

Supporting Information

Dinuclear palladium–azido complexes containing thiophene derivatives: reactivity toward organic isocyanides and isothiocyanates

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Experimental

Data for **3**: δ_{H} (CDCl₃): 1.22 (36 H, s, P(CH₃)₃), 7.12 (2 H, br), 7.19 (2 H, s), 7.72 (2 H, br), 8.57 (2 H, d, $J = 4.2$ Hz), 9.27 (2 H, d, $J = 7.3$ Hz); δ_{P} (CDCl₃): -17.01 (s).

Data for **4**: δ_{H} (CDCl₃): 1.05 (36 H, qnt, $J = 7.7$ Hz, P(CH₂CH₃)₃), 1.60 (24 H, br, P(CH₂CH₃)₃), 7.11 (2 H, m), 7.22 (2 H, s), 7.68 (2 H, m), 8.58 (2 H, br), 9.43 (2 H, br); δ_{P} (CDCl₃): 14.06 (s).

Data for **5**: δ_{H} (CDCl₃): 1.22 (36 H, s, P(CH₃)₃), 7.10 (2 H, dd, $J = 1.1, 4.8$ Hz), 7.11 (2 H, s), 7.72 (2 H, dd, $J = 1.8, 7.8$ Hz), 8.57 (2 H, m), 9.03 (2 H, d, $J = 7.7$ Hz); δ_{C} (CDCl₃): 13.6 (s, P(CH₃)₃), 118.1, 120.8, 130.8, 136.2, 138.4, 138.6, 145.1, 149.5, 154.6; δ_{P} (CDCl₃): -14.19 (s).

Data for **6**: 2037 (vs); δ_{H} (CDCl₃): 1.08 (36 H, br, P(CH₂CH₃)₃), 1.56 (24 H, br, P(CH₂CH₃)₃), 7.09 (2 H, dd, $J = 1.1, 5.0$ Hz), 7.14 (2H, s), 7.66 (2 H, dd, $J = 1.8, 7.5$

Hz), 8.56 (2 H, m), 9.19 (2 H, br); δ_{C} (CDCl_3): 8.0 (s, $\text{P}(\text{CH}_2\text{CH}_3)_3$), 14.7 (br, $\text{P}(\text{CH}_2\text{CH}_3)_3$), 119.0, 120.8, 131.6, 135.9, 138.1, 138.5, 144.6, 149.4, 154.9; δ_{P} (CDCl_3): -14.70 (s).

Data for **7**: δ_{H} (CDCl_3): 1.23 (36 H, s, $\text{P}(\text{CH}_3)_3$), 7.11 (2 H, dd, $J = 1.0, 5.0$ Hz), 7.15 (2 H, s), 7.17 (2 H, s), 7.70 (2 H, dd, $J = 1.8, 8.0$ Hz), 8.56 (2 H, m), 9.24 (2 H, d, $J = 8.0$ Hz); δ_{P} (CDCl_3): -14.65 (s).

Data for **8**: δ_{H} (CDCl_3): 1.22 (36H, s, $\text{P}(\text{CH}_3)_3$), 7.10 (2 H, s), 7.11 (2 H, dd, $J = 1.1, 4.9$ Hz), 7.16 (2 H, s), 7.71 (2 H, dd, $J = 1.8, 8.0$ Hz), 8.56 (2 H, m), 9.01 (2 H, d, $J = 8$ Hz); δ_{C} (CDCl_3): 13.8 (s, $\text{P}(\text{CH}_3)_3$), 118.3, 121.1, 124.8, 130.5, 136.5, 136.7, 138.3, 139.0, 145.4, 149.7, 154.7; δ_{P} (CDCl_3): -14.21 (s).

Data for **9**: δ_{H} (CDCl_3): 1.80 (18 H, d, $J = 11$ Hz, $\text{P}(\text{CH}_3)_3$), 7.01 (2 H, d, $J = 1.8$ Hz), 7.13 (4 H, m), 7.32 (2 H, m), 7.72 (2 H, dd, $J = 1.7, 7.7$ Hz), 8.57 (2 H, m), 9.22 (2 H, m); δ_{P} (CDCl_3): -3.97 (s).

Data for **10**: δ_{H} (CDCl_3): 1.80 (18 H, d, $J = 11$ Hz, $\text{P}(\text{CH}_3)_3$), 6.95 (2 H, d, $J = 1.7$ Hz), 7.14 (4 H, overlap), 7.34 (2 H, dq, $J = 1.0, 7.9$ Hz), 7.77 (2 H, dd, $J = 1.7, 7.7$ Hz), 8.76 (2 H, m); δ_{C} (CDCl_3): 15.9 (d, $J(\text{CP}) = 33$ Hz, $\text{P}(\text{CH}_3)_3$), 117.5, 120.5 (d, $J(\text{CP}) = 3.1$ Hz), 125.1, 129.9, 130.1, 136.4, 138.3, 139.7, 148.7, 155.7, 158.7; δ_{P} (CDCl_3): -4.10(s).

Data for **11**: δ_{H} (CDCl_3): 1.19 (36 H, s, $\text{P}(\text{CH}_3)_3$), 2.43 (12 H, s, CH_3), 6.71 (2 H, t, $J = 7.3$ Hz, -Ph), 6.95 (4 H, d, $J = 7.5$ Hz, -Ph), 7.11 (2 H, dd, $J = 1.1, 4.8$ Hz), 7.15 (2 H, s), 7.71 (2 H, dd, $J = 1.8, 7.5$ Hz), 8.56 (2 H, m), 9.13 (2 H, d, $J = 7.6$ Hz); δ_{C} (CDCl_3): 14.0 (br, $\text{P}(\text{CH}_3)_3$), 19.4 (CH_3), 118.2, 119.6, 120.8, 127.8, 131.0, 131.2, 136.2, 138.7, 144.8, 149.5, 154.9; δ_{P} (CDCl_3): -14.63 (s).

Data for **12**: δ_{H} (CDCl_3): 1.19 (36 H, s, $\text{P}(\text{CH}_3)_3$), 1.30 (24 H, d, $J = 6.9$ Hz,

$\text{CH}(\text{CH}_3)_2$, 3.73 (4 H, sep, $J = 6.9$ Hz, $\text{CH}(\text{CH}_3)_2$), 6.92 (2 H, dd, $J = 6.9, 8.3$ Hz, –Ph), 7.06 (4 H, d, $J = 7.5$ Hz, –Ph), 7.10 (2 H, dd, $J = 1.0, 4.9$ Hz), 7.13 (2 H, s), 7.68 (2 H, dd, $J = 1.8, 7.5$ Hz), 8.56 (2 H, m), 9.14 (2 H, br); δ_{C} (CDCl_3): 14.1 (br, $\text{P}(\text{CH}_3)_3$), 23.6 ($\text{CH}(\text{CH}_3)_2$), 28.4 ($\text{CH}(\text{CH}_3)_2$), 118.2, 120.7, 120.8, 122.5, 131.3, 136.2, 138.7, 138.8, 141.8, 142.0, 149.5, 155.0; δ_{P} (CDCl_3): –14.68 (s).

Data for **13**: δ_{H} (CDCl_3): 1.20 (36 H, s, $\text{P}(\text{CH}_3)_3$), 2.43 (12 H, s, CH_3), 6.71 (2 H, t, $J = 7.3$ Hz, –Ph), 6.95 (4 H, d, $J = 7.5$ Hz, –Ph), 7.11 (2 H, dd, $J = 1.1, 4.8$ Hz), 7.13 (4 H, d, $J = 12$ Hz), 7.70 (2 H, dd, $J = 1.8, 7.5$ Hz), 8.56 (2 H, m), 9.11 (2 H, d, $J = 7.9$ Hz); δ_{C} (CDCl_3): 14.1 (br, $\text{P}(\text{CH}_3)_3$), 19.5 (CH_3), 118.2, 119.6, 120.9, 124.6, 127.8, 130.6, 130.9, 136.2, 136.6, 138.1, 139.1, 144.8, 146.1, 149.5, 154.8; δ_{P} (CDCl_3): –14.63 (s).

Data for **14**: 2115 (vs); δ_{H} (CDCl_3): 1.19 (36 H, s, $\text{P}(\text{CH}_3)_3$), 1.30 (24 H, d, $J = 7.0$ Hz, $\text{CH}(\text{CH}_3)_2$), 3.75 (4 H, sep, $J = 7.0$ Hz, $\text{CH}(\text{CH}_3)_2$), 6.91 (2 H, dd, $J = 7.0$ Hz, –Ph), 7.06 (4 H, d, $J = 7$ Hz, –Ph), 7.10 (4 H, m), 7.11 (2 H, d, $J = 7.0$ Hz), 7.68 (2 H, dd, $J = 1.7, 4.0$ Hz), 8.55 (2 H, d, $J = 4.0$ Hz), 9.12 (2 H, d, $J = 7.5$ Hz); δ_{C} (CDCl_3): 14.1 (br, $\text{P}(\text{CH}_3)_3$), 23.6 ($\text{CH}(\text{CH}_3)_2$), 28.4 ($\text{CH}(\text{CH}_3)_2$), 118.2, 120.6, 120.9, 122.5, 124.6, 130.2, 130.6, 136.1, 136.6, 138.1, 139.2, 141.8, 142.0, 149.5, 154.8; δ_{P} (CDCl_3): –14.72 (s).

Data for **15**: δ_{H} (CDCl_3): 1.25 (24 H, d, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)_2$), 1.76 (18 H, d, $J = 11$ Hz, $\text{P}(\text{CH}_3)_3$), 3.73 (4 H, sep, $J = 6.9$ Hz, $\text{CH}(\text{CH}_3)_2$), 6.91 (2 H, dd, $J = 6.8, 8.2$ Hz, –Ph), 6.99 (2 H, d, $J = 1.7$ Hz, –Ph), 7.04 (4 H, d, $J = 7.4$ Hz), 7.07–7.09 (2H, m, –Ph), 7.34 (2 H, m), 7.74 (2 H, dd, $J = 1.7, 7.7$ Hz), 8.81 (2 H, m); δ_{C} (CDCl_3): 16.5 (d, $J(\text{CP}) = 33$ Hz $\text{P}(\text{CH}_3)_3$), 23.6 ($\text{CH}(\text{CH}_3)_2$), 28.3 ($\text{CH}(\text{CH}_3)_2$), 117.3 (d, $J(\text{CP}) = 2.5$ Hz), 120.3, 120.4, 120.8, 122.6, 130.8 (d, $J = 8.7$ Hz), 139.5, 141.5, 141.9, 149.2, 158.8 (d, $J(\text{CP}) = 2.5$ Hz); δ_{P} (CDCl_3): –3.66 (s).

Data for **16**: δ_{H} (CDCl_3): 1.26 (24 H, d, $J = 7.0$ Hz, $\text{CH}(\text{CH}_3)_2$), 1.77 (18 H, d, $J = 11$ Hz, $\text{P}(\text{CH}_3)_3$), 3.74 (4 H, sep, $J = 6.9$ Hz, $\text{CH}(\text{CH}_3)_2$), 6.90 (2 H, dd, $J = 6.8, 8.2$ Hz, -Ph), 6.97 (2 H, d, $J = 1.5$ Hz, -Ph), 7.03–7.08 (6H, overlap), 7.14 (2 H, s), 7.33 (2 H, dd, $J = 1.0, 8.0$ Hz), 7.74 (2 H, dd, $J = 1.7, 7.7$ Hz), 8.80 (2 H, m); δ_{C} (CDCl_3): 16.6 (d, $J(\text{CP}) = 33$ Hz $\text{P}(\text{CH}_3)_3$), 23.6 ($\text{CH}(\text{CH}_3)_2$), 28.4 ($\text{CH}(\text{CH}_3)_2$), 117.4 (d, $J(\text{CP}) = 1.9$ Hz), 120.3 (d, $J(\text{CP}) = 3.7$ Hz), 120.8, 122.6, 125.0, 130.2 (d, $J(\text{CP}) = 8.1$ Hz), 130.5 (d, $J(\text{CP}) = 2.5$ Hz), 136.4, 138.2 (d, $J(\text{CP}) = 4.3$ Hz), 139.5, 141.9, 142.1 (d, $J(\text{CP}) = 2.5$ Hz), 149.1, 156.7 (d, $J(\text{CP}) = 10$ Hz), 158.9 (d, $J(\text{CP}) = 3.1$ Hz); δ_{P} (CDCl_3): -3.59 (s)

Data for **17**: δ_{H} (CD_2Cl_2): 1.10 (36 H, s, $\text{P}(\text{CH}_3)_3$), 1.57 (12 H, d, $J = 6.7$ Hz, $\text{CH}(\text{CH}_3)_2$), 5.02 (2 H, sep, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)_2$), 7.11 (2 H, dd, $J = 1.0, 4.9$ Hz), 7.24 (2 H, s), 7.93 (2 H, dd, $J = 1.8, 6.3$ Hz), 8.54 (2 H, m), 9.80 (2 H, br); δ_{C} (CD_2Cl_2): 14.1 (t, $J = 14$ Hz, $\text{P}(\text{CH}_3)_3$), 21.9 ($\text{CH}(\text{CH}_3)_2$), 49.9 ($\text{CH}(\text{CH}_3)_2$), 120.7, 121.1, 131.5, 137.1, 139.0, 140.2, 149.2, 155.2; δ_{P} (CD_2Cl_2): -15.24 (s).

Data for **18**: δ_{H} (CDCl_3): 1.11 (36 H, s, $\text{P}(\text{CH}_3)_3$), 1.53 (6 H, t, $J = 7.2$ Hz, CH_2CH_3), 4.40 (4 H, q, $J = 7.2$ Hz, CH_2CH_3), 7.11 (2 H, dd, $J = 1.0, 4.9$ Hz), 7.24 (2 H, s), 7.93 (2 H, dd, $J = 1.8, 8.0$ Hz), 8.54 (2 H, br), 9.77 (2 H, br); δ_{C} (CDCl_3): 14.1 (t, $J = 16$ Hz, $\text{P}(\text{CH}_3)_3$), 14.3 (CH_2CH_3), 42.2 (CH_2CH_3), 120.5 (br), 121.0, 131.3 (br), 137.1, 138.6, 139.7 (br), 149.1, 154.6; δ_{P} (CDCl_3): -15.25 (s).

Data for **19**: δ_{H} (CDCl_3): 1.11 (36 H, s, $\text{P}(\text{CH}_3)_3$), 4.97 (4 H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.28 (4 H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 6.05 (2 H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 7.11 (2 H, dd, $J = 1.0, 4.9$ Hz), 7.18 (2 H, s), 7.21 (2 H, s), 7.90 (2 H, dd, $J = 1.8, 8.0$ Hz), 8.54 (2 H, m), 9.68 (2 H, br); δ_{C} (CDCl_3): 14.1 (t, $J = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 49.7 ($\text{CH}_2\text{CH}=\text{CH}_2$), 118.9 ($\text{CH}_2\text{CH}=\text{CH}_2$), 120.3, 121.1, 124.5, 130.6, 131.1 ($\text{CH}_2\text{CH}=\text{CH}_2$), 136.7, 137.0, 137.9, 149.2; δ_{P} (CDCl_3): -15.29 (s).

Data for **20**: δ_{H} (CDCl_3): 1.29 (36 H, t, $J = 3.7$ Hz, $\text{P}(\text{CH}_3)_3$), 6.45 (2 H, d, $J = 3.5$ Hz), 6.95 (2 H, d, $J = 3.5$ Hz); δ_{C} (CDCl_3): 13.4 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 122.6, 129.3 (t, $J(\text{CP}) = 5.0$ Hz), 139.9 (t, $J(\text{CP}) = 11$ Hz), 141.0 (m); δ_{P} (CDCl_3): -15.11 (s).

Data for **21**: δ_{H} (CDCl_3): 1.27 (36 H, t, $J = 3.7$ Hz, $\text{P}(\text{CH}_3)_3$), 6.51 (2 H, d, $J = 3.3$ Hz), 6.89 (2 H, s), 7.07 (2 H, d, $J = 3.5$ Hz); δ_{C} (CDCl_3): 13.3 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 122.1, 124.0, 129.7 (t, $J(\text{CP}) = 5.0$ Hz), 135.9, 139.5 (t, $J(\text{CP}) = 3.1$ Hz), 143.0 (t, $J(\text{CP}) = 11$ Hz); δ_{P} (CDCl_3): -14.87 (s).

Data for **22**: δ_{H} (CDCl_3): 1.52 (24 H, t, $J = 3.5$ Hz, $\text{P}(\text{CH}_3)_2\text{Ph}$), 6.09 (2 H, d, $J = 3.5$ Hz), 6.76 (2 H, s), 6.88 (2 H, d, $J = 3.5$ Hz), 7.35–7.40 (12 H, m), 7.57–7.64 (8 H, m); δ_{C} (CDCl_3): 12.4 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_2\text{Ph}$), 122.1, 123.9, 128.4 (t, $J(\text{CP}) = 4.7$ Hz), 129.8, 130.1, 131.1 (t, $J(\text{CP}) = 5.9$ Hz), 134.0 (t, $J(\text{CP}) = 22$ Hz), 135.6, 135.9, 139.9, 142.3 (t, $J(\text{CP}) = 11$ Hz); δ_{P} (CDCl_3): -6.60 (s).

Data for **23**: δ_{H} (CDCl_3): 1.29 (36 H, t, $J = 3.5$ Hz, $\text{P}(\text{CH}_3)_3$), 6.42 (2 H, d, $J = 3.5$ Hz), 6.95 (2 H, d, $J = 3.3$ Hz); δ_{C} (CDCl_3): 13.1 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 122.7, 129.5 (t, $J(\text{CP}) = 5.0$ Hz), 138.3 (t, $J(\text{CP}) = 12$ Hz), 141.1; δ_{P} (CDCl_3): -14.26 (s).

Data for **24**: δ_{H} (CDCl_3): 1.28 (36 H, t, $J = 3.5$ Hz, $\text{P}(\text{CH}_3)_3$), 6.48 (2 H, d, $J = 3.5$ Hz), 6.89 (2 H, s), 7.06 (2 H, d, $J = 3.5$ Hz); δ_{C} (CDCl_3): 13.0 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 121.2, 124.0, 129.8 (t, $J(\text{CP}) = 4.3$ Hz), 135.8, 139.6 (t, $J(\text{CP}) = 3.2$ Hz), 141.3 (t, $J(\text{CP}) = 11$ Hz); δ_{P} (CDCl_3): -14.10 (s).

Data for **25**: δ_{H} (CDCl_3): 1.52 (24 H, t, $J = 3.4$ Hz, $\text{P}(\text{CH}_3)_2\text{Ph}$), 6.10 (2H, d, $J = 3.5$ Hz), 6.78 (2 H, s), 6.90 (2 H, d, $J = 3.5$ Hz), 7.38–7.44 (12 H, m, Ph), 7.53–7.60 (8 H, m, Ph); δ_{C} (CDCl_3): 11.9 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_2\text{Ph}$), 122.1, 123.9, 128.6 (t, $J(\text{CP}) = 5.0$ Hz), 129.8, 130.1, 130.8 (t, $J(\text{CP}) = 5.9$ Hz), 133.0 (t, $J(\text{CP}) = 22$ Hz), 135.9, 139.9, 140.8 (t, $J(\text{CP}) = 11$ Hz); δ_{P} (CDCl_3): -5.41 (s).

Data for **26**: δ_{H} (CDCl_3): 1.27 (36 H, t, $J = 3.5$ Hz, $\text{P}(\text{CH}_3)_3$), 2.38 (12 H, s, CH_3), 6.44 (2 H, d, $J = 3.5$ Hz), 6.68 (2 H, t, $J = 7.4$ Hz, $-\text{Ph}$), 6.92 (2 H, d, $J = 7.4$ Hz, $-\text{Ph}$), 6.95 (2 H, d, $J = 3.3$ Hz); δ_{C} (CDCl_3): 13.5 (t, $J = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 19.4 (CH_3), 119.5, 122.6, 127.7, 129.7 (t, $J = 5.0$ Hz), 131.0, 141.2, 144.6; δ_{P} (CDCl_3): -14.87 (s).

Data for **27**: δ_{H} (CDCl_3): 1.25 (24 H, d, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)_2$), 1.26 (36 H, t, $J = 3.5$ Hz, $\text{P}(\text{CH}_3)_3$), 3.68 (4 H, sep, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)_2$), 6.44 (2 H, d, $J = 3.4$ Hz), 6.68 (2 H, dd, $J = 6.9, 8.3$ Hz, $-\text{Ph}$), 6.95 (2 H, d, $J = 3.4$ Hz), 7.02 (4 H, d, $J = 7.4$ Hz, $-\text{Ph}$); δ_{C} (CDCl_3): 13.4 (t, $J = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 23.5 ($\text{CH}(\text{CH}_3)_2$), 28.3 ($\text{CH}(\text{CH}_3)_2$), 120.6, 122.5, 122.6, 129.7 (t, $J = 5.0$ Hz), 130.0, 139.0, 141.2, 141.8, 141.9; δ_{P} (CDCl_3): -14.99 (s).

Data for **28**: δ_{H} (CDCl_3): 1.20 (36 H, t, $J = 3.7$ Hz, $\text{P}(\text{CH}_3)_3$), 4.93 (4 H, dt, $J = 1.4, 5.9$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.21 (1 H, d, $J = 1.1$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.27 (m, 2H, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.30 (1 H, d, $J = 1.1$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 6.02 (2 H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 6.60 (2 H, d, $J = 3.3$ Hz), 7.04 (2 H, d, $J = 3.5$ Hz); δ_{C} (CDCl_3): 13.6 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 49.1 ($\text{CH}_2\text{CH}=\text{CH}_2$), 118.9 ($\text{CH}_2\text{CH}=\text{CH}_2$), 122.8, 130.1 (t, $J = 5.0$ Hz), 131.1 ($\text{CH}_2\text{CH}=\text{CH}_2$), 141.5 (t, $J(\text{CP}) = 2.5$ Hz); δ_{P} (CDCl_3): -15.25 (s).

Data for **29**: δ_{H} (CDCl_3): 1.20 (36 H, t, $J = 3.7$ Hz, $\text{P}(\text{CH}_3)_3$), 1.49 (6 H, t, $J = 7.2$ Hz, CH_2CH_3), 4.36 (4 H, q, $J = 7.2$ Hz, CH_2CH_3), 6.61 (2 H, d, $J = 3.4$ Hz), 7.04 (2 H, d, $J = 3.3$ Hz); δ_{C} (CDCl_3): 13.6 (t, $J(\text{CP}) = 15$ Hz, $\text{P}(\text{CH}_3)_3$), 14.2 (CH_2CH_3), 42.1 (CH_2CH_3), 122.9, 130.1 (t, $J(\text{CP}) = 4.7$ Hz); δ_{P} (CDCl_3): -15.21 (s).