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ORGANOMETALLICS
Note

Use of Aryliron Complexes [$CpFe(CO)_2Ar$] as Arylcarbonyl Cation Equivalents in the Reactions with Organolithium Reagents To Yield Ketones

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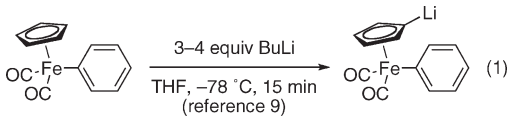
Summary: Treatment of aryliron complexes [*CpFe*(CO)$_2$*Ar*] *with organolithium reagents RLi in THF affords the corresponding ketones RCOAr. The reaction, wherein the aryliron complexes serve as arylcarbonyl cation equivalents, would begin with nucleophilic addition of RLi to one of the carbonyl ligands to form acyliron* [*CpFe*(*CO*)(*RCO*)*Ar*] *without lithiation of the Cp ring.*

Dicarbonylcyclopentadienylorganoiron complexes [$CpFe(CO)_2R$] represent an important class of organometallic compounds due to their rich coordination chemistry.[1] The corresponding aryliron complexes [$CpFe(CO)_2Ar$] are fundamental 18-electron arylmetals, and several reliable methods for the synthesis of the aryliron complexes have been reported.[2] On the other hand, the chemical reactivity of the aryliron complexes has not been well investigated.[3−8] Thanks to the ubiquity and abundance of iron as a transition metal, we have envisioned that [$CpFe(CO)_2Ar$] would serve as interesting arylating agents in organic synthesis and have been studying the reactivity and utility of [$CpFe(CO)_2Ar$].[2e,2f]

The reactions of [$CpFe(CO)_2Ar$] with organometallic reagents are rare. The reaction of [$CpFe(CO)_2Ph$] with excess butyllithium in THF at −78 °C was reported, leading to the lithiation of the cyclopentadienyl ring (eq 1).[9,10] To our surprise, a similar reaction at a higher reaction temperature led to a completely different outcome. Treatment of [$CpFe(CO)_2$(4-tolyl)] (**1a**) with 2 equiv of butyllithium at −20 °C afforded butyl 4-tolyl ketone (**2a**) in 59% yield (Table 1, entry 1). Iron complex **1a** thus behaved as an arylcarbonyl cation equivalent, the utility of which we describe here in detail.



Whereas bulky secondary and tertiary butyllithiums reacted with **1a** to afford the corresponding ketones in modest yields (entries 2 and 3), methylation proceeded smoothly (entry 4). The reaction of **1a** with phenyllithium furnished phenyl 4-tolyl ketone (**2e**) in excellent yield (entry 5). The use of a smaller amount, 1.2 equiv, of phenyllithium diminished the yield of **2e** (entry 6). Grignard reagents were ineffective for the formation of ketone, even at an elevated temperature (entry 7).

Other aryllithium reagents, prepared by bromine−lithium exchange, also engaged in the reaction (entries 8−14). Despite their steric hindrance, bulky 2-tolyllithium and 1-naphthyllithium converted **1a** smoothly to the corresponding ketones in high yields (entries 8 and 9). Although 4-methoxyphenyllithium reacted efficiently (entry 10), electron-deficient aryllithium reagents were less reactive (entries 11−13).

The reactions of various aryliron complexes with phenyllithium were investigated (Table 2). Sterically demanding arylirons **1d** and **1f** were available for use (entries 3 and 5), while the 1-naphthyl group of **1d** retarded the reaction. Halophenylirons **1g**−**1i** and electron-deficient aryliron **1j** underwent the reaction to afford the corresponding benzophenone derivatives in high yields (entries 6−9). The reaction of electron-rich 4-methoxyphenyliron **1k** was slow, requiring

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(5) Iron−carbon bond cleavage in strong carboxylic acids: De Luca, N.; Wojcicki, A. *J. Organomet. Chem.* **1980**, *193*, 359–378.

(6) Reaction with HgX_2 affording ArHgX: (a) Dizikes, L. J.; Wojcicki, A. *J. Am. Chem. Soc.* **1977**, *99*, 5295–5303. (b) Reference 2c.

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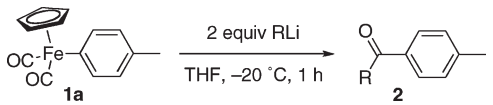
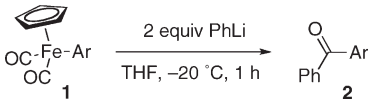
Table 1. Reactions of [CpFe(CO)$_2$(4-tolyl)] with Organolithium Reagents



entry	R	2	yield/%
1	Bu	**2a**	59
2	*s*-Bu	**2b**	37
3	*t*-Bu	**2c**	36
4	Me	**2d**	63
5	Ph	**2e**	91
6[a]	Ph	**2e**	68
7[b]	Ph	**2e**	4
8[c]	2-MeC$_6$H$_4$	**2f**	71
9[c]	1-naphthyl	**2g**	75
10[c]	4-MeOC$_6$H$_4$	**2h**	65
11[c]	4-FC$_6$H$_4$	**2i**	52
12[c,d]	4-CF$_3$C$_6$H$_4$	**2j**	47
13[c]	4-ClC$_6$H$_4$	**2k**	46
14[c]	4-PhC$_6$H$_4$	**2l**	79

[a] 1.2 equiv of PhLi. [b] Instead of PhLi, PhMgBr was used at 25 °C. [c] The aryllithium reagent was prepared from 2 equiv of the corresponding aryl bromide and 4 equiv of *tert*-butyllithium at −20 °C. [d] The aryllithium reagent was prepared at −78 °C and reacted with **1a** at −20 °C.

Table 2. Scope of Aryliron Complexes



entry	Ar	1	2	yield/%
1	4-PhC$_6$H$_4$	**1b**	**2m**	74
2	2-naphthyl	**1c**	**2n**	86
3[a]	1-naphthyl	**1d**	**2o**	65
4	4-*t*-BuC$_6$H$_4$	**1e**	**2p**	91
5	2-MeC$_6$H$_4$	**1f**	**2q**	76
6	4-ClC$_6$H$_4$	**1g**	**2r**	85
7	4-FC$_6$H$_4$	**1h**	**2s**	83
8	4-BrC$_6$H$_4$	**1i**	**2t**	68
9	3-CF$_3$C$_6$H$_4$	**1j**	**2u**	89
10[b]	4-MeOC$_6$H$_4$	**1k**	**2v**	59

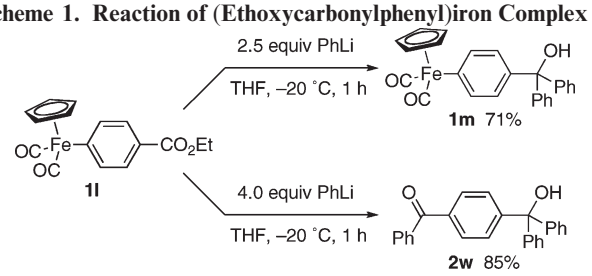
[a] Performed at 25 °C. [b] Performed at 0 °C.

a higher temperature to obtain **2v** in moderate yield (entry 10). The methoxy group lowered the electrophilicity of **1k**.

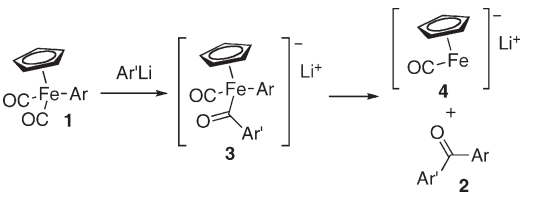
The reaction of iron complex **1l**, bearing an ester group, with 2.5 equiv of phenyllithium resulted in selective nucleophilic addition at the ester moiety, providing iron complex **1m** (Scheme 1). The reactivity of the carbonyl ligands proved to be lower than that of the ester carbonyl. With 4 equiv of phenyllithium, the aryliron moiety acted as an arylcarbonyl cation equivalent to yield ketone **2w**.

We had initially proposed a draft mechanism as shown in Scheme 2. The reaction would begin with nucleophilic addition of Ar′Li to the carbonyl group of [CpFe(CO)$_2$Ar] **1** to

Scheme 1. Reaction of (Ethoxycarbonylphenyl)iron Complex 1l



Scheme 2. Draft Mechanism



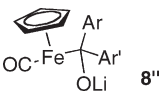
form acylferrate [CpFe(CO)(Ar′CO)Ar]Li (**3**).[11] Acylferrate **3** might undergo reductive elimination to yield ketone **2**. However, there are two issues to be addressed. While the mechanism in Scheme 2 should require only 1 equiv of PhLi in principle, the higher yield of **2e** was obtained when 2 equiv of PhLi was used (Table 1, entry 5 vs entry 6). It is also worth noting that no further nucleophilic attack of organolithium to ketone **2** was observed despite the use of 2 equiv of organolithium.

To gain more information about the reaction mechanism, we examined the following experiment. Treatment of **1a** with 2 equiv of PhLi in THF at −20 °C for 1 h was followed by an addition of 4-isopropylbenzaldehyde (**5**, 1.0 equiv) (Scheme 3). After quenching with water, the reaction afforded (4-isopropylphenyl)(phenyl)methanol (**6**) in 27% yield along with an 80% yield of **2e** and a 30% recovery of the aldehyde **5**. The formation of **6** and the absence of triarylmethanol **7** suggest that ketone **2e** is protected from the nucleophilic attack of organolithium during the reaction. We presume that ketone **2e** exists in the resonance of **8** and **8′** (Scheme 4).[12] The (η^2-ketone)iron complex would be formed via nucleophilic migration of the reactive Ar anion to the acyl carbonyl.[13] The η^2 complexation is not robust, and liberation of ketone **2** would take place upon addition of water.

The formation of **6** clarifies that 1.7 equiv of phenyllithium was consumed before the addition of **5** and suggests that 0.7 equiv of aryllithium might attack the second carbonyl ligand to yield diacylferrate **9** (Scheme 4). The Ar group of the higher order ferrate **9** would be more reactive than that of **3** and undergo smoother migration to yield a hybrid of **10** and **10′**. The subsequent protonolysis would afford **2**.[14]

(11) Nucleophilic attack of hydride to the carbonyl ligand of [CpFe(CO)$_2$R]: (a) Kubo, K.; Nakazawa, H.; Nakahara, S.; Yoshino, K.; Mizuta, T.; Miyoshi, K. *Organometallics* **2000**, *19*, 4932–4934. (b) Tobita, H.; Shiozawa, R.; Ogino, H. *Chem. Lett.* **1997**, *26*, 805–806. (c) Shiozawa, R.; Tobita, H.; Ogino, H. *Organometallics* **1998**, *17*, 3497–3504.

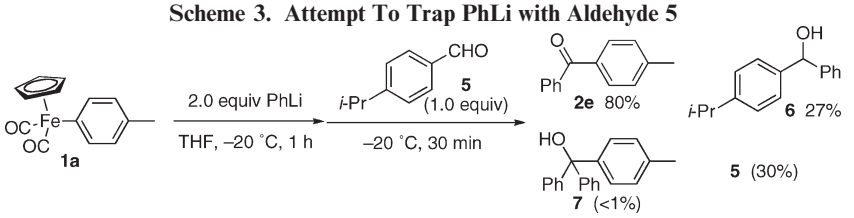
(12) Complexes having an open structure such as **8″** are also conceivable.



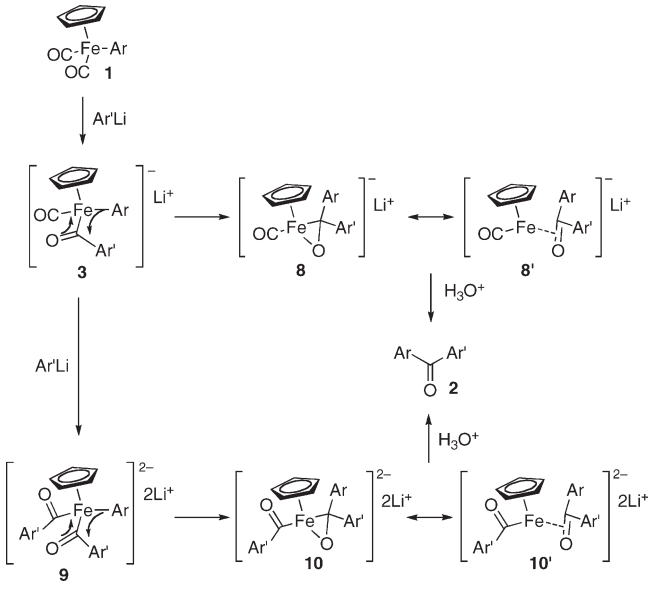
(13) The conventional reductive elimination process may provide the same complex.

(14) Although the protonolysis is likely to yield aldehyde Ar′CHO, none of the aldehyde was detected. Prior to the protonolysis of the (arylcarbonyl)iron moiety Ar′C(=O)Fe, decarbonylation could occur to yield Ar′Fe(CO), which then undergoes protonolysis.

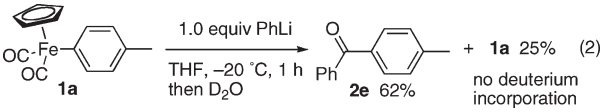
Scheme 3. Attempt To Trap PhLi with Aldehyde 5



Scheme 4. Detailed Plausible Reaction Mechanism



The following experiments verified that the lithiation of the Cp ring by the excess amount of RLi[9,10] did not occur under the present reaction conditions. Treatment of **1a** with only 1.0 equiv of PhLi was followed by quenching with D_2O (eq 2). The reaction afforded **2e** in 62% yield, and **1a** was recovered in 25% yield. The recovered **1a** had only the natural abundance of deuterium. The use of 1.0 equiv of BuLi at −20 °C also resulted in no deuterium incorporation (47% of **2a** and 37% of **1a**). Given that the lithiation of the Cp ring is irreversible, the lithiation would predominate kinetically at −78 °C and the nucleophilic attack would occur under kinetic control at −20 °C.



(2)

In summary, $[CpFe(CO)_2Ar]$ complexes have emerged as arylcarbonyl cation equivalents in the reaction with organolithium reagents. In contrast to the previous report on the reaction of $[CpFe(CO)_2Ar]$ with organolithium, no metalation of the Cp ring was observed. This finding represents a new chemical reactivity of $[CpFe(CO)_2Ar]$ toward organometallic reagents and will be applicable to organic synthesis.

Experimental Section

General Procedures. ^1H NMR (500 and 300 MHz) and ^{13}C NMR (125.7 and 75.3 MHz) spectra were taken on a Varian UNITY INOVA 500 spectrometer and a Varian Gemini 300 spectrometer. ^1H NMR and ^{13}C NMR spectra were obtained in $CDCl_3$ [using tetramethylsilane (for ^1H, δ = 0.00 ppm) and $CDCl_3$ (for ^{13}C, δ = 77.2 ppm) as an internal standard] or C_6D_6 [using C_6H_6 (for ^1H, δ = 7.15 ppm) and C_6D_6 (for ^{13}C, δ = 128.6 ppm) as an internal standard]. IR spectra were determined on a JASCO IR-810 spectrometer. Mass spectra (EI unless otherwise noted) were determined on a JEOL Mstation 700 spectrometer. Elemental analyses were carried out at the Elemental Analysis Center of Kyoto University.

Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. $[CpFe(CO)_2Ar]$ was synthesized by palladium-catalyzed transmetalation between $[CpFe(CO)_2I]$ and arylmetal reagents according to the methods reported previously.[2e,2f] THF was purchased from Kanto Chemical Co., stored under nitrogen, and used as is. Organolithium reagents were commercially available or prepared as described below. Silica gel (Wakogel 200 mesh) was used for column chromatography.

Typical Procedure for Reactions of $[CpFe(CO)_2Ar]$ with Organolithium Reagents (Tables 1, 2 and Scheme 1). Dicarbonylcyclopentadienyl(4-methylphenyl)iron (**1a**, 80 mg, 0.30 mmol) and THF (1.5 mL) were added in a 20 mL reaction flask under argon. The mixture was cooled to −20 °C, and then phenyllithium (1.1 M cyclohexane/Et_2O solution, 0.55 mL, 0.60 mmol) was added. After being stirred for 1 h at −20 °C, saturated ammonium chloride solution (0.13 mL) was added to the reaction mixture. The mixture was filtered through a pad of silica gel, and the filtrate was concentrated. Silica gel column purification (eluent: hexane/ethyl acetate, 40:1) of the crude product provided phenyl 4-tolyl ketone (**2e**, 53 mg, 0.27 mmol, 91% yield).

Typical Procedure for Reactions of $[CpFe(CO)_2(4\text{-tolyl})]$ with Aryllithium Reagents Generated in Situ by Br/Li Exchange Reactions (Table 1, entry 14 as a representative). 4-Bromobiphenyl (140 mg, 0.60 mmol) and THF (1.0 mL) were added in a 20 mL reaction flask under argon. The mixture was cooled to −20 °C, and then *tert*-butyllithium (1.6 M pentane solution, 0.75 mL, 1.2 mmol) was added. After being stirred for 30 min at −20 °C, dicarbonylcyclopentadienyl(4-methylphenyl)iron (**1a**, 80 mg, 0.30 mmol) in THF (1.0 mL) was added to the reaction mixture. After being stirred for 1 h at −20 °C, saturated ammonium chloride solution (0.26 mL) was added to the reaction mixture. The mixture was filtered through a pad of silica gel, and the filtrate was concentrated. Silica gel column purification (eluent: hexane/ethyl acetate, 40:1) of the crude product provided 4-biphenylyl phenyl ketone (**2l**, 65 mg, 0.24 mmol, 79% yield).

Characterization Data. The product **2p** showed spectra identical to that of the commercially available authentic sample. The spectral data of the products **1a**,[2e] **1b**,[2e] **1d**,[2f] **1f**−**1l**,[2e,2f] **2a**,[15] **2b**,[15] **2c**,[16] **2d**,[17] **2e**,[15] **2f**,[15] **2g**,[17] **2h**,[15] **2i**,[15] **2k**,[17] **2m**,[18] **2n**,[17] **2o**,[16] 2q,[17] **2r**,[16] **2s**,[17] **2t**,[17] and **2v**[16] can be found in the literature.

Dicarbonylcyclopentadienyl(2-naphthyl)iron (1c): IR (Nujol) 2342, 2021, 1998, 1970, 1944, 1574, 1456, 1419, 813, 745, 644, 628 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.90 (s, 5H), 7.28 (dd, J = 7.5,

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(18) Li, B.-J.; Li, Y.-Z.; Lu, X.-Y.; Liu, J.; Guan, B.-T.; Shi, Z.-J. *Angew. Chem., Int. Ed.* **2008**, *47*, 10124–10127.

7.0 Hz, 1H), 7.36 (dd, J = 8.0, 7.0 Hz, 1H), 7.44 (d, J = 8.5 Hz, 1H), 7.55−7.59 (m, 2H), 7.69 (d, J = 8.0 Hz, 1H), 7.93 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 86.14, 124.08, 125.51, 125.53, 125.94, 127.73, 131.09, 134.20, 143.07, 143.30, 143.90, 216.27. Anal. Found: C, 67.11; H, 3.94. Calcd for $C_{17}H_{12}FeO_2$: C, 67.14; H, 3.98. Mp: 94−95 °C.

Dicarbonylcyclopentadienyl (4-*tert*-butylphenyl)iron (1e): IR (Nujol) 2002, 1966, 1945, 1917, 1479, 1464, 1362, 1116, 1004, 809, 729, 632, 594 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.27 (s, 9H), 4.86 (s, 5H), 7.02 (d, J = 7.5 Hz, 2H), 7.35 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 31.44, 33.80, 85.70, 124.83, 139.89, 144.45, 145.60, 216.21. Anal. Found: C, 65.57; H, 5.75. Calcd for $C_{17}H_{18}FeO_2$: C, 65.83; H, 5.85. Mp: 106−107 °C.

Dicarbonylcyclopentadienyl (4-(hydroxydiphenylmethyl)phenyl)-iron (1m): IR (Nujol) 3548, 2022, 1970, 1957, 1448, 1157, 998, 818, 757, 700 cm^{-1}; ^{1}H NMR (C_6D_6) δ 2.41 (s, 1H), 4.01 (s, 5H), 7.03−7.06 (m, 2H), 7.09−7.11 (m, 6H), 7.42 (d, J = 8.5 Hz, 2H), 7.45 (d, J = 7.5 Hz, 4H); ^{13}C NMR (C_6D_6) δ 82.67, 86.27, 127.67, 128.57, 128.91, 129.14, 143.46, 144.97, 145.23, 148.85, 217.27. Anal. Found: C, 71.31; H, 4.52. Calcd for $C_{26}H_{20}FeO_3$: C, 71.58; H, 4.62. Mp: 116−117 °C.

4-Tolyl 4-trifluoromethylphenyl ketone (2j): IR (Nujol) 1647, 1605, 1407, 1329, 1171, 1136, 1110, 1067 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.46 (s, 3H), 7.30−7.32 (m, 2H), 7.71−7.73 (m, 2H), 7.75 (d, J = 7.5 Hz, 2H), 7.87 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 22.07, 124.04 (q, J_{C-F} = 271 Hz), 125.63 (q, J_{C-F} = 3.9 Hz), 129.58, 130.36, 130.69, 133.83 (q, J_{C-F} = 32.5 Hz), 134.40, 141.46, 144.44, 195.64. Anal. Found: C, 68.13; H, 4.35. Calcd for $C_{15}H_{11}F_3O$: C, 68.18; H, 4.20. Mp: 136−137 °C.

4-Biphenylyl 4-tolyl ketone (2l): IR (Nujol) 1644, 1604, 1275, 933, 855, 826, 776, 739, 691 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.46 (s, 3H), 7.30 (d, J = 8.0 Hz, 2H), 7.39−7.42 (m, 1H), 747−7.50 (m, 2H), 7.65 (d, J = 8.0 Hz, 2H), 7.70 (d, J = 8.0 Hz, 2H), 7.76 (d, J = 8.0 Hz, 2H), 7.88 (d, J = 9.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 22.01, 127.25, 127.64, 128.47, 129.30, 129.35, 130.60, 130.95, 135.37, 136.94, 140.40, 143.54, 145.33, 196.47. Anal. Found: C, 88.29; H, 5.94. Calcd for $C_{20}H_{16}O$: C, 88.20; H, 5.92. Mp: 122−124 °C.

Phenyl 3-trifluoromethylphenyl ketone (2u): IR (Nujol) 1654, 1337, 1266, 1170, 1118, 1073, 725, 715, 691, 660 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.50−7.54 (m, 2H), 7.62−7.65 (m, 2H), 7.79−7.81 (m, 2H), 7.85 (d, J = 7.5 Hz, 1H), 7.98 (d, J = 7.5 Hz, 1H), 8.07 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 123.91 (q, J_{C-F} = 271.1 Hz), 126.90 (q, J_{C-F} = 3.9 Hz), 128.78, 129.03 (q, J_{C-F} = 3.4 Hz), 129.16, 130.23, 131.22 (q, J_{C-F} = 32.3 Hz), 133.22, 133.32, 136.98, 138.51, 195.41. Anal. Found: C, 67.24; H, 3.37. Calcd for $C_{14}H_9F_3O$: C, 67.20; H, 3.63; HRMS (m/z) obsd 250.0603 (Δ = −1.0 ppm); calcd for $C_{14}H_9F_3O$ 250.0605. Mp: 48−49 °C.

4-(Hydroxydiphenylmethyl)phenyl phenyl ketone (2w): IR (neat) 3444, 1658, 1599, 1447, 1405, 1318, 1279, 764, 732, 701, 680 cm^{-1}; ^{1}H NMR (C_6D_6) δ 2.51 (s, 1H), 7.06−7.19 (m, 9H), 7.33 (d, J = 7.5 Hz, 4H), 7.38 (d, J = 9.0 Hz, 2H), 7.71−7.75 (m, 4H); ^{13}C NMR (C_6D_6) δ 82.55, 128.19, 128.53, 128.71, 128.93, 128.97, 130.51, 130.79, 132.64, 137.43, 138.83, 147.68, 152.12, 196.01. Anal. Found: C, 85.67; H, 5.66. Calcd for $C_{26}H_{20}O_2$: C, 85.69; H, 5.53.

(4-Isopropylphenyl)(phenyl)methanol (6): ^{1}H NMR ($CDCl_3$) δ 1.22 (d, J = 7.0 Hz, 6H), 2.29 (d, J = 8.0 Hz, 1H), 2.88 (sept, J = 7.0 Hz, 1H), 5.79 (d, J = 8.0 Hz, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.23−7.26 (m, 1H), 7.27 (d, J = 8.0 Hz, 2H), 7.30−7.33 (m, 2H), 7.37 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 24.17, 33.99, 76.33, 126.67, 126.77, 126.79, 127.64, 128.62, 141.47, 144.08, 148.47.

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