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Supporting Information

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Supporting Information

Tetraarylphosphonium halides as Arylating Reagents in the Pd-Catalyzed Heck and Cross-Coupling Reactions: Spectroscopic Observation of Intermediates and Reaction Scope

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General. NMR spectra were obtained on a Bruker AVANCE 400 spectrometer (^{1}H at 400 MHz and ^{13}C at 100 MHz). Chemical shifts were quoted in parts per million (ppm) referenced to the appropriate solvent peak or 0.0 ppm for tetramethylsilane (TMS). Column chromatography was carried out using silica gel 60 (230-400 mesh). Solvents were purified by standard procedures. Chemicals including Pd catalysts, boronic acids, alkynes and alkenes were purchased from Aldrich or TCI, and used without further purification. Tetraarylphosphonium salts were prepared according to the literature procedure starting from aryl halides and triarylphosphine [for references, see: a) T. Migita, T. Nagai, K. Kiuchi, M. Kosugi, *Bull. Chem. Soc. Jpn*. **1983**, *56*, 2869; b) M. H. Kowalski, R. J. Hinkle, P. J. Stang, P. J. *J. Org. Chem*. **1989**, *54*, 2783]. ESI (electrospray ionization) mass spectra were acquired using a Navigator quadruple mass spectrometer equipped with an electrospray ion source. The instrument was operated in the positive-ion mode (ES+) at a probe tip voltage of 3 kV.

Representative procedure for the Heck olefination reactions. $Pd(OAc)_2$ (2.5 mg, 0.011 mmol), NaOAc (27.1 mg, 0.33 mmol) and *n*-butyl acrylate (42.3 mg, 0.33 mmol) were added to a solution of

tetraphenylphosphonium chloride (**1**, 41.2 mg, 0.11 mmol) in DMF (1.0 mL) at room temperature. After stirring the reaction mixture for 12 h at 130 ^{o}C under O_2 (1 atm using a balloon), the organic layer was extracted with ethyl acetate (10 mL x 3), washed with brine (20 mL x 3), and then dried over $MgSO_4$. The extracts were evaporated under reduced pressure followed by a column chromatography (EtOAc/hexane, 1:10) on silica gel to give *n*-butyl cinnamate (19.1 mg, 85%). When the same reaction was carried out under N_2 atmosphere rather than O_2, the product was isolated in 66% yield.

Representative procedure for the Suzuki coupling reactions. To a solution of tetraphenylphosphonium chloride (**1**, 41.2 mg, 0.11 mmol) in DMF (1.0 mL) was added $Pd(OAc)_2$ (2.5 mg, 0.011 mmol), NaOAc (27.1 mg, 0.33 mmol) and *o*-tolueneboronic acid (23.1 mg, 0.17 mmol) at room temperature. After the reaction mixture was stirred for 12 h at 100 ^{o}C under N_2, the organic layer was extracted with ethyl acetate (20 mL x 3), washed with water, and then dried over $MgSO_4$. The extracts were evaporated under reduced pressure followed by a column chromatography (EtOAc/hexane, 1:10) on silica gel to provide 4-phenylanisole (15.4 mg, 83%). When the same reaction was carried out under O_2 atmosphere rather than N_2, the product was isolated in 25% yield.

Representative procedure for the Sonogashira coupling reactions. 4-(Phenoxy)phenylacetylene (77.7 mg, 0.40 mmol), $Pd(OAc)_2$ (1.5 mg, 0.0067 mmol) and triethylamine (26.3 mg, 0.26 mmol) were added to a solution of tetraphenylphosphonium chloride (**1**, 48.7 mg, 0.13 mmol) in DMF (1.0 mL) at room temperature. After stirring the reaction mixture for 12 h at 100 ^{o}C under the nitrogen atmosphere, the organic layer was extracted with ethyl acetate (20 mL x 3), washed with water, and then dried over $MgSO_4$. The extracts were evaporated under reduced pressure followed by a column chromatography (EtOAc/hexane, 1:10) on silica gel to afford 4-(phenoxy)diphenylacetylene (34.8 mg, 99%) as a colorless solid. When the same reaction was carried out under O_2 atmosphere rather than N_2, the product was isolated in 54% yield.

Experimental details for the ESI-MS studies. To a solution of tetraphenylphosphonium chloride (**1**, 41.2 mg, 0.11 mmol) in CH_3CN (5.0 mL) was added $Pd_2(dba)_3$ (50.4 mg, 0.055 mmol) at room

temperature. While stirring the mixture at 100 ^{o}C, the sample aliquot was taken from the reaction vessel using a syringe at the indicated interval (5, 30, 60, 120, and 360 min, respectively) and was used subsequently for the ESI-MS analysis.

Experimental details for the crystal structure determination of [$(PPh_3)_2$PdPhCl] (2). $Pd_2(dba)_3$ (50.4 mg, 0.055 mmol) and PPh_3 (28.9 mg, 0.11 mmol) was added to a solution of tetraphenylphosphonium chloride (**1**, 41.2 mg, 0.11 mmol) in THF (5.0 mL) at room temperature. After stirring the mixture for 3 h at 80 ^{o}C, organic solvent was removed under reduced pressure. Ether (20 mL) was added to the residue and resulting precipitate was filtered washing with ether. Filtered solid was dissolved in acetone (1.0 mL), and a monocrystalline solid (**2**) was obtained by a slow diffusion of pentane into the acetone solution by standing for 2 days at room temperature in 54% yield, which structure was determined by a X-ray crystallographic analysis.

Reaction of the isolated arylpalladium intermediate 2 with an olefin. To a solution of [$(PPh_3)_2$PdPhCl] (**2**, 14.9 mg, 0.02 mmol), obtained from the above experiment, in DMF (0.4 mL) was added *n*-butyl acrylate (7.7 mg, 0.60 mmol) and sodium acetate (5.0 mg, 0.06 mmol) at room temperature. After stirring the reaction mixture for 12 h at 80 ^{o}C under O_2 atmosphere, the organic layer was extracted with ethyl acetate (10 mL x 3), washed with brine (20 mL x 3), and then dried over $MgSO_4$. The extracts were evaporated under reduced pressure followed by a column chromatography (EtOAc/hexane, 1:10) on silica gel to give *n*-butyl cinnamate (2.2 mg, 55%).

Figure S1. Comparison of the obtained ESI-MS data with calculated ones for the activated palladium intermediate complexes.

(Obtained ESI-MS data) (Calculated isotope ratio)

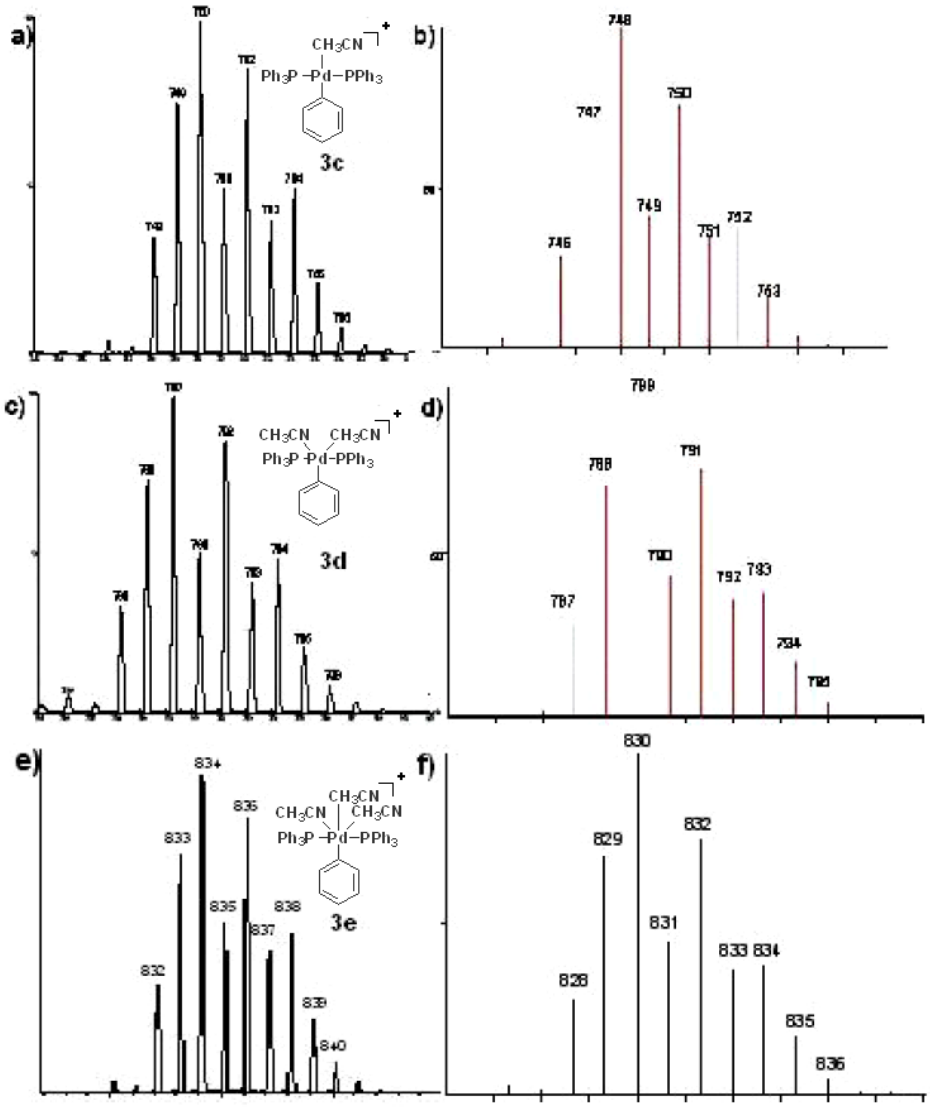

Figure S2. ORTEP drawing of the crystal structure of [$(PPh_3)_2$PdPhCl] (**2**).

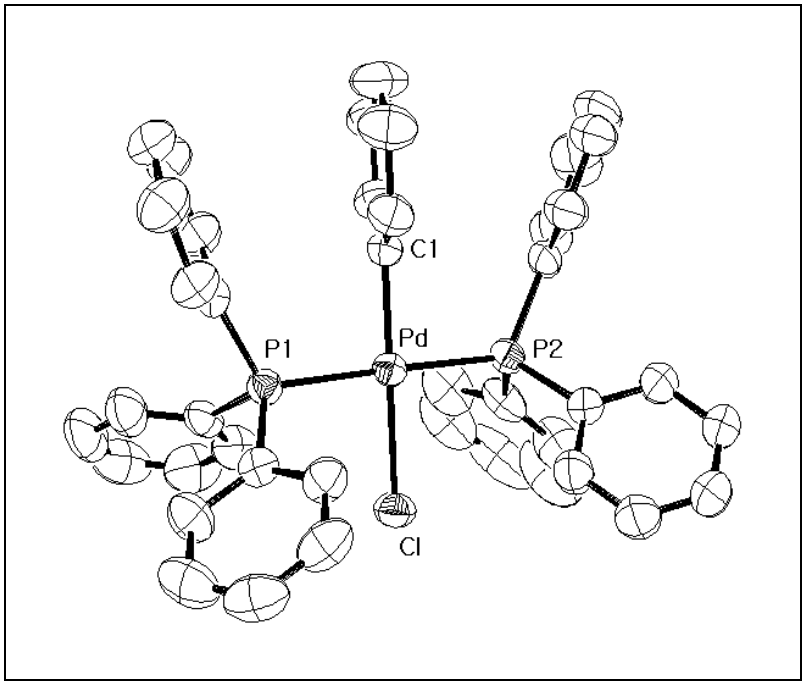

Table S1: Crystallographic data and structure refinement parameters of **2**.

	2
Empirical formula	$C_{42}H_{35}ClP_2Pd$
Formula weight	743.49
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
space group	P(bca)
Unit cell dimensions	a = 11.873 (16) Å alpha = 90 deg b = 23.80 (3) Å beta = 90 deg c = 25.49 (2) Å gamma = 90 deg
Volume	7205(14) $Å^3$
Z	8
Calculated density	1.371 Mg/m^3
Absorption coefficient	0.707 mm^{-1}
F(000)	3040
Theta range for data collection	1.60 to 28.00 deg
Limiting indices	-14<=h<=12, -29<=k<=28, -33<=l<=33
Reflections collected / unique	40191 / 8046 [R(int) = 0.0497]
Completeness to theta = 28.00	92.6 %
Absorption correction	empirical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8046 / 0 / 551
Goodness-of-fit on F^2	1.003
Final R indices [I>2sigma(I)]	R1 = 0.0368, wR2 = 0.0827
R indices (all data)	R1 = 0.0712, wR2 = 0.0967
Largest diff. peak and hole	0.400 and -0.502 $e.A^{-3}$

Table S2: Atomic coordinates and equivalent isotropic displacement parameters (A^2) for **2**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	**y**	**z**	**U (eq)**
Pd(1)	0.096201(17)	0.332742(9)	0.141891(8)	0.03665(8)
P(1)	0.12897(6)	0.42900(3)	0.14002(3)	0.03827(17)
Cl	0.08772(7)	0.33421(3)	0.04778(3)	0.0523(2)
P(2)	0.07053(6)	0.23617(3)	0.14073(3)	0.03843(17)
C(1)	0.1028(2)	0.33169(12)	0.22102(11)	0.0413(7)
C(25)	0.0839(2)	0.19596(13)	0.20140(12)	0.0458(7)
C(7)	0.1330(2)	0.46607(12)	0.20243(11)	0.0431(7)
C(19)	0.0173(2)	0.46411(12)	0.10311(10)	0.0423(7)
C(13)	0.2633(2)	0.44844(14)	0.10947(11)	0.0490(8)
C(32)	-0.1009(3)	0.15663(15)	0.12625(14)	0.0569(9)
C(24)	-0.0867(3)	0.43869(16)	0.10151(13)	0.0537(8)
C(31)	-0.0628(2)	0.21079(13)	0.11481(11)	0.0447(7)
C(30)	0.0025(4)	0.20328(17)	0.24003(13)	0.0608(9)
C(37)	0.1761(3)	0.20592(14)	0.09753(13)	0.0575(9)
C(8)	0.2208(3)	0.45479(16)	0.23645(13)	0.0562(9)
C(26)	0.1714(3)	0.15999(16)	0.21128(16)	0.0673(10)
C(2)	0.1972(3)	0.31179(15)	0.24686(13)	0.0582(9)
C(12)	0.0463(3)	0.49956(15)	0.21990(14)	0.0578(9)
C(20)	0.0312(3)	0.51469(15)	0.07735(14)	0.0615(9)
C(10)	0.1323(4)	0.50810(19)	0.30353(17)	0.0736(12)
C(28)	0.0977(5)	0.1398(2)	0.2960(2)	0.0971(17)
C(35)	-0.2230(4)	0.2219(2)	0.05858(17)	0.0882(15)

C(9)	0.2214(3)	0.47613(17)	0.28652(15)	0.0663(10)
C(6)	0.0137(3)	0.35132(15)	0.25039(13)	0.0575(9)
C(36)	-0.1265(3)	0.24388(17)	0.08145(14)	0.0642(10)
C(29)	0.0095(5)	0.1750(2)	0.28668(16)	0.0799(13)
C(22)	-0.1609(4)	0.5130(2)	0.04921(15)	0.0730(12)
C(14)	0.3101(3)	0.50086(19)	0.11715(17)	0.0698(10)
C(21)	-0.0577(5)	0.5384(2)	0.05051(17)	0.0800(14)
C(34)	-0.2566(4)	0.1689(2)	0.06929(17)	0.0906(15)
C(11)	0.0459(4)	0.52077(18)	0.27046(15)	0.0740(11)
C(17)	0.4228(4)	0.4238(3)	0.05632(18)	0.0906(15)
C(42)	0.2835(4)	0.2255(2)	0.1002(2)	0.0884(14)
C(18)	0.3201(3)	0.4102(2)	0.07906(14)	0.0648(10)
C(27)	0.1778(5)	0.1326(2)	0.2596(2)	0.0950(17)
C(5)	0.0192(5)	0.3502(2)	0.30489(16)	0.0817(14)
C(4)	0.1120(5)	0.3299(2)	0.32980(16)	0.0924(16)
C(3)	0.2011(5)	0.3111(2)	0.30137(16)	0.0795(13)
C(41)	0.3677(5)	0.2005(4)	0.0657(3)	0.112(2)
C(40)	0.3399(8)	0.1600(3)	0.0344(3)	0.140(4)
C(39)	0.2353(8)	0.1417(3)	0.0305(3)	0.140(3)
C(38)	0.1514(4)	0.16396(16)	0.06212(17)	0.0906(14)
C(33)	-0.1978(4)	0.13623(19)	0.10298(15)	0.0742(11)
C(16)	0.4684(4)	0.4752(3)	0.0640(2)	0.0970(18)
C(15)	0.4135(4)	0.5139(3)	0.0938(2)	0.0954(18)

Table S3: Bond lengths [Å] and angles [deg] for **2**.

Bond length [Å]		**Bond angles [deg]**	
Pd(1)-C(1)	2.019(3)	C(1)-Pd(1)-P(2)	90.32(8)
Pd(1)-P(2)	2.319(3)	C(1)-Pd(1)-P(1)	91.50(8)
Pd(1)-P(1)	2.325(3)	P(2)-Pd(1)-P(1)	177.18(3)
Pd(1)-Cl	2.401(2)	C(1)-Pd(1)-Cl	179.78(10)
P(1)-C(7)	1.820(3)	P(2)-Pd(1)-Cl	89.78(3)
P(1)-C(19)	1.828(3)	P(1)-Pd(1)-Cl	88.41(3)
P(1)-C(13)	1.835(4)	C(7)-P(1)-C(19)	104.33(14)
P(2)-C(37)	1.817(3)	C(7)-P(1)-C(13)	103.05(14)
P(2)-C(31)	1.819(3)	C(19)-P(1)-C(13)	107.26(15)
P(2)-C(25)	1.826(3)	C(7)-P(1)-Pd(1)	117.67(10)
C(1)-C(6)	1.377(5)	C(19)-P(1)-Pd(1)	109.87(11)
C(1)-C(2)	1.384(4)	C(13)-P(1)-Pd(1)	113.76(11)
C(25)-C(26)	1.370(5)	C(37)-P(2)-C(31)	104.40(18)
C(25)-C(30)	1.391(5)	C(37)-P(2)-C(25)	104.22(15)
C(7)-C(12)	1.377(5)	C(31)-P(2)-C(25)	102.08(14)
C(7)-C(8)	1.382(4)	C(37)-P(2)-Pd(1)	108.05(12)
C(19)-C(24)	1.377(5)	C(31)-P(2)-Pd(1)	116.63(10)
C(19)-C(20)	1.381(4)	C(25)-P(2)-Pd(1)	119.83(11)
C(13)-C(18)	1.373(5)	C(6)-C(1)-C(2)	118.7(3)
C(13)-C(14)	1.380(5)	C(6)-C(1)-Pd(1)	120.6(2)
C(32)-C(33)	1.382(5)	C(2)-C(1)-Pd(1)	120.7(2)
C(32)-C(31)	1.397(5)	C(26)-C(25)-C(30)	118.4(3)
C(24)-C(23)	1.385(5)	C(26)-C(25)-P(2)	123.3(3)
C(31)-C(36)	1.384(4)	C(30)-C(25)-P(2)	118.3(3)
C(30)-C(29)	1.369(5)	C(12)-C(7)C(8)	118.3(3)

C(37)-C(42)	1.360(6)	C(12)-C(7)-P(1)	122.9(2)
C(37)-C(38)	1.378(5)	C(8)-C(7)-P(1)	118.3(3)
C(8)-C(9)	1.374(5)	C(24)-C(19)-C(20)	118.4(3)
C(26)-C(27)	1.396(6)	C(24)-C(19)-P(1)	117.7(3)
C(2)-C(3)	1.390(5)	C(20)-C(19)-P(1)	123.8(3)
C(12)-C(11)	1.384(5)	C(18)-C(13)-C(14)	118.9(4)
C(20)-C(21)	1.379(5)	C(18)-C(13)-P(1)	119.9(3)
C(10)-C(11)	1.362(6)	C(14)-C(13)-P(1)	121.2(3)
C(10)-C(9)	1.373(6)	C(33)-C(32)-C(31)	120.3(3)
C(28)-C(27)	1.341(7)	C(19)-C(24)-C(23)	120.6(4)
C(28)-C(29)	1.362(7)	C(36)-C(31)-C(32)	118.5(3)
C(35)-C(34)	1.352(6)	C(36)-C(31)-P(2)	120.7(3)
C(35)-C(36)	1.388(5)	C(32)-C(31)-P(2)	120.8(2)
C(6)-C(5)	1.391(5)	C(29)-C(30)-C(25)	120.7(4)
C(22)-C(23)	1.357(6)	C(42)-C(37)-C(38)	118.7(4)
C(22)-C(21)	1.366(6)	C(42)-C(37)-P(2)	118.7(3)
C(14)-C(15)	1.399(6)	C(38)-C(37)-P(2)	122.5(3)
C(34)-C(33)	1.352(6)	C(9)-C(8)-C(7)	121.0(4)
C(17)-C(16)	1.351(7)	C(25)-C(26)-C(27)	119.7(4)
C(17)-C(18)	1.388(6)	C(1)-C(2)-C(3)	120.4(4)
C(42)-C(41)	1.458(8)	C(7)-C(12)-C(11)	121.3(3)
C(5)-C(4)	1.360(6)	C(21)-C(20)-C(19)	120.1(4)
C(4)-C(3)	1.359(6)	C(11)-C(10)-C(9)	120.5(4)
C(41)-C(40)	1.295(10)	C(27)-C(28)-C(29)	120.2(4)
C(40)-C(39)	1.319(11)	C(34)-C(35)-C(36)	120.7(4)
C(39)-C(38)	1.386(7)	C(10)-C(9)-C(8)	119.6(4)
C(16)-C(15)	1.362(7)	C(1)-C(6)-C(5)	120.0(4)

		C(31)-C(36)-C(35)	119.7(4)
		C(28)-C(29)-C(30)	120.1(5)
		C(23)-C(22)-C(21)	119.0(4)
		C(13)-C(14)-C(15)	119.6(5)
		C(22)-C(23)-C(24)	120.7(4)
		C(22)-C(21)-C(20)	121.2(4)
		C(35)-C(34)-C(33)	120.8(4)
		C(10)-C(11)-C(12)	119.5(4)
		C(16)-C(17)-C(18)	120.2(5)
		C(37)-C(42)-C(41)	118.2(6)
		C(13)-C(18)-C(17)	120.8(5)
		C(28)-C(27)-C(26)	120.8(5)
		C(4)-C(5)-C(6)	120.7(4)
		C(3)-C(4)-C(5)	119.9(4)
		C(4)-C(3)-C(2)	120.2(4)
		C(40)-C(41)-C(42)	120.0(6)
		C(41)-C(40)-C(42)	122.2(6)
		C(40)-C(39)-C(38)	120.4(8)
		C(37)-C(38)-C(39)	120.4(6)
		C(34)-C(33)-C(32)	120.0(4)
		C(17)-C(16)-C(15)	120.1(5)
		C(16)-C(15)-C(14)	120.5(5)

Table S4: Anisotropic displacement parameters (A^2) for **2**. The anisotropic displacement factor exponent takes a form: $-2\ pi^2\ [h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12]$.

	U11	**U22**	**U33**	**U23**	**U13**	**U12**
Pd(1)	0.037(13)	0.0383(13)	0.0346(12)	0.0028 (9)	-0.00098(9)	0.0017(9)
P(1)	0.037(4)	0.0394(4)	0.0382(4)	0.0027(3)	-0.0018(3)	0.0009(3)
Cl	0.063(5)	0.0585(5)	0.0354(4)	0.0037(3)	0.0000(3)	0.0007(4)
P(2)	0.039(4)	0.0372(4)	0.0387(4)	0.0033(3)	0.0012(3)	0.0047(3)
C(1)	0.044(16)	0.0431(16)	0.0367(15)	0.0047(12)	-0.0035(12)	-0.0029(14)
C(25)	0.050(18)	0.0391(17)	0.0478(17)	0.0106(13)	-0.0091(14)	-0.0051(14)
C(7)	0.045(17)	0.0391(17)	0.0453(17)	-0.0002(13)	-0.0016(14)	-0.0068(14)
C(19)	0.040(17)	0.0447(18)	0.0419(16)	-0.0031(13)	-0.0009(12)	0.0067(14)
C(13)	0.040(17)	0.059(2)	0.0476(17)	0.0159(15)	-0.0049(14)	-0.0048(15)
C(32)	0.064(2)	0.051(2)	0.055(2)	0.0065(16)	-0.0089(17)	-0.0066(18)
C(24)	0.051(2)	0.054(2)	0.056(2)	-0.0032(16)	-0.0064(15)	0.0060(17)
C(31)	0.047(18)	0.0463(18)	0.0406(16)	0.0001(13)	-0.0007(13)	-0.0028(14)
C(30)	0.065(2)	0.068(3)	0.049(2)	0.0075(17)	0.0009(18)	-0.003(2)
C(37)	0.055(2)	0.054(2)	0.064(2)	0.0201(17)	0.0194(16)	0.0209(17)
C(8)	0.050(2)	0.062(2)	0.057(2)	-0.0032(17)	-0.0060(17)	0.0022(18)
C(26)	0.058(2)	0.067(3)	0.076(3)	0.022(2)	-0.008(2)	0.0043(19)
C(2)	0.059(2)	0.063(2)	0.052(2)	0.0007(16)	-0.0100(17)	0.0031(19)
C(12)	0.055(2)	0.062(2)	0.056(2)	-0.0103(17)	-0.0040(17)	0.0051(18)
C(20)	0.061(2)	0.055(2)	0.068(2)	0.0139(17)	0.0006(19)	0.0095(19)
C(10)	0.095(3)	0.077(3)	0.049(2)	-0.015(2)	-0.002(2)	-0.016(2)
C(28)	0.110(4)	0.113(4)	0.069(3)	0.045(3)	-0.027(3)	-0.032(3)
C(35)	0.083(3)	0.102(4)	0.080(3)	0.037(3)	-0.040(2)	-0.030(3)
C(9)	0.065(3)	0.076(3)	0.058(2)	-0.0031(19)	-0.018(2)	-0.011(2)
C(6)	0.057(2)	0.065(2)	0.0495(19)	0.0040(16)	0.0082(17)	0.0014(19)

C(36)	0.060(2)	0.064(2)	0.069(2)	0.0208(19)	-0.0187(18)	-0.012(19)
C(29)	0.087(3)	0.101(4)	0.052(2)	0.016(2)	-0.004(2)	-0.023(3)
C(22)	0.071(3)	0.090(3)	0.059(2)	-0.007(2)	-0.014(2)	0.040(3)
C(14)	0.062(2)	0.068(3)	0.080(3)	0.013(2)	-0.003(2)	-0.012(2)
C(23)	0.047(2)	0.079(3)	0.071(2)	-0.019(2)	-0.0163(19)	0.018(2)
C(21)	0.096(4)	0.073(3)	0.071(3)	0.022(2)	0.004(2)	0.034(3)
C(34)	0.088(3)	0.110(4)	0.075(3)	0.025(3)	-0.033(2)	-0.051(3)
C(11)	0.080(3)	0.073(3)	0.069(3)	-0.020(2)	0.006(2)	0.006(2)
C(17)	0.053(3)	0.144(5)	0.074(3)	0.001(3)	0.013(2)	-0.003(3)
C(42)	0.058(3)	0.097(4)	0.110(4)	0.020(3)	0.022(3)	0.021(3)
C(18)	0.047(2)	0.087(3)	0.061(2)	0.004(2)	0.0048(17)	-0.001(2)
C(27)	0.086(4)	0.092(4)	0.108(4)	0.043(3)	-0.032(3)	0.011(3)
C(5)	0.089(3)	0.104(4)	0.051(2)	-0.003(2)	0.022(2)	0.007(3)
C(4)	0.132(5)	0.106(4)	0.039(2)	0.003(2)	-0.007(3)	-0.002(3)
C(3)	0.095(3)	0.091(3)	0.053(2)	0.008(2)	-0.027(2)	0.005(3)
C(41)	0.080(4)	0.115(5)	0.141(6)	0.049(4)	0.031(4)	0.029(4)
C(40)	0.171(8)	0.108(5)	0.142(6)	0.063(5)	0.103(6)	0.086(6)
C(39)	0.183(7)	0.100(5)	0.136(5)	-0.002(4)	0.100(5)	0.043(5)
C(38)	0.128(4)	0.062(3)	0.082(3)	-0.011(2)	0.045(3)	0.010(3)
C(33)	0.086(3)	0.072(3)	0.064(2)	0.012(2)	-0.021(2)	-0.033(2)
C(16)	0.053(3)	0.163(6)	0.075(3)	0.036(3)	0.003(2)	-0.015(3)

Table S5: Hydrogen coordinates and isotropic displacement parameters (A^2) for **2**.

	x	y	z	U (eq)
H(38)	0.0781	0.1505	0.0594	0.109
H(24)	-0.098(3)	0.4069(15)	0.1183(13)	0.064(11)
H(36)	-0.103(2)	0.2815(13)	0.0738(11)	0.046(8)
H(8)	0.275(2)	0.4346(13)	0.2252(11)	0.042(9)
H(23)	-0.237(3)	0.4460(16)	0.0764(13)	0.072(13)
H(32)	-0.060(3)	0.1358(15)	0.1491(11)	0.060(10)
H(9)	0.281(3)	0.4686(13)	0.3080(11)	0.053(9)
H(18)	0.294(2)	0.3760(12)	0.0733(11)	0.044(9)
H(30)	-0.053(3)	0.2257(16)	0.2336(14)	0.081(14)
H(33)	-0.220(3)	0.0975(15)	0.1086(13)	0.078(11)
H(20)	0.101(2)	0.5292(14)	0.0758(12)	0.057(10)
H(42)	0.303(2)	0.2534(12)	0.1217(11)	0.038(9)
H(35)	-0.257(4)	0.247(2)	0.0379(17)	0.121(17)
H(6)	-0.052(3)	0.3659(14)	0.2354(12)	0.060(10)
H(12)	-0.014(3)	0.5075(14)	0.1979(12)	0.063(10)
H(28)	0.101(3)	0.1195(19)	0.3301(18)	0.112(15)
H(21)	-0.048(4)	0.5647(18)	0.0342(16)	0.093(16)
H(2)	0.266(3)	0.2961(14)	0.2285(12)	0.070(10)
H(15)	0.435(4)	0.5470(18)	0.0996(15)	0.088(15)
H(26)	0.223(3)	0.1536(13)	0.1841(12)	0.058(10)
H(22)	-0.225(3)	0.5304(16)	0.0291(14)	0.093(12)
H(4)	0.107(4)	0.325(2)	0.368(2)	0.13(2)
H(17)	0.458(4)	0.3954(19)	0.0355(16)	0.113(17)
H(3)	0.265(3)	0.2986(16)	0.3148(15)	0.083(13)
H(14)	0.277(3)	0.5263(14)	0.1391(11)	0.055(10)

H(10)	0.136(3)	0.5178(17)	0.3339(16)	0.089(14)
H(34)	-0.315(4)	0.1518(17)	0.0539(16)	0.101(14)
H(27)	0.232(4)	0.1129(18)	0.2610(16)	0.087(15)
H(11)	-0.020(4)	0.5434(17)	0.2820(15)	0.102(14)
H(5)	-0.042(3)	0.3579(17)	0.3178(14)	0.076(13)
H(16)	0.538(4)	0.4811(18)	0.0444(16)	0.111(15)
H(41)	0.414(3)	0.2266(15)	0.0857(13)	0.053(12)
H(29)	-0.043(3)	0.1805(17)	0.3107(16)	0.088(14)
H(40)	0.383(4)	0.145(2)	0.008(2)	0.14(2)
H(39)	0.224(7)	0.106(4)	0.006(3)	0.26(4)