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Supplementary Material

Hybrid Orthorhombic Carbon Flakes Intercalated with Bimetallic Au-Ag Nanoclusters: Influence of Synthesis Parameters on Optical Properties

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SMC solution preparation

$(AuC_2Ph)_n$ [1], $(AgC_2Ph)_n$ [2], and $(PhC_2Au)_2\{PPh_2(C_6H_4)_3PPh_2\}$ [3] were synthesized according to published procedures. Other reagents and solvents were used as received. Solution 1H and 31P NMR spectra were recorded on Bruker Avance 400 spectrometer. Microanalyses were carried out in the analytical laboratory of University of Eastern Finland (Department of Chemistry).

$[\{Au_{10}Ag_{12}(C_2Ph)_{20}\}Au_3\{PPh_2(C_6H_4)_3PPh_2\}_3][PF_6]_5$ (SMC) was synthesized according to published procedure [4].

$(PhC_2Au)_2\{PPh_2(C_6H_4)_3PPh_2\}$ (50 mg, 0.042 mmol), $(AuC_2Ph)_n$ (29 mg, 0.097 mmol) and $(AgC_2Ph)_n$ (20 mg, 0.096 mmol) were suspended in acetone (12 ml) and a solution of $AgPF_6$ (18 mg, 0.071 mmol) in acetone (3 ml) was added resulting in an orange solution. The reaction mixture was stirred for 20 hours in the absence of light in argon atmosphere. After filtration, the resulting solution was evaporated and the target compound was recrystallized twice by gas-phase diffusion of pentane into its acetone solution (89 mg, 75%). $^{31}P\{^1H\}$ NMR ($(CD_3)_2CO$; δ): 43.5 (s, 6P), −144.8 (sept, 5P, PF_6). ^1H NMR ($(CD_3)_2CO$; δ): $\{PPh_2(C_6H_4)_3PPh_2\}$: 8.42 (s, –C_6H_4–, 12H), 8.35 (d, *m*–H, (–C_6H_4–P), 12H, $J_{(H–H)}$ = 8.2 Hz), 7.93 (m, *o*–H, (Ph–P), 24H, $J_{(H–H)}$ = 6.7, $J_{(P–H)}$ = 14 Hz), 7.71 (m, *o*–H, (–C_6H_4–P), 12H, $J_{(H–H)}$ = 8.2, $J_{(P–H)}$ = 15 Hz), 7.67 (t, *p*–H, (PhP), 12H, $J_{(H–H)}$ = 7.7 Hz), 7.42 (dd, *m*–H, (Ph–P), 24H, $J_{(H–H)}$ = 8.2, 7.7 Hz); $\{Au(C_2Ph)_2\}$ (three sets A:B:C = 1:3:6); {A}: 7.02 (t, *p*–H, 2H, $J_{(H–H)}$ =.4 Hz), 6.85 (d, *o*–H, 4H, $J_{(H–H)}$ = 7.0 Hz), 5.86 (dd, *m*–H, 4H, $J_{(H–H)}$ = 7.0, 7.4 Hz); {B}: 7.27 (t, *p*–H, 6H, $J_{(H–H)}$ = 7.4 Hz), 7.00 (dd, *m*–H, 12H, $J_{(H–H)}$ = 7.4, 8.0 Hz), 6.49 (d, *o*–H, 12H, $J_{(H–H)}$ = 8.0 Hz); {C}: 7.14 (t, *p*–H, 12H, $J_{(H–H)}$ = 7.5 Hz), 6.92 (d, *o*–H, 24H, $J_{(H–H)}$ = 8.1 Hz), 6.55 (dd, *m*–H, 24H, $J_{(H–H)}$ = 7.5, 8.1 Hz). Anal. calcd for $C_{286}H_{196}Ag_{12}Au_{13}F_{30}P_{11}$: C, 40.90; H, 2.35. Found: C, 40.77; H, 2.38.

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