Unusual Regioselectivity of Pd(0)-Catalyzed Coupling Reaction of Electron-deficient Alkenyl Halides with Allenic/Propargylic Zinc Reagents. Highly Selective Synthesis of 6-Phenylhex-5-yn-2(or 3)-enoates/enitrile and 4-Phenyl-6-substitutedhexa-2,4,5-trienoates

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Supplemental Materials

X-Ray single crystal analysis of E-5b.

X-ray data were collected using a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα radiation and a 12kw rotating anode generator. At a temperature of 20±1°C using the o-20 scan technique to a maximum 20 value of 51.0°. The structure was solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full matrix least-squares refinement was based on 1355 observed reflections (I > 2.00σ(I)) and 218 variable parameters and converged (largest parameter was 0.01 times its esd) with unweighed and weighed agreement factors of: R = [Σ(|Fo| - |Fc|)/Σ|Fo|], Rw = [Σw(|Fo| - |Fc|)^2/ΣwFo^2]^{1/2}. All calculations were performed using the teXsan crystallographic software package of Molecular Structure Corporation.

Crystal data of E-5b: C_{19}H_{15}O_{4}N, M = 321.33, yellow prismatic, space group P2_1/a(#4), a = 9.113(2), b = 17.467(2), c = 10.186(2) Å, β = 92.36(1)°, V = 1623.6(4) Å^3, Z = 4, D_{calc} = 1.315 g/cm^3, F_{000} = 672.00. μ(MoKα) = 0.93 cm^{-1}, Radiation: MoKα (λ = 0.71069 Å). Crystal dimensions = 0.20 x 0.20 x 0.30 mm, R = 0.047, R_w = 0.048, (Δρ)_{max} = 0.16 and (Δρ)_{min} = -0.17 e/Å^3.
Figure 1. ORTEP Presentation of \textit{E}-5b