WCl(η^2-C_2Ph_2)(η^6-C_6Ph_6H): A Compound Containing a Metallacycloheptatriene Unit from Trimerization of Diphenylacetylene with W(NMe_3)(η^2-C_2Ph_2)$_3$

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*Summary: W(NCMe)(η^2-C_2Ph_2)$_3$ (**1**) has been previously synthesized by the reaction of W(CO)(η^2-C_2Ph_2)$_3$ with Me_3NO in acetonitrile, while the same treatment in THF leads to the trimethylamine complex W(NMe_3)(η^2-C_2Ph_2)$_3$ (**2**). Compound **2** is reactive. Stirring a mixture of **2** and diphenylacetylene in dichloromethane at room temperature affords WCl(η^2-C_2Ph_2)(η^6-C_6Ph_6H) (**3**), which apparently arises from trimerization of the alkyne ligands concomitant with solvent activation. The structures of **1**−**3** have been determined by an X-ray diffraction study. A carbene W═C double bond is evidenced in **3**.*

Introduction

One of the most fascinating aspects pertaining to organometallic chemists concerns the cyclooligomerization of alkynes.[1] Mechanistic steps involving the formation of metallacycles, and their interconversions and subsequent decay, are implicated in a number of important catalytic transformations. For example, metal-promoted cyclization of alkynes to give cyclobutadienes, arenes, and cyclooctatetraenes has frequently been shown to proceed via metallacyclic intermediates.[2]

Polyalkyne complexes of the formula M(L)(η^2-RC≡CR′)$_3$, first prepared by Tate and co-workers,[3] have attracted much attention owing to their unique modes of bonding[4] and the potential to promote alkyne coupling and polymerization.[5] The parent compound W(CO)(C_2Ph_2)$_3$ is robust[6] and only reacts with molten diphenylacetylene to produce complexes containing cyclobutadiene and cyclopentadienyl ligands.[7] However, by replacing CO with NCMe, W(NCMe)(C_2Ph_2)$_3$ (**1**)[8] is able to undergo multiple alkyne−alkyne coupling reactions under mild conditions to yield the metallacyclononapentaene complex W(η^2-C_2Ph_2)(η^8-C_8Ph_8) (**4**)[9] and the tungstenocene compound W(η^5-C_5Ph_5)$_2$ (**5**).[10] In this paper we wish to report the synthesis and reactivity of W(NMe_3)(η^2-C_2Ph_2)$_3$ (**2**), which contains a more labile trimethylamine ligand.

Results and Discussion

Previously, the reaction of W(CO)(η^2-C_2Ph_2)$_3$ with Me_3NO in an acetonitrile solvent was shown to produce **1**,[8] whereas the same treatment in THF affords the trimethylamine complex **2** in 41% yield. In dichloromethane, the product mixture is complex and only a low yield of **2** is obtained. Me_3NO is a nucleophile and attacks the electropositive carbon atom of the carbonyl to produce Me_3N and CO_2.[11] The Me_3N weakly coordinates to the vacant coordination site and is readily displaced by a stronger electron-donating ligand, such as nitrile, isonitrile, and phosphine. Although amines are classical ligands in coordination chemistry, there are not often used with organotransition metal complexes.[12] An analogous primary amine complex W(NH_2Et)(η^2-C_2Ph_2)$_3$ was obtained from reduction of the NCMe ligand on **1**,[13] while treating **1** with NH_2Et or NMe_3 does not lead to the corresponding amine complex.

Compound **2** forms an air-stable, white solid but decomposes slowly in solution. It displays a higher lability than **1**, such that, reaction of **1** and diphenylacetylene requires a gentle heating,[9] while the same

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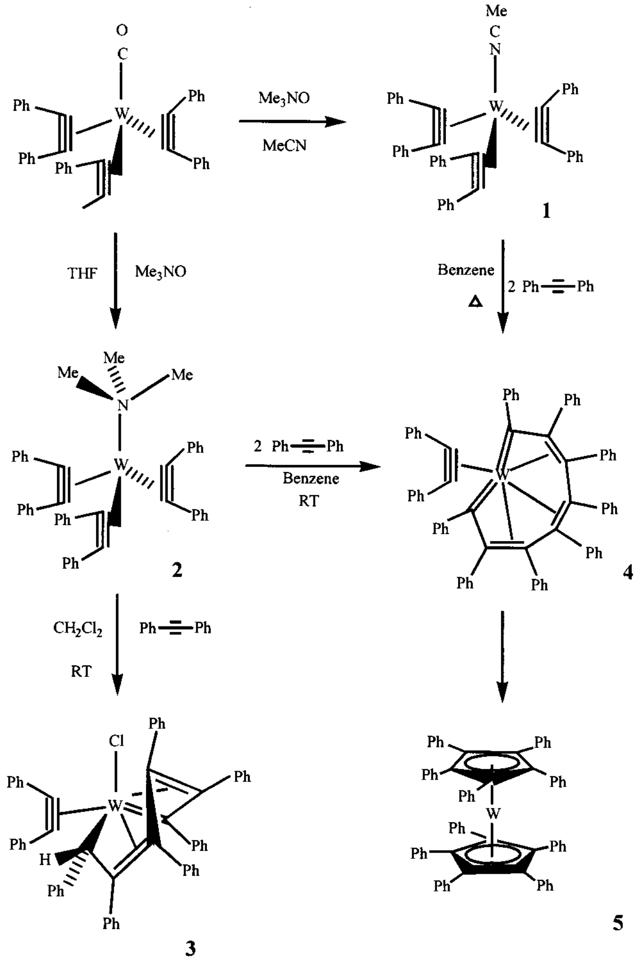
Scheme 1



reaction with **2** takes place at room temperature, and the products are dependent on the solvents used. In benzene solution compounds **4** and **5** are obtained predominantly. On the contrary, the reaction in dichloromethane (or 1,2-dichloroethane) leads to the metallacycloheptatriene complex $WCl(\eta^2\text{-}C_2Ph_2)(\eta^6\text{-}C_6Ph_6H)$ (**3**), which apparently arises from trimerization of diphenylacetylene concomitant with activation of the solvent molecule (Scheme 1). A radical pathway is probably involved, but we do not have mechanistic information. This $\eta^6\text{-}C_6R_6H$ type of bonding is rare in the literature,[14] although the $\eta^4\text{-}C_4R_4H$ ligands are relatively common.[15] On the other hand, both **1** and **2** react with diphenylacetylene to produce **4** and **5** in refluxing benzene or 1,2-dichloroethane solvent, suggesting that **4** and **5** are the kinetic products at elevated temperatures.

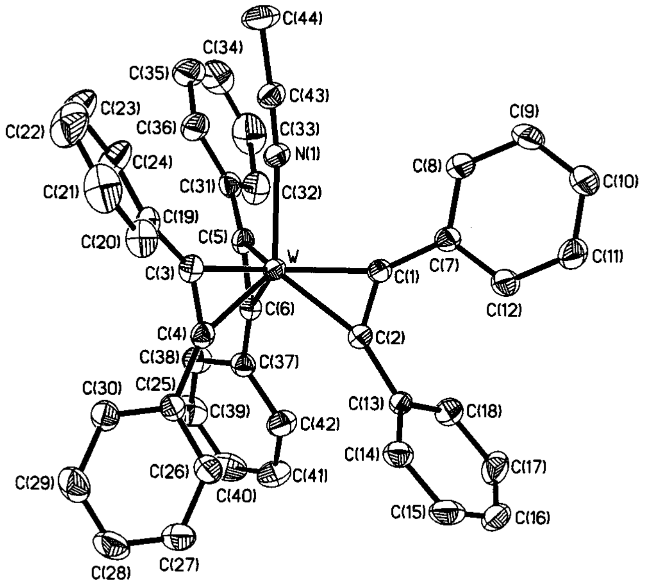


Figure 1. Molecular structure of **1**. Selected bond distances (Å): W−N(1) = 2.174(3), C(43)−N(1) = 1.124(4), C(43)−C(44) = 1.462(5), W−C(1) = 2.065(3), W−C(2) = 2.074(3), W−C(3) = 2.049(3), W−C(4) = 2.065(4), W−C(5) = 2.065(3), W−C(6) = 2.071(4), C(1)−C(2) = 1.326(5), C(3)−C(4) = 1.312(5), C(5)−C(6) = 1.313(5). Selected bond angles (deg): W−N(1)−C(43) = 173.1(3), N(1)−C(43)−C(44) = 177.0(4), N(1)−W−C(1) = 86.7(1), N(1)−W−C(2) = 124.1(1), N(1)−W−C(3) = 83.8(1), N(1)−W−C(4) = 121.0(1), N(1)−W−C(5) = 84.5(1), N(1)−W−C(6) = 121.4(1), W−C(1)−C(2) = 71.7(2), W−C(2)−C(1) = 70.9(2), W−C(3)−C(4) = 72.1(2), W−C(4)−C(3) = 70.8(2), W−C(5)−C(6) = 71.7(2), W−C(6)−C(5) = 71.3(2), C(1)−W−C(3) = 119.9(1), C(1)−W−C(4) = 116.5(1), C(1)−W−C(5) = 124.6(1), C(1)−W−C(6) = 119.0(1), C(2)−W−C(3) = 118.5(1), C(2)−W−C(4) = 93.2(1), C(2)−W−C(5) = 122.1(1), C(2)−W−C(6) = 95.0(1), C(3)−W−C(5) = 113.3(1), C(3)−W−C(6) = 116.3(1), C(4)−W−C(5) = 114.8(1), C(4)−W−C(6) = 94.7(1).

The new compounds **2** and **3** have been characterized by mass and ^1H and ^{13}C NMR spectroscopy. To evaluate the electronic and steric effects of N≡CMe and NMe_3 ligands on the coordination configuration around the W atom, single crystals of **1** and **2** were studied by X-ray diffraction. The molecular structures of **1** and **2**, shown in Figures 1 and 2, are closely resemblant. If taking the centers of the alkynes, the coordination about each tungsten atom can be described as a distorted tetrahedron. The three acetylene groups are essentially eclipsed but inclined with respect to the W−N(1) bond by an average tilt angle of 13.5° for **1** and 16.2° for **2**, which are comparable with those measured for $W(CO)(\eta^2\text{-}C_2Ph_2)_3$ (av 13.4°),[4b] $W(PMe_3)(\eta^2\text{-}C_2Ph_2)_3$ (av 13.2°),[16] $W(NH{=}CMe_2)(\eta^2\text{-}C_2Ph_2)_3$ (av 13.1°),[13] and $[W(SnPh_3)(\eta^2\text{-}C_2Ph_2)_3]^-$ (av 15.3°).[4c] The coordinated NCMe (W−N(1) = 2.174(3) Å) and NMe_3 (W−N(1) = 2.329(2) Å) ligands give rise to slightly different W−C distances, such that in **1** the upper W−C lengths are ca. 0.01 Å shorter than the lower W−C lengths, while in **2** the order is reversed, with the upper W−C lengths being slightly longer. The acetylene C≡C lengths are about equal for **1** (1.32 ± 0.01 Å) and **2** (1.316 ± 0.006 Å), and the phenyl groups are bent back from the C≡C axes with the angles ranging from 133.2(3)° to 142.9(4)°. It is noteworthy that the space-congested NMe_3 group is sterically forcing the upper phenyl rings to lie proximately perpendicular to the W−C_2(alkyne) planes, whereas they are arranged in a paddlewheel feature in **1**.

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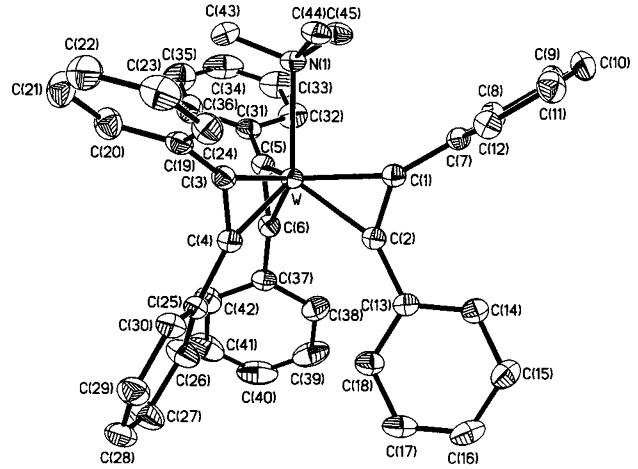

Figure 2. Molecular structure of **2**. Selected bond distances (Å): W−N(1) = 2.329(2), C(43)−N(1) = 1.480(4), C(44)−N(1) = 1.489(4), C(45)−N(1) = 1.496(4), W−C(1) = 2.069(3), W−C(2) = 2.054(3), W−C(3) = 2.066(3), W−C(4) = 2.059(3), W−C(5) = 2.070(3), W−C(6) = 2.057(3), C(1)−C(2) = 1.314(4), C(3)−C(4) = 1.318(4), C(5)−C(6) = 1.314(4). Selected bond angles (deg): N(1)−W−C(1) = 87.2(1), N(1)−W−C(2) = 124.2(1), N(1)−W−C(3) = 86.4(1), N(1)−W−C(4) = 123.3(1), N(1)−W−C(5) = 86.6(1), N(1)−W−C(6) = 123.7(1), W−C(1)−C(2) = 70.8(2), W−C(2)−C(1) = 72.0(2), W−C(3)−C(4) = 71.1(2), W−C(4)−C(3) = 71.7(2), W−C(5)−C(6) = 70.9(2), W−C(6)−C(5) = 72.0(2), C(1)−W−C(3) = 117.6(1), C(1)−W−C(4) = 118.7(1), C(1)−W−C(5) = 118.3(1), C(1)−W−C(6) = 114.2(1), C(2)−W−C(3) = 112.5(1), C(2)−W−C(4) = 92.0(1), C(2)−W−C(5) = 117.8(1), C(2)−W−C(6) = 92.0(1), C(3)−W−C(5) = 123.1(1), C(3)−W−C(6) = 120.6(1), C(4)−W−C(5) = 115.6(1), C(4)−W−C(6) = 92.0(1).

The molecular structure of **3** is illustrated in Figure 3. The diphenylacetylene ligand is eclipsed but tilted (15.8°) with respect to the W−Cl bond. The W(η^6-C_6Ph_6H) moiety forms a metallacycle, which appears to have a tungsten−carbon double bond to C(8) (2.025(7) Å), a single bond to C(3) (2.220(6) Å), and π-donation from the diene unit (C(4)−C(7)) to the tungsten atom. The W−C distances to the diene linkage are variable, such that W−C(7) = 2.504(6) Å and W−C(6) = 2.408(6) Å are significantly longer than W−C(5) = 2.309(6) Å and W−C(4) = 2.346(6) Å. The bond lengths and angles around the η^6-C_6Ph_6H skeleton are illustrated in Figure 4. The C(3), C(4), C(27), C(5), C(6), and C(33) atoms and the C(39), C(6), C(7), and C(45) atoms are about coplanar. The atoms W, C(8), C(51), and C(7) are also on the same plane, and the angle C(7)−C(8)−C(51) is 128.6(6)°, implying an alkylidene carbon for the C(8) atom. The hydrogen atom, giving a proton resonance at δ 3.96, is believed to bind the terminal carbon C(3) on the basis of the W−C(3)−C(21), W−C(8)−C(4), and C(4)−C(3)-C(51) bond angles, which sum up to 309.5°.

Formation of a W═C double bond in **3** is further evidenced by the $^{13}C\{^1H\}$ NMR spectrum, which shows a low-field signal at δ 261.3 characteristic of an alkylidene type carbon.[17] A high-field resonance at δ 73.4 is attributed to the *C*HPh carbon. The acetylene carbons give a sharp singlet at δ 218.8, suggesting a facile rotation for the W−alkyne bonding.

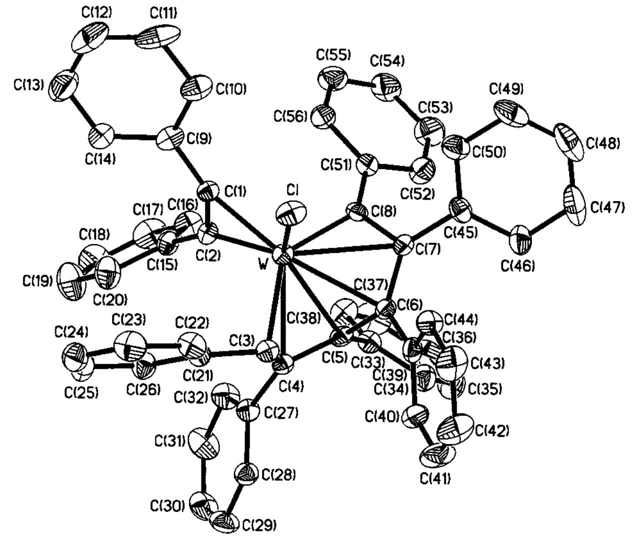

Figure 3. Molecular structure of **3**. Selected bond distances (Å): W−Cl= 2.360(2), W−C(1) = 2.058(6), W−C(2) = 2.065(6), W−C(3) = 2.200(6), W−C(4) = 2.346(6), W−C(5) = 2.309(6), W−C(6) = 2.408(6), W−C(7) = 2.504(6), W−C(8) = 2.025(7), C(1)−C(2) = 1.308(9). Selected bond angles (deg): Cl−W−C(1) = 87.6(2), Cl−W−C(2) = 123.3(2), Cl−W−C(3) = 88.0(2), Cl−W−C(4) = 124.1(2), Cl−W−C(5) = 128.2(2), Cl−W−C(6) = 94.3(2), Cl−W−C(7) = 88.7(2), Cl−W−C(8) = 104.1(2), W−C(1)−C(2) = 71.8(4), W−C(2)−C(1) = 71.2(4), C(1)−W−C(3) = 121.8(2), C(2)−W−C(3) = 109.2(3), C(1)−W−C(8) = 102.7(3), C(2)−W−C(8) = 100.3(3).

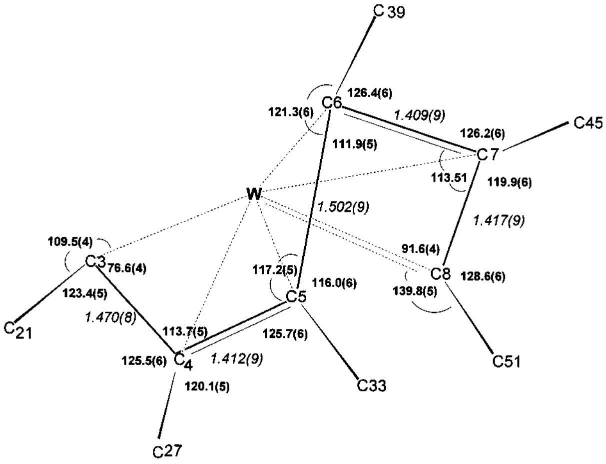

Figure 4. Bond distances (Å) and bond angles (deg) around the η^6-C_6Ph_6H skeleton of **3**.

In summary, the results presented here describe synthesis of a labile NMe_3 complex **2**, which undergoes an unusual alkyne coupling reaction in chlorinated solvents to yield **3** containing a *trans* (or *E*)-η^6-C_6Ph_6H ligand. Currently we are engaged in the reactions of **2** and acids in attempting to remove the basic NMe_3 ligand to produce binary complexes of the type $[W_x(C_2Ph_2)_y]$.

Experimental Section

General Comments. All manipulations were carried out under an atmosphere of purified dinitrogen with standard Schlenk techniques. Solvents were dried over appropriate reagents under dinitrogen.[18] W(CO)(η^2-$C_2Ph_2)_3$[3] and W(NCMe)(η^2-$C_2Ph_2)_3$ (**1**)[8] were prepared as described in the literature.

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[17] Mann, B. E.; Taylor, B. F. *^{13}C NMR Data for Organometallic Compounds*; Academic Press: New York, 1981.

Anhydrous Me_3NO was obtained from $Me_3NO{\cdot}2H_2O$ (Aldrich) by sublimation under vacuum twice. Preparative thin-layer chromatographic (TLC) plates were prepared from silica gel (Kieselgel, DGF_{254}). Infrared spectra were recorded on a Hitachi I-2001 IR spectrometer. ^1H and ^{13}C NMR spectra were obtained on a Varian VXR-300 spectrometer. Fast-atom-bombardment (FAB) mass spectra were recorded by using a VG Blotch-5022 mass spectrometer. Elemental analyses were performed at the National Chen-Kung University, Tainan, Taiwan.

Preparation of 2. $W(CO)(\eta^2\text{-}C_2Ph_2)_3$ (745 mg, 1.0 mmol) and Me_3NO (150 mg, 1.95 mmol) were placed in an oven-dried 100 mL Schlenk flask, equipped with a magnetic stir bar and a rubber serum stopper. Freshly distilled THF (20 mL) was introduced into the flask via a syringe through the serum stopper. The mixture was stirred at room temperature for 4 h, resulting in a pale yellow solution plus a gray solid precipitate. The mixture was filtered, and the filtrate was concentrated to ca. 5 mL on a rotary evaporator. Methanol (15 mL) was added, and the solution was cooled to −10 °C in a freezer. A colorless crystalline solid was afforded, characterized as $W(NMe_3)(\eta^2\text{-}C_2Ph_2)_3$ (**2**; 318 mg, 41%). MS(FAB): *m*/*z* 777 (M^+, ^{184}W), 718 ($M^+ - NMe_3$). ^1H NMR ($CDCl_3$, 20 °C): δ 7.34−6.84 (m, Ph), 3.15 (s, NMe_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 20 °C): δ 204.2, 188.6 ($\equiv$C), 146.6, 143.2 (*ipso*-C_6H_5), 129.5, 128.1, 127.4, 127.1, 125.6, 124.8 (*o*, *m*, *p*-C_6H_5), 57.0 ($N(CH_3)_3$). Anal. Calcd for $C_{45}H_{39}NW$: C, 69.50; H, 5.06; N, 1.80. Found: C, 69.31; H, 5.00; N, 1.68.

Preparation of 3. Compound **2** (150 mg, 0.19 mmol) and $PhC{\equiv}CPh$ (42 mg, 0.23 mmol) were placed in a 50 mL Schlenk flask. Dichloromethane (10 mL) was added into the flask, and the resulting mixture was stirred at ambient temperature for 48 h and then filtered. The filtrate was concentrated to ca. 2 mL and subjected to TLC, with *n*-hexane/dichloromethane (3:1, v/v) as eluant. The material forming the major orange-yellow band was isolated and crystallized from *n*-hexane/dichloromethane to afford air-stable, orange-red crystals of $WCl(\eta^2\text{-}C_2Ph_2)(\eta^6\text{-}C_6Ph_6H)$ (**3**; 29 mg, 16%). MS(FAB): *m*/*z* 932 (M^+, ^{184}W and ^{35}Cl), 897 ($M^+ - Cl$), 719 ($M^+ - Cl - C_2Ph_2$). ^1H NMR ($CDCl_3$, 20 °C): δ 7.48−6.31 (m, Ph), 3.96 (s, CHPh). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 20 °C): δ 261.3 (W=C), 218.8 (C≡C), 141.0−118.8 ($C-C_6H_5$), 73.4 (W−CH). Anal. Calcd for $C_{56}H_{41}ClW$: C, 72.07; H, 4.43. Found: C, 72.32; H, 4.53.

X-ray Structure Determination. A crystal of **1** (ca. 0.60 × 0.14 × 0.09 mm^3), **2** (ca. 0.40 × 0.22 × 0.18 mm^3), and **3** (ca. 0.40 × 0.30 × 0.20 mm^3) were each mounted in a thin-walled glass capillary and aligned on the Bruker SMART-CCD (for **1** and **2**) and Nonius CAD4 (for **3**) diffractometers, with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). The data were collected at 295(2) K. All data were corrected for the effects of absorption. The structures were solved by the direct method and refined by full-matrix least-squares on F^2. The program used was the SHELXTL package.[19] All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were included but not refined. A summary of relevant crystallographic data is provided in Table 1.

Table 1. Crystallographic Data for $W(NCM)(\eta^2\text{-}C_2Ph_2)_3$ (1), $W(NMe_3)(\eta^2\text{-}C_2Ph_2)_3$ (2), and $WCl(\eta^2\text{-}C_2Ph_2)(\eta^6\text{-}C_6Ph_6H)$ (3)

	1	**2**	**3**
chem formula	$C_{44}H_{33}NW$	$C_{45}H_{39}NW$	$C_{56}H_{41}ClW$
cryst syst	monoclinic	monoclinic	monoclinic
space group	$P2_1/c$	$P2_1/c$	Cc
fw	759.56	777.62	934.20
a, Å	17.2850(2)	16.3984(2)	13.286(3)
b, Å	9.9082(1)	15.5170(3)	15.150(3)
c, Å	19.9590(2)	16.6394(3)	21.303(4)
α, deg	90	90	90
β, deg	92.291(1)	118.933(1)	98.60(3)
γ, deg	90	90	90
V, Å^3	3415.51(6)	3705.5(1)	4240(2)
Z	4	4	4
D_{calc}, g cm^{-1}	1.477	1.394	1.464
μ, mm^{-1}	3.413	3.148	2.862
R_1/wR_2[a] ($I > 2\sigma(I)$)	0.0347/0.0585	0.0313/0.0489	0.0313/0.0751
GOF on F^2	1.031	1.040	1.048

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$; $wR_2 = \{\sum[w(|F_o|^2 - |F_c|^2)^2]/\sum w|F_o|^4\}\}^{1/2}$.

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Supporting Information Available: Complete tables of crystallographic data, positional parameters, anisotropic thermal parameters, bond angles, bond distances, and torsional angles of **1**−**3**. This material is available free of charge via the Internet at http://pubs.acs.org.

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