Supporting Information for

Nickel–Catalyzed Cross–Coupling Reaction of Niobium(III)–Alkyne Complexes with Aryl Iodides

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X-ray crystallographic analysis of (*E*)-**4j**:

Single crystals suitable for X-ray diffraction study were obtained by slow evaporation of diethyl ether solution. The data were collected on a Rigaku Saturn70 CCD diffractometer using graphite-monochromated Mo K α radiation ($\lambda = 0.71070$ Å) at -160 °C. An empirical absorption correction was applied. The structure was solved by direct methods using the program SHELX-97¹ and expanded using Fourier techniques. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. A Sheldrick weighing scheme was used. All calculations were preformed using the CrystalStructure crystallographic package (ver 3.7).² Details of the crystal data are given in Table S1.

Figure S1. Ortep drawing of (*E*)-**4j** with thermal ellipsoids at the 50% probability level.

Table S1. Crystal and Structure Refinement Data of (E)-4j.

Chemical formula	C ₂₂ H ₂₀	D_c (g/cm ⁻¹)	1.179
Formula weight	284.40	F_{000}	608
Temperature (°C)	-160	μ (g/cm ⁻¹)	0.66
Wavelength (Å)	0.7107	Crystal Size (mm)	0.40, 0.40, 0.20
Crystal System	monoclinic	No. of reflections collected	3676
Space group	$P2_1/c$ (#14)	variable	279
a (Å)	8.674(9)	Refinement on	F^2
b (Å)	9.156(9)	R_{int}	0.039
c (Å)	20.18(3)	R^a (all data)	0.048
β (°)	90.30(4)	wR_2^b (all data)	0.127
U (Å ³)	1602.6(35)	GOF	0.950
Z	4	Min/max residual electron densities (e-/ Å ³)	0.28, -0.24

$$^a R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|, \quad ^b wR_2 = [\Sigma (w(F_o^2 - F_c^2))^2 / \Sigma w(F_o^2)^2]^{1/2}.$$

References

- (1) Sheldrick, G. M. *SHELX97*, 1997.
- (2) (a) Crystal Structure Analysis Package, Rigaku and Rigaku/MSD, 9009 New Trails Dr., The Woodlands TX 77381 USA, 2000-2004. (b) Watkin, D. J.; Prout, C. K.; Carruthers, J. R.; Betteridge, P. W. Chemical Crystallography Laboratory, Oxford, U.K., 1996.