure S17. ^{1}H NMR of sugar/iminosugar ligands mixture before (left) and after (right) formation of AuGNP **2** (400 MHz, CD_3OD).

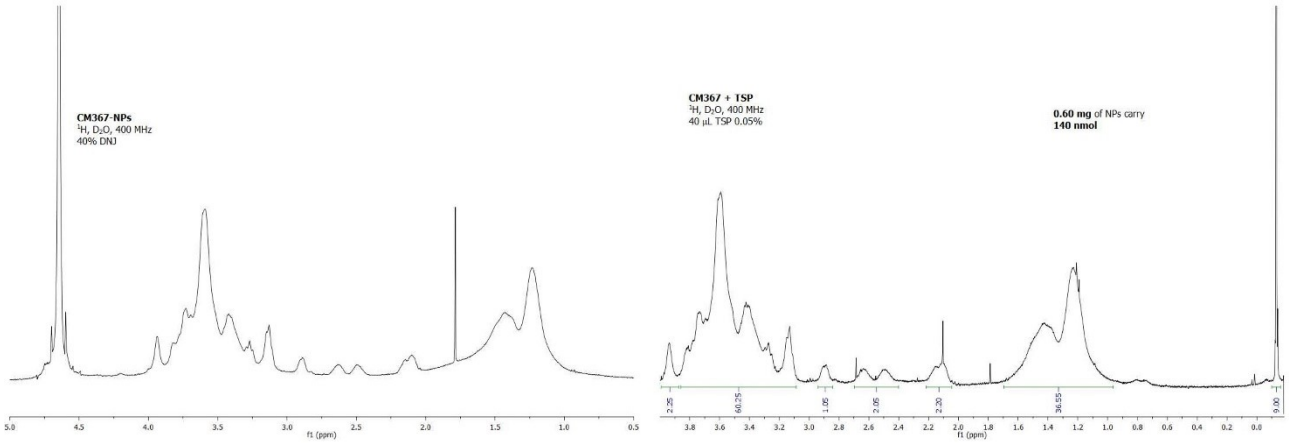


Figure S18. ^{1}H NMR and ^{1}H qNMR with TSP-d_4 of AuGNP **2** (400 MHz, D_2O).

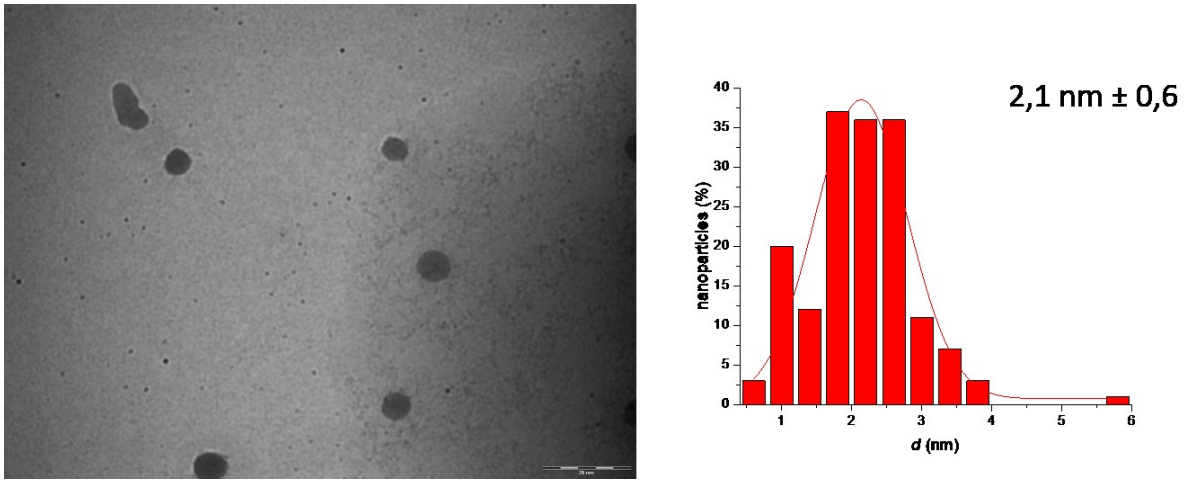


Figure S19. TEM micrograph (scale = 20 nm) in H_2O and size-distribution histogram obtained by measuring 300 nanoparticles of AuGNP **2** (average diameter: 2.1 ±0.6 nm, less than 3% shows a >5 nm diameter).

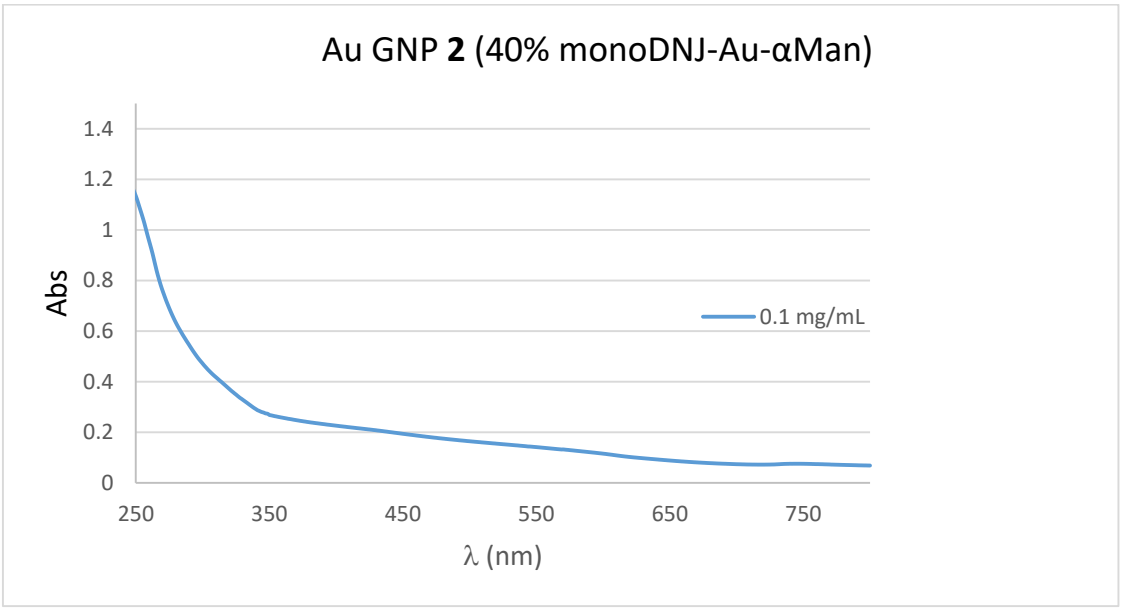


Figure S20. UV/vis spectrum of H_2O solution of AuGNP **2** recorded at concentration of 0.1 mg/mL.

*Characterization of AuGNP **3***

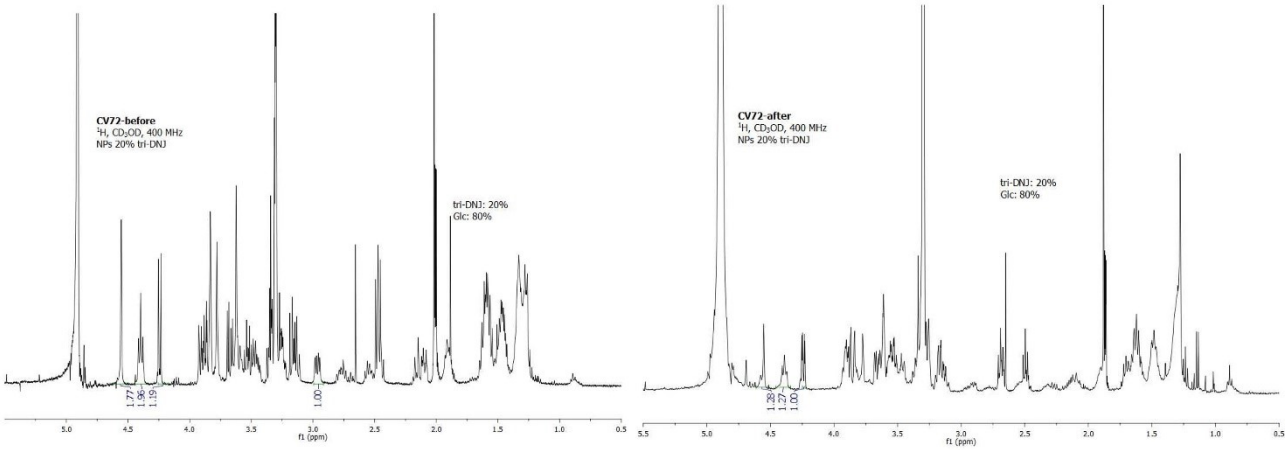


Figure S21. ^{1}H NMR of sugar/iminosugar ligands mixture before (left) and after (right) formation of AuGNP **3** (400 MHz, CD_3OD).

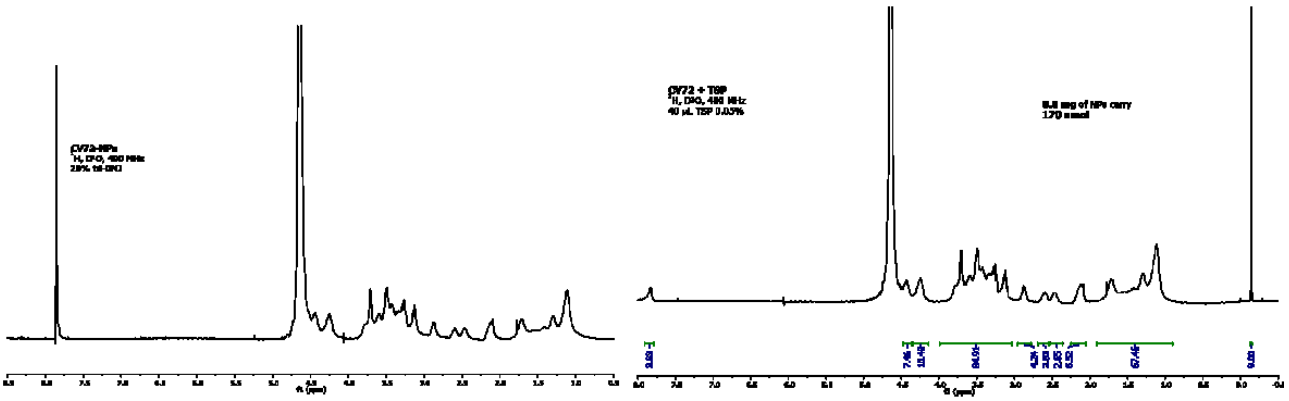


Figure S22. ^{1}H NMR and ^{1}H qNMR with TSP-d_4 of AuGNP **3** (400 MHz, D_2O).

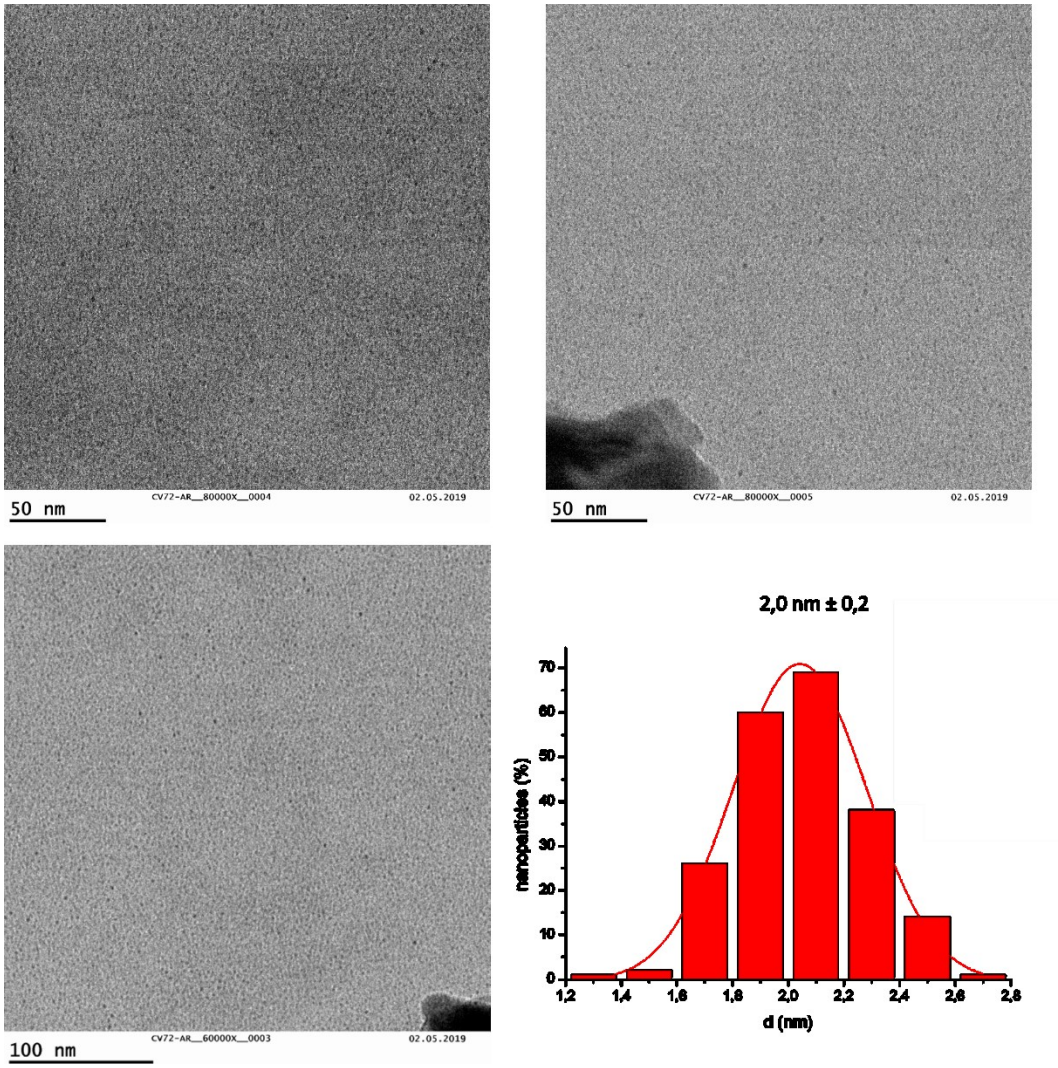


Figure S23. TEM micrographs in H_2O (magnification 80000x and 60000x) and size-distribution histogram obtained by measuring 300 nanoparticles of AuGNP **3** (average diameter: 2.1 ±0.5 nm).

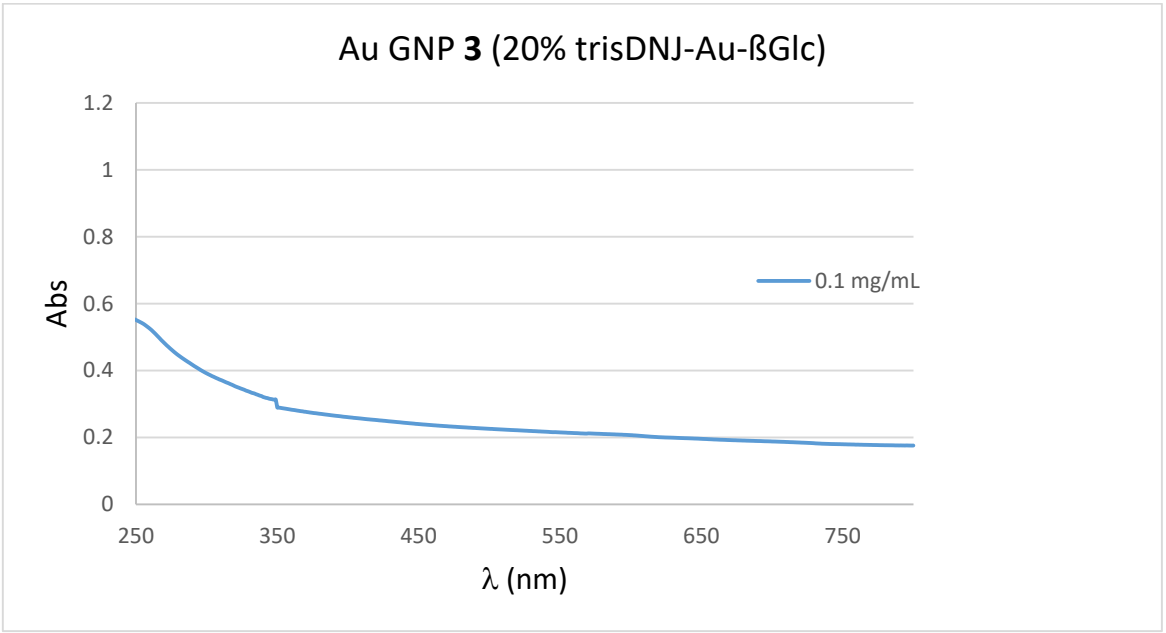


Figure S24. UV/vis spectrum of H_2O solution of AuGNP **3** recorded at concentration of 0.1 mg/

*Characterization of AuGNP **4***

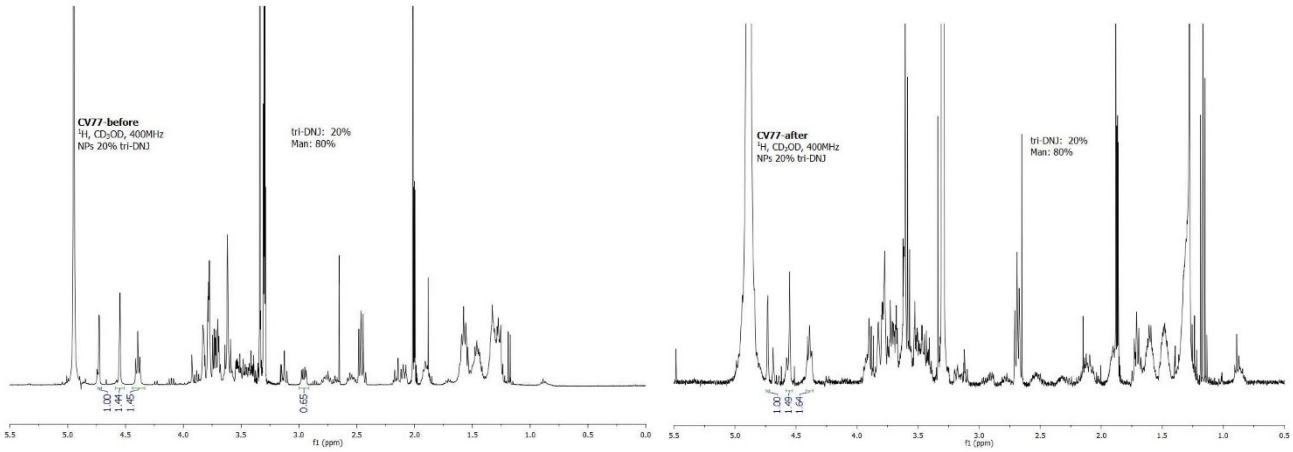


Figure S25. ^{1}H NMR of sugar/iminosugar ligands mixture before (left) and after (right) formation of AuGNP **4** (400 MHz, CD_3OD).

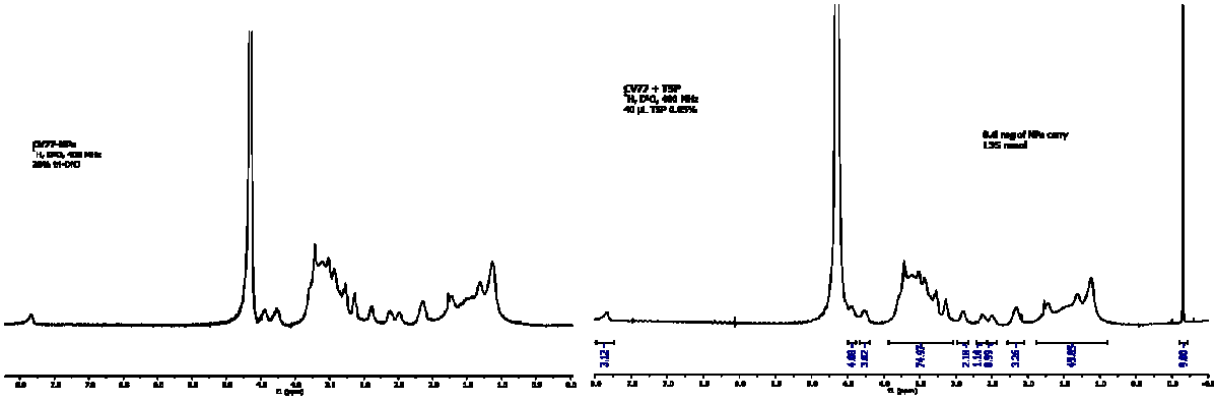


Figure S26. ^{1}H NMR and ^{1}H qNMR with TSP-d_4 of AuGNP **4** (400 MHz, D_2O).

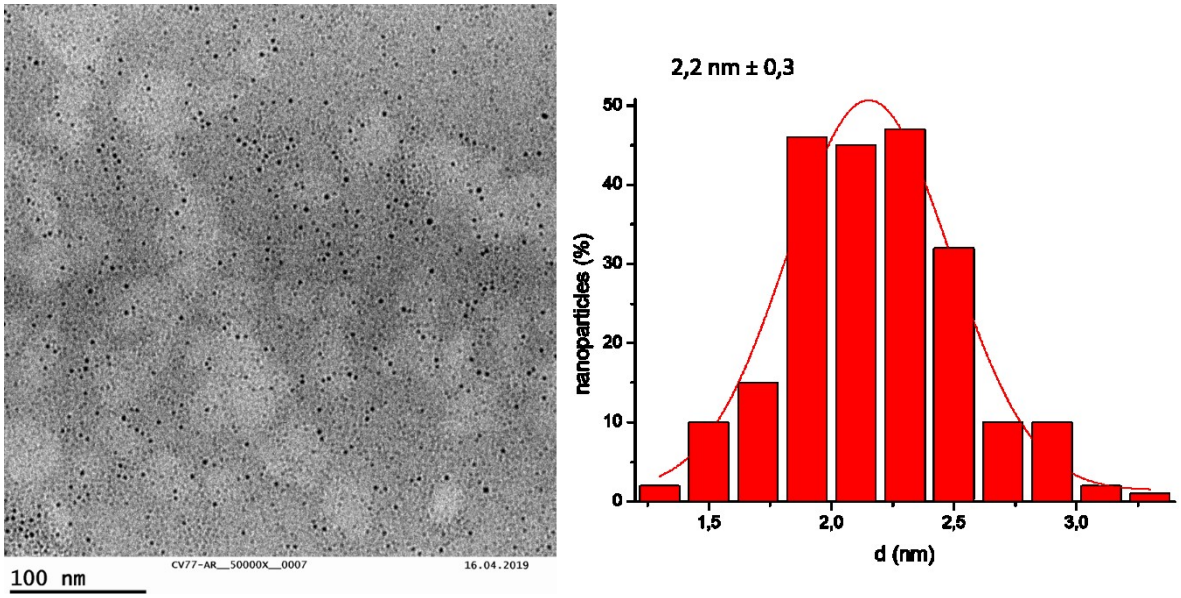


Figure S27. TEM micrograph in H_2O and size-distribution histogram obtained by measuring 300 nanoparticles of AuGNP **4** (average diameter: 2.0 ±0.4 nm).

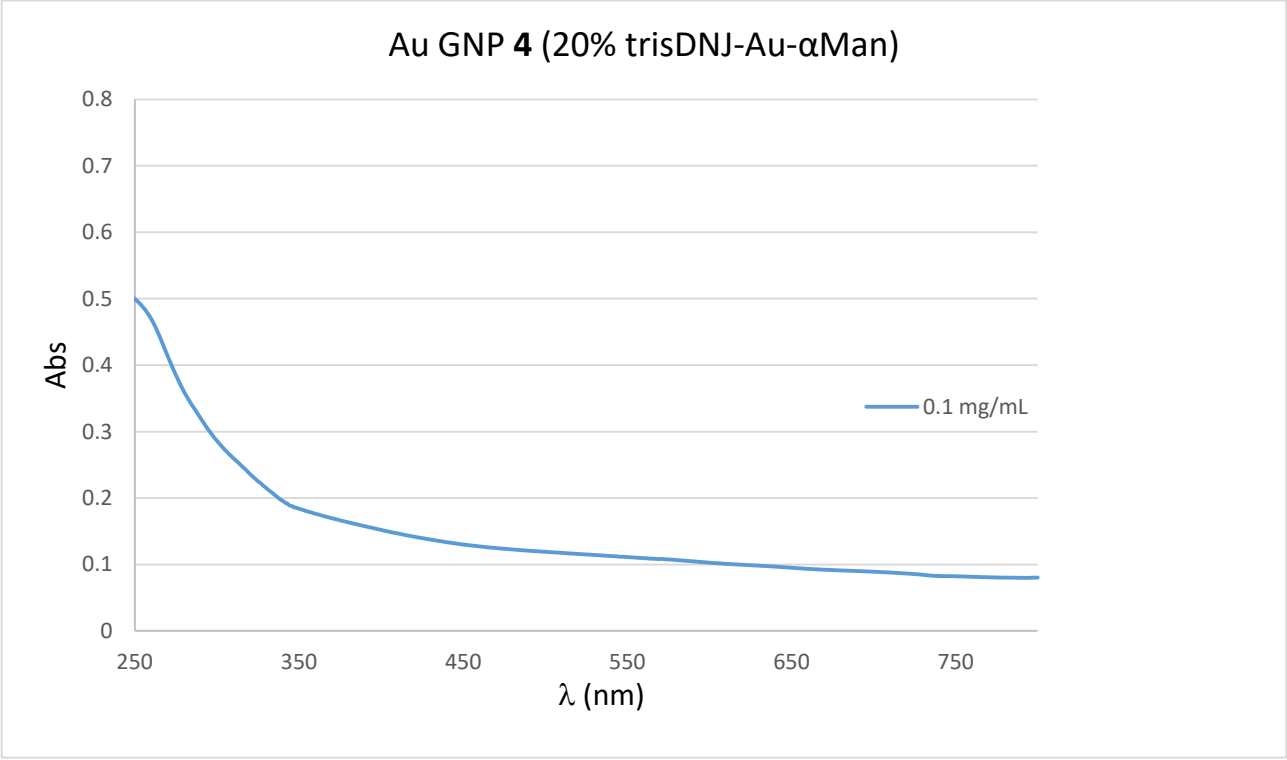


Figure S28. UV/vis spectrum of H_2O solution of AuGNP **4** recorded at concentration of 0.1 mg/mL.

*Characterization of AuGNP **5***

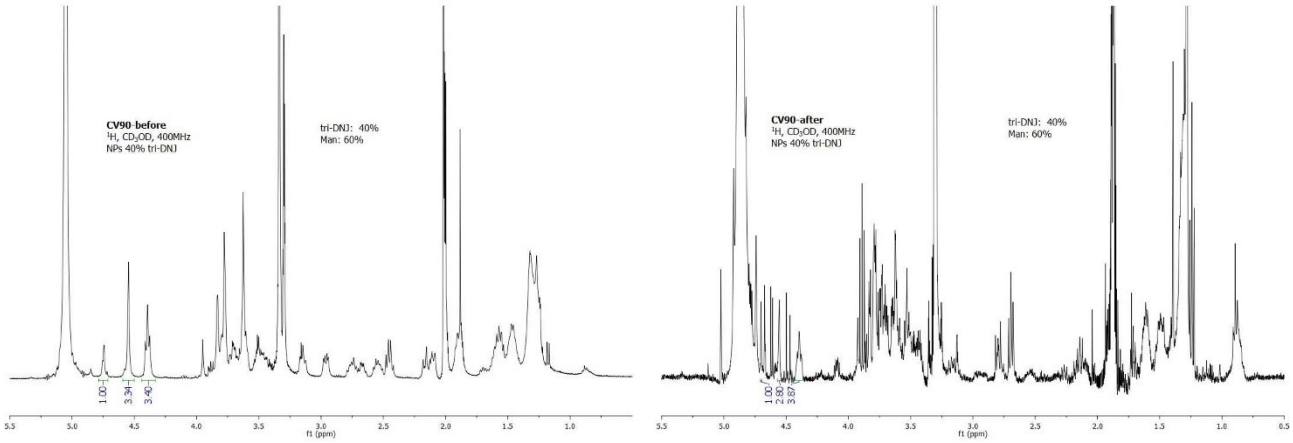


Figure S29. ^{1}H NMR of sugar/iminosugar ligands mixture before (left) and after (right) formation of AuGNP **5** (400 MHz, CD_3OD).

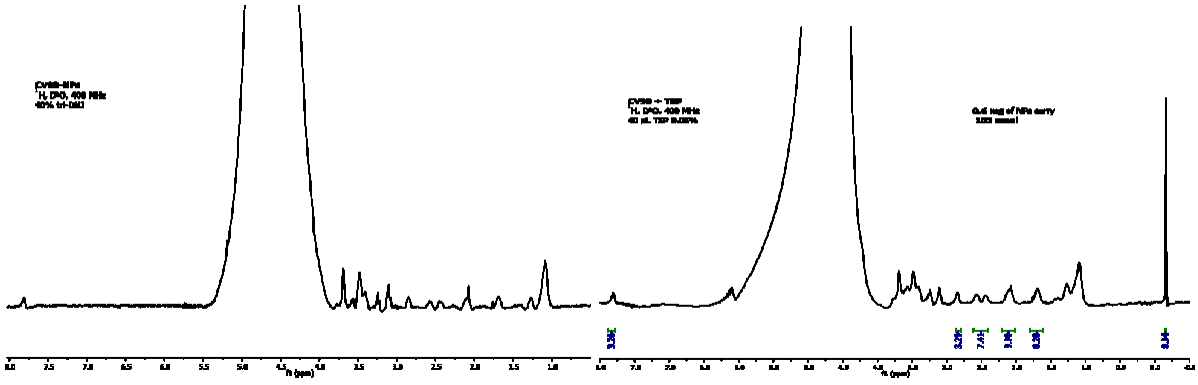


Figure S30. ^{1}H NMR and ^{1}H qNMR with TSP-d_4 of AuGNP **5** (400 MHz, D_2O).

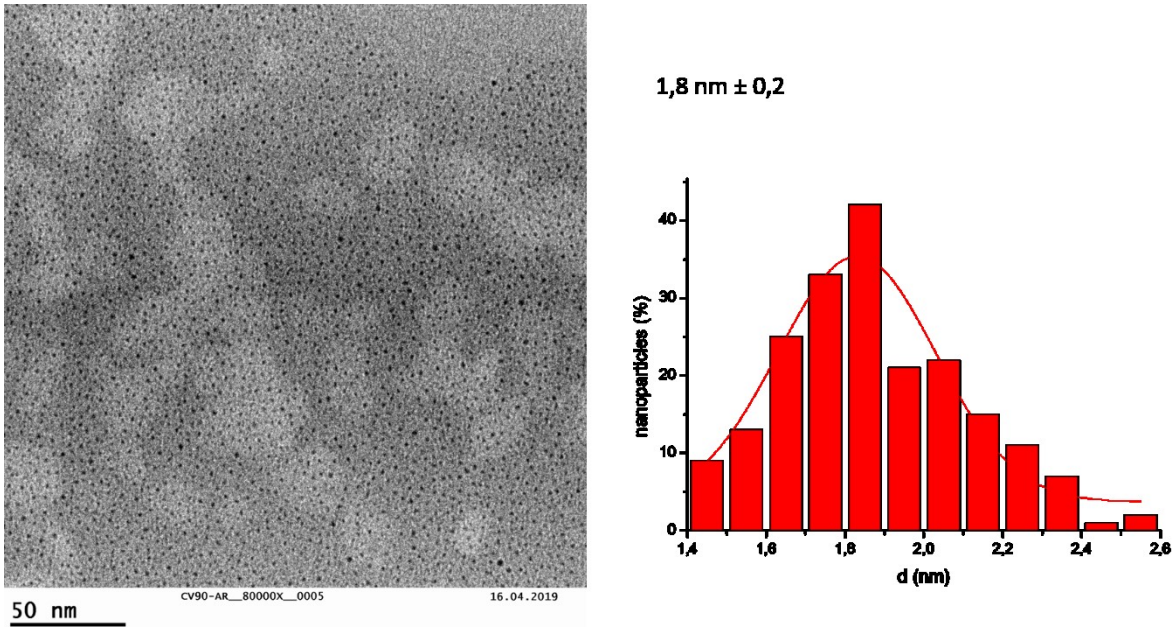


Figure S31. TEM micrograph in H_2O and size-distribution histogram obtained by measuring 300 nanoparticles of AuGNP **5** (average diameter: 2.1 ±0.5 nm).

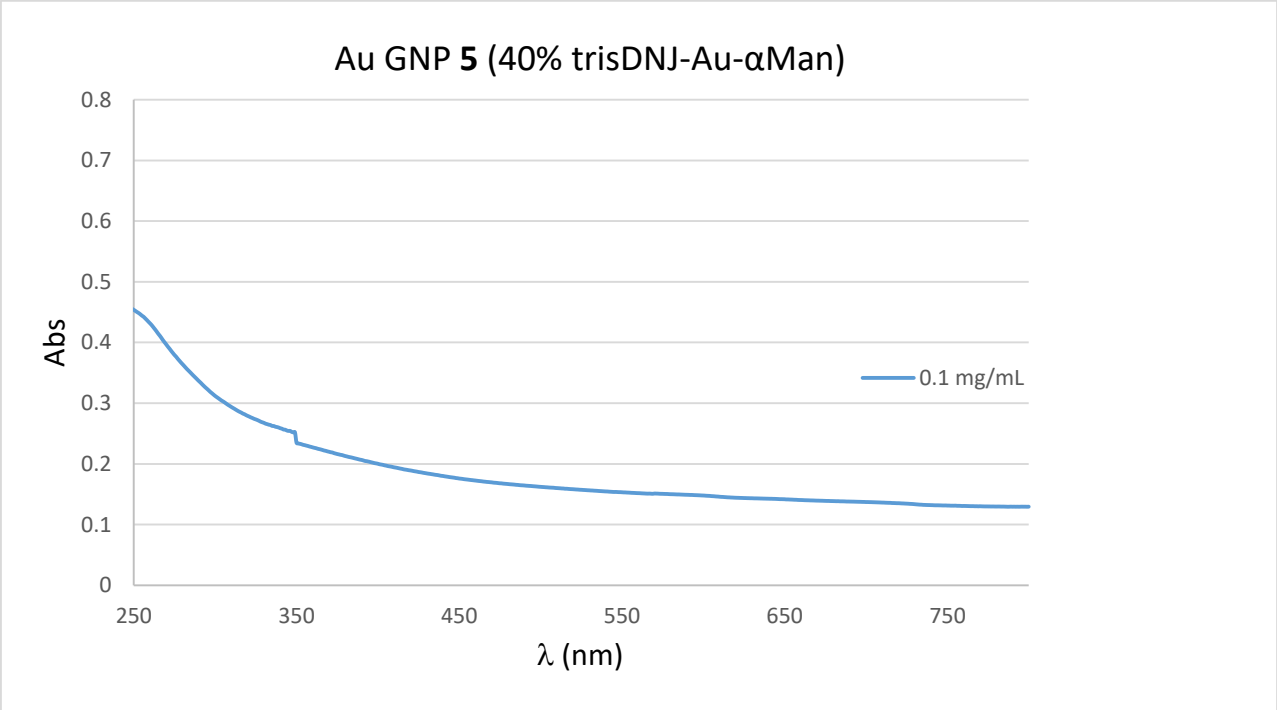
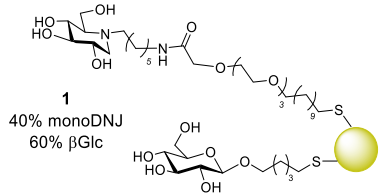
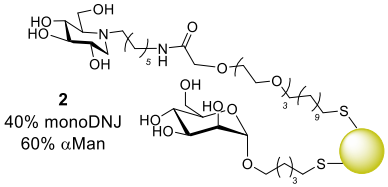
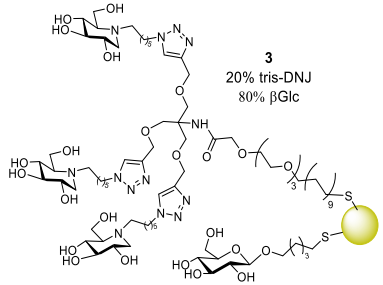
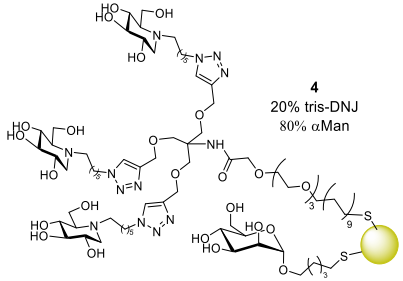
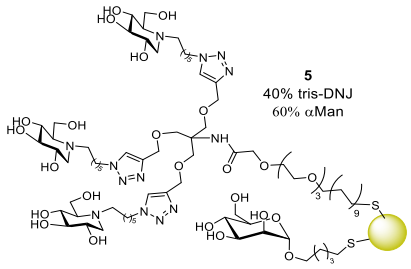
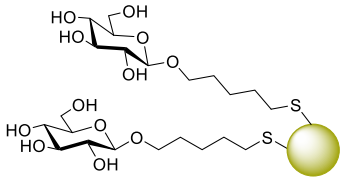
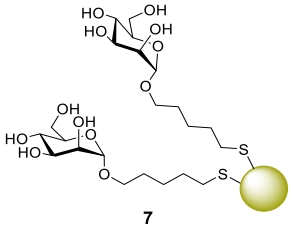


Figure S32. UV/vis spectrum of H_2O solution of AuGNP **5** recorded at concentration of 0.1 mg/mL.

Table S1. Summary table of DNJ-based AuGNPs **1**-**7** and their characterization.

Au-GNPs[1]	Characterization	
	Gold core size[2]	DNJ concentration for 2 mg/mL concentration of AuGNP[3]
 **1** 40% monoDNJ 60% βGlc	1.8±0.4 nm	413 µM
 **2** 40% monoDNJ 60% αMan	2.1±0.6 nm	467 µM
 **3** 20% tris-DNJ 80% βGlc	2.1±0.5 nm	567 µM
 **4** 20% tris-DNJ 80% αMan	2.0±0.4 nm	450 µM

 **5** 40% tris-DNJ 60% αMan	2.1±0.5 nm	503 µM
 **6** 100% Au-βGlc	1.7±0.4 nm	/
 **7** 100% Au-αMan	1.6±0.4 nm	/

[1] The given percentages refer to the proportion of the ligands on the gold surface as determined by recording ^{1}H NMR spectrum of their initial mixture before the formation of the AuGNPs and confirmed by ^{1}H NMR spectrum of the supernatant after the AuGNPs formation. [2] Determined by Transmission Electron Microscopy (TEM). [3] Determined on the basis of qNMR analysis.

Inhibition assays

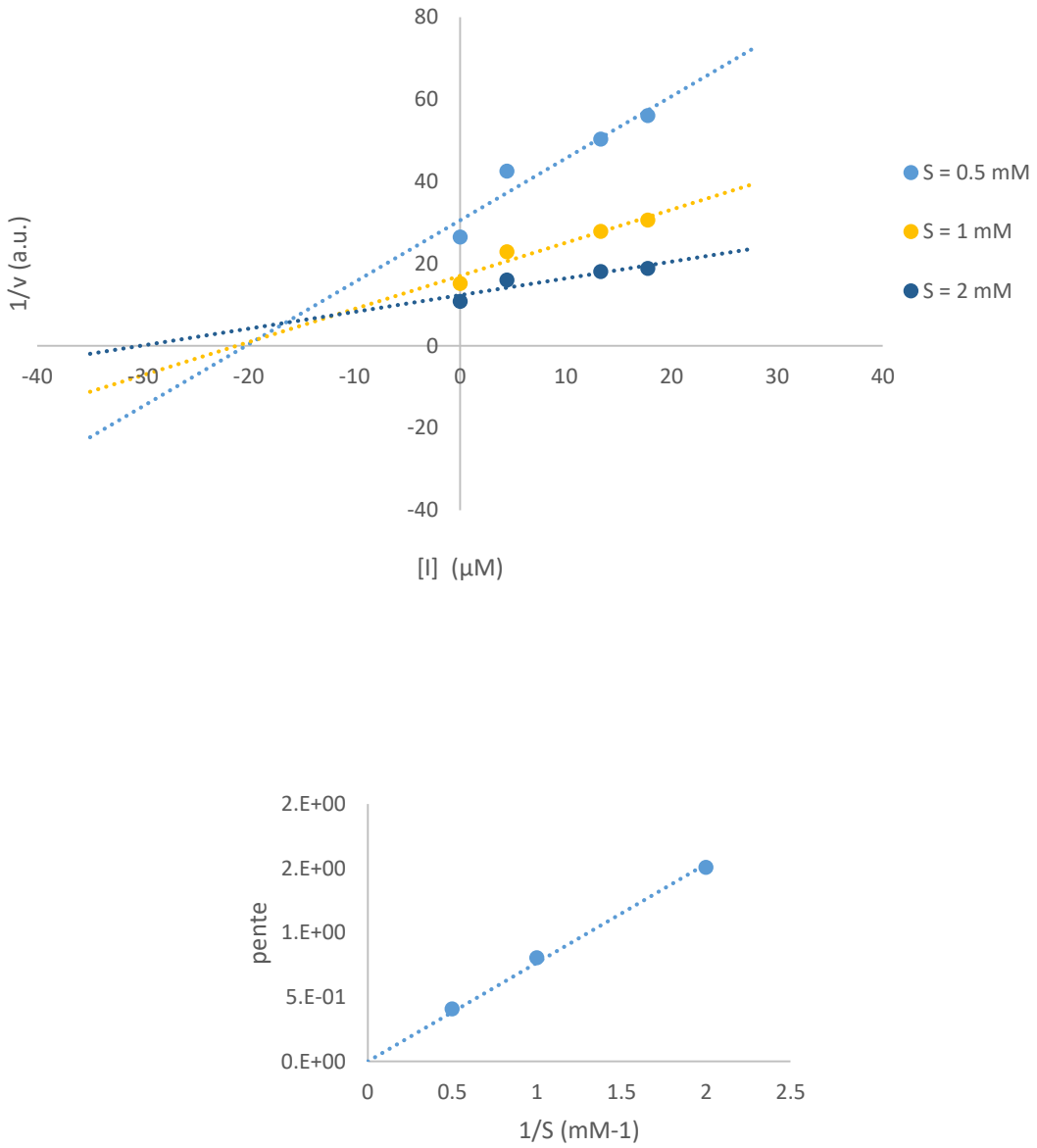


Figure S33. Dixon plot for K_i determination of compound AuGNP **1** against JBα-man and replot of the slopes showing competitive mode. K_i = 16 ± 2 µM.

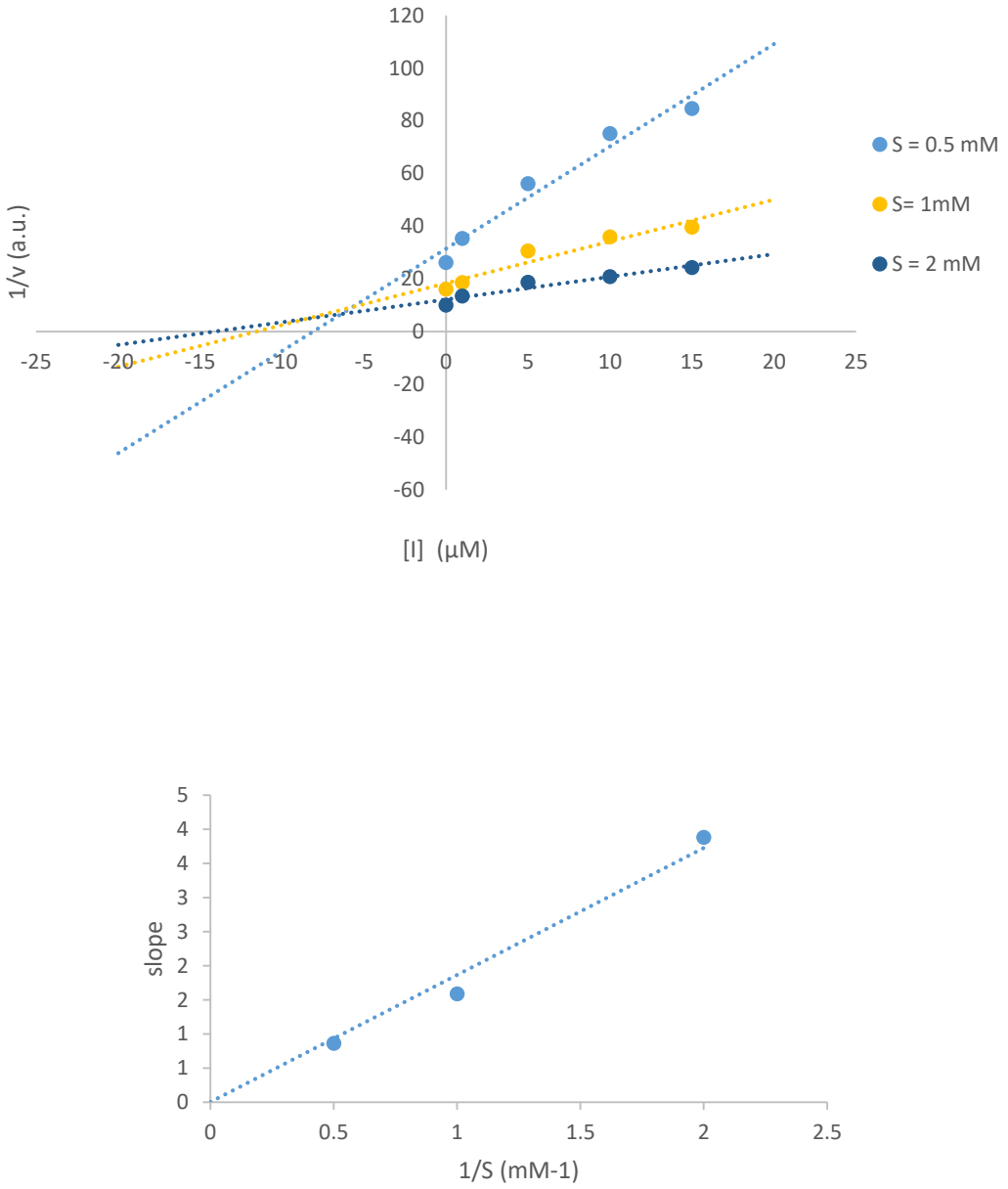
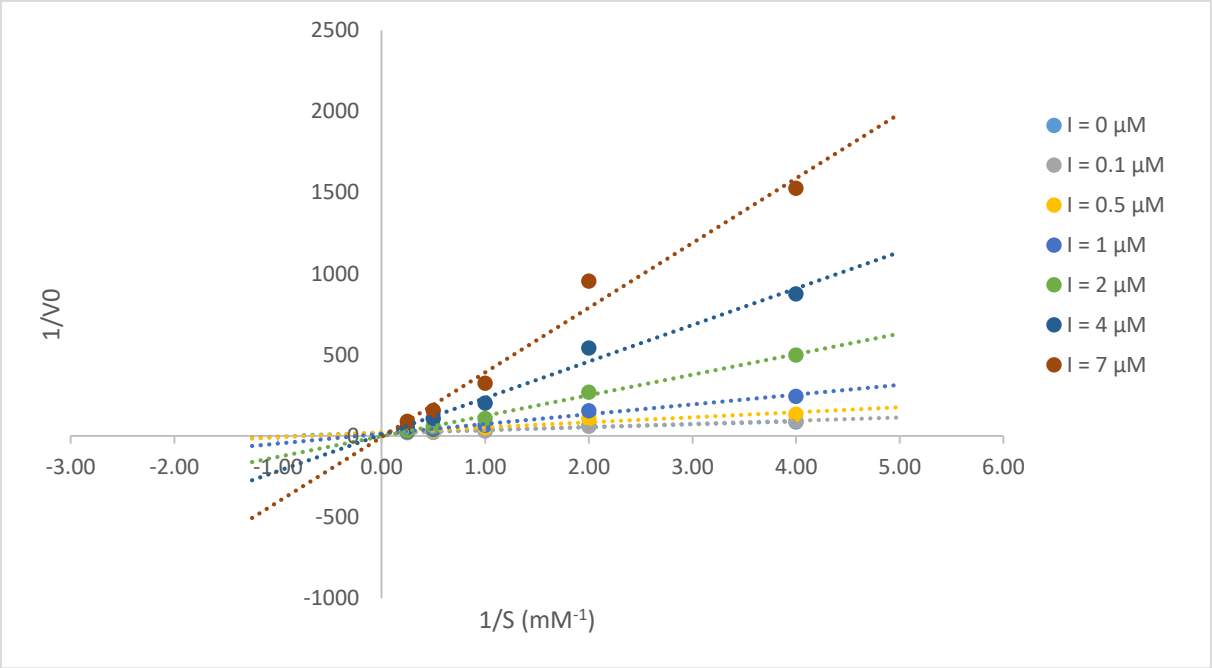


Figure S34. Dixon plot for K_i determination of compound AuGNP **2** against JBα-man and replot of the slopes showing competitive mode. K_i = 8 ± 2 µM.



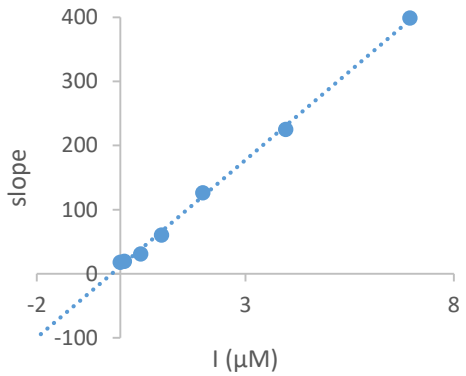


Figure S35. Lineweaver-Burk plots and replot of the slope versus inhibitor concentration for K_i determination of compound AuGNP **3** against JBα-man.

K_i = 0.198 ± 0.060 µM.

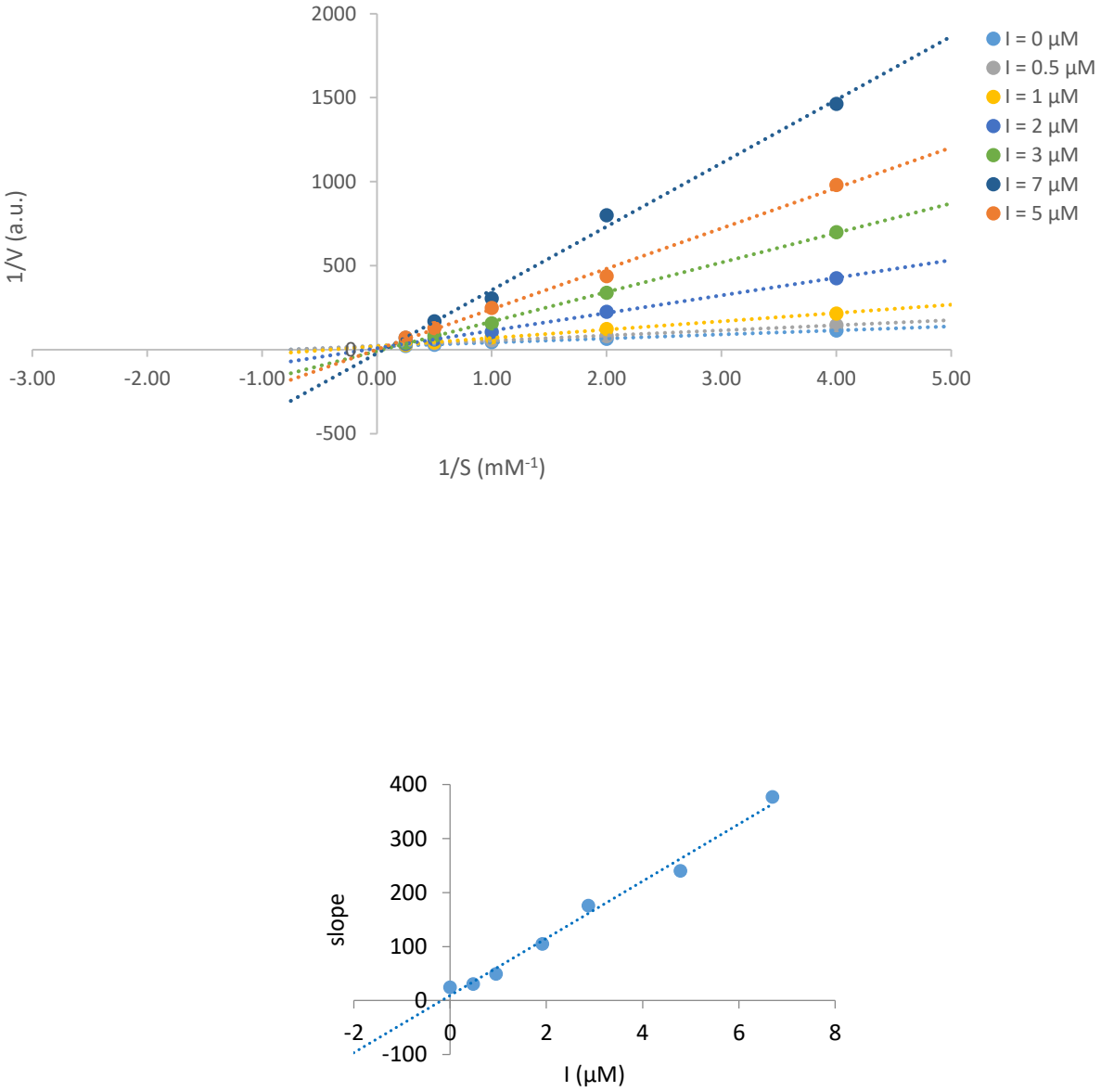


Figure S36. Lineweaver-Burk plots and replot of the slope versus inhibitor concentration for K_i determination of compound AuGNP **4** against JBα-man.

K_i = 0.175 ± 0.171 µM.

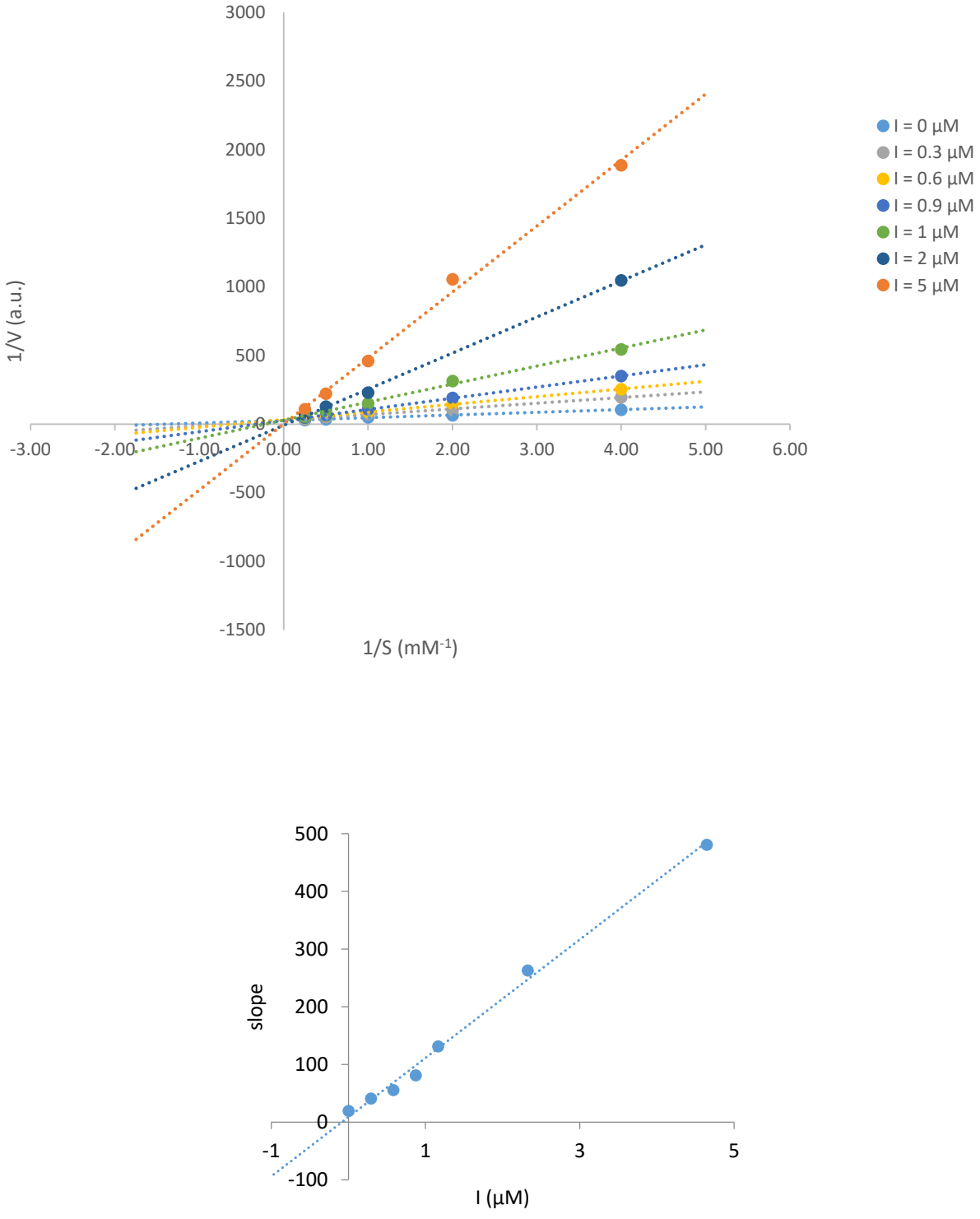


Figure S37. Lineweaver-Burk plots and replot of the slope versus inhibitor concentration for K_i determination of compound AuGNP **5** against JBα-man.
K_i = 0.084 ± 0.066 µM.