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(N-Heterocyclic Carbene)-Pd-Catalyzed Anaerobic Oxidation of Secondary Alcohols and Domino Oxidation−Arylation Reactions

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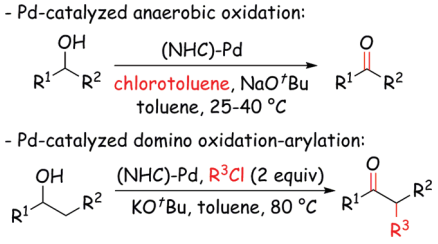
Received December 3, 2010



The use of commercially available (SIPr)Pd(cinnamyl)Cl (SIPr = 1,3-bis(2,6-diisopropylphenyl)-4,5-dihydroimidazol-2-ylidene) as a precatalyst for the anaerobic oxidation of secondary alcohols is described. The use of this complex allows for a drastic reduction in the reaction times and catalyst loading when compared to the unsaturated counterpart. This catalytic system is compatible with the use of microwave dielectric heating, decreasing even further catalyst loading and reaction times. Domino Pd-catalyzed oxidation−arylation reactions of secondary alcohols are also presented.

Introduction

The oxidation of alcohols to their corresponding carbonyl products stands out as a very important and useful organic transformation.[1] Aldehydes or ketones of industrial significance are mainly used as solvents, perfumes, and flavoring agents or as intermediates in the manufacture of resins, plastics, dyes, and pharmaceuticals.[2] The use of transition metal catalysts in combination with an oxidizing agent has emerged as a very interesting alternative to traditional methods that require stoichiometric amounts of toxic reagents and forcing conditions.[3] Among many others,[3] special attention has been paid to Pd-catalyzed systems in which molecular oxygen is used to oxidize the reduced catalyst intermediate during the catalytic cycle.[4] Although this is a very user-friendly, attractive, and economical option, it could be problematic in certain cases due to the hazard of oxygen pressures when running oxidations in flammable organic solvents, particularly when moving from laboratory settings to industrial scale.[4c,5,6]

Recently, our group reported on the use of aryl chlorides as oxidants for the selective oxidation of a variety of secondary alcohols under very mild reaction conditions using (NHC)-Pd[7] or (NHC)-Ni[7,8] systems (NHC = N-heterocyclic carbene).[9] The use of an inexpensive aryl chloride as oxidant results in the formation of the corresponding inert, dehalogenated aryl compound (for instance, benzene or toluene) as a byproduct that becomes part of the solvent. Our initial Pd system made use of commercially available

(1) (a) Hudlicky, M. *Oxidations in Organic Chemistry*, ACS Monograph Series; American Chemical Society: Washington, DC, 1990. (b) Sheldon, R. A.; Kochi, J. K. *Metal-Catalyzed Oxidations of Organic Compounds*; Academic Press: New York, 1981.
(2) *Concise Encyclopedia of Chemistry*; McGraw-Hill: New York, 2004.
(3) Muzart, J. *Tetrahedron* **2003**, *59*, 5789–5816.
(4) For reviews on aerobic alcohol oxidations, see the following: (a) Sheldon, R. A.; Arends, I. W. C. E.; Ten Brink, G. J.; Dijksman, A. *Acc. Chem. Res.* **2002**, *35*, 774–781. (b) Nishimura, T.; Uemura, S. *Synlett* **2004**, 201–216. (c) Stahl, S. S. *Angew. Chem., Int. Ed.* **2004**, *43*, 3400–3420. (d) Sigman, M. S.; Jensen, D. R. *Acc. Chem. Res.* **2006**, *39*, 221–229.
(5) Schultz, M. J.; Sigman, M. S. *Tetrahedron* **2006**, *62*, 8227–8241.
(6) Steinhoff, B. A.; Stahl, S. S. *J. Am. Chem. Soc.* **2006**, *128*, 4348–4355.
(7) Berini, C.; Brayton, D. F.; Mocka, C.; Navarro, O. *Org. Lett.* **2009**, *11*, 4244–4247.
(8) Berini, C.; Winkelmann, O. H.; Otten, J.; Vicic, D. A.; Navarro, O. *Chem.—Eur. J.* **2010**, *16*, 6857–6860.
(9) For recent, general reviews on NHCs, see: (a) Nolan, S. P., Ed. *N-Heterocyclic Carbenes in Synthesis*; Wiley-VCH: Weinheim, Germany, 2006. (b) Glorius, F., Ed. *N-Heterocyclic Carbenes in Transition Metal Catalysis*; Springer: Berlin, Germany, 2007. (c) Díez-González, S.; Nolan, S. P. *Coord. Chem. Rev.* **2007**, *251*, 874–883. (d) Kantchev, E. A. B.; O'Brien, C. J.; Organ, M. G. *Angew. Chem., Int. Ed.* **2007**, *46*, 2768–2813. (e) Hahn, F. E.; Jahnke, M. C. *Angew. Chem., Int. Ed.* **2008**, *47*, 3122–3172. (f) Würtz, S.; Glorius, F. *Acc. Chem. Res.* **2008**, *41*, 1523–1533. (g) Dröge, T.; Glorius, F. *Angew. Chem., Int. Ed.* **2010**, *49*, 6940–6952.

Published on Web 01/21/2011 DOI: 10.1021/jo102385u

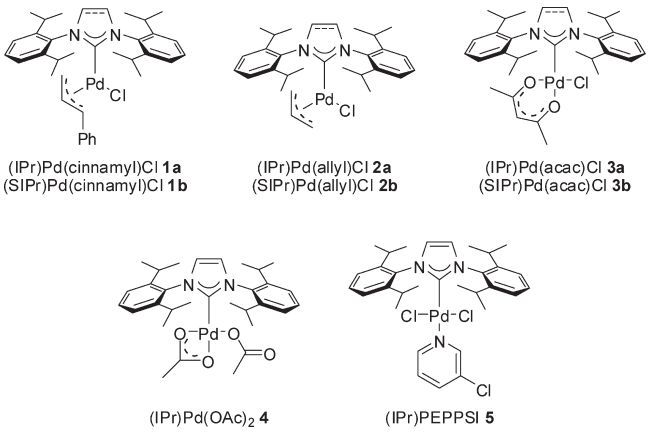
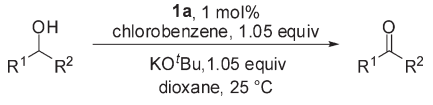
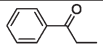
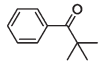
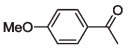
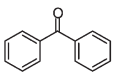
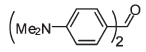
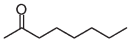
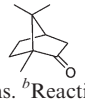


FIGURE 1. (NHC)-Pd complexes screened in this study.

TABLE 1. Preliminary Results for the Anaerobic Oxidation of Secondary Alcohols in 1,4-Dioxane[7]



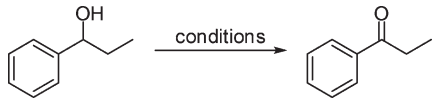
entry	product	time (h)	yield (%)[a]
1		11	91
2		12	91
3		14	90
4		12	85[b]
5		26	96[b]
6		20	71
7		27	73
8		16	93

[a]Average of two runs. [b]Reaction performed at 40 °C.

complex (IPr)Pd(cinnamyl)Cl (**1a**, Figure 1) (IPr = 1,3-bis-(2,6-diisopropylphenyl)imidazol-2-ylidene) in dioxane and using chlorobenzene as oxidant.[7] The system allowed for the synthesis of the corresponding ketones in high yields, at 25 °C or slightly higher temperature and with reaction times ranging from half to about one day (Table 1).

We have conducted a thorough optimization of this catalytic system and the results are depicted in Table 2. As we previously reported,[7] the use of 2- or 4-chlorotoluene led to similar results as those obtained with chlorobenzene, therefore avoiding the quantitative formation of benzene as a byproduct of the reaction (Table 2, entries 1−3). We have also found that switching from potassium to sodium *tert*-butoxide allows for a drastic reduction of the reaction

TABLE 2. Optimization of Catalytic Conditions for the Oxidation of 1-Phenylpropanol with (NHC)-Pd[a]



entry	complex (mol %)	chloride[b]	base	solvent	time (h)	yield (%)[c]
1	**1a** (1.0)	Ph-Cl	KO^{t}Bu	dioxane	8	91
2	**1a** (1.0)	2-Cl-Tol	KO^{t}Bu	dioxane	8	100[d]
3	**1a** (1.0)	4-Cl-Tol	KO^{t}Bu	dioxane	8	100[d]
4	**1a** (1.0)	4-Cl-Tol	NaO^{t}Bu	dioxane	1.25	100[d]
5	**1a** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.5	98
6	**1a** (1.0)	4-Cl-Tol	KO^{t}Bu	toluene	1	10[d]
7	**1a** (1.0)	4-Cl-Tol	NaO^{t}Bu	DME	0.5	100[d]
8	**1a** (1.0)	4-Cl-Tol	NaO^{t}Bu	MTBE	1.25	100[d]
9	**1a** (1.0)	4-Cl-Tol	NaO^{t}Bu	THF	1.5	100[d]
10	**2a** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.75	98
11	**3a** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.75	96
12	**4** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.75	97
13	**5** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	2	97
14	**1b** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.25	98
15	**2b** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.5	97
16	**3b** (1.0)	4-Cl-Tol	NaO^{t}Bu	toluene	0.75	97
17	**1b** (0.5)	4-Cl-Tol	NaO^{t}Bu	toluene	0.5	97
18	**1b** (0.25)	4-Cl-Tol	NaO^{t}Bu	toluene	1	99
19	**1b** (0.1)	4-Cl-Tol	NaO^{t}Bu	toluene	3.5	97

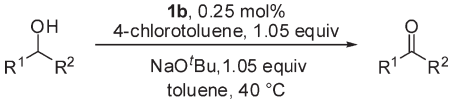
[a]Reaction conditions: alcohol, 0.50 mmol; aryl halide, 0.53 mmol; base, 0.53 mmol; solvent, 1 mL; room temperature. [b]Ph-Cl = chlorobenzene; 2-Cl-Tol = 2-chlorotoluene; 4-Cl-Tol = 4-chlorotoluene. [c]Average of two runs. [d]GC yields.

time, probably due to a better solubility in dioxane (Table 2, entry 4). We were delighted to observe that the system was compatible with a variety of solvents (Table 2, entries 4, 5, 7−9) and IPr-bearing Pd complexes (**1a**, **2a**, **3a**, **4**, **5**),[10] with only some slight differences in the reaction times (Table 2, entries 5, 10−13). In a following step, we compared the difference in performance between SIPr- and IPr-bearing complexes (SIPr = 1,3-bis(2,6-diisopropylphenyl)-4,5-dihydroimidazol-2-ylidene), since major differences in activity between these ligands have been described in a variety of catalytic reactions (Table 2, entries 14−16).[10a,c,11] A remarkable difference in performance was observed when SIPr-bearing complexes were tested as precatalysts for this reaction, especially when complex **1b**, also commercially available, was used. Using this complex, we were able to reduce the catalyst loading up to 1 order of magnitude with no detriment in the yield of the desired ketone (Table 2, entries 17−19). Unfortunately, we found later that these conditions were somehow substrate dependent and, in order to establish a general procedure, the temperature needed to be raised to 40 °C for most substrates in order to obtain good conversions

(10) (a) Cinnamyl chloride complexes: Marion, N.; Navarro, O.; Mei, J.; Stevens, E. D.; Scott, N. M.; Nolan, S. P. *J. Am. Chem. Soc.* **2006**, *128*, 4101–4111. (b) Allyl chloride complexes: Navarro, O.; Nolan, S. P. *Synthesis* **2006**, 366–367. (c) Acetylacetonate chloride complexes: Winkelmann, O. H.; Riekstins, A.; Nolan, S. P.; Navarro, O. *Organometallics* **2009**, *28*, 5809–5813. (d) Bis(acetate) complex: Jensen, D. R.; Schultz, M. J.; Mueller, J. A.; Sigman, M. S. *Angew. Chem., Int. Ed.* **2003**, *42*, 3810–3813. (e) (3-Chloropyridine) dichloride complex: O'Brien, C. J.; Kantchev, E. A. B.; Valente, C.; Hadei, N.; Chass, G. A.; Lough, A.; Hopkinson, A. C.; Organ, M. G. *Chem.—Eur. J.* **2006**, *12*, 4743–4748.

(11) (a) Stauffer, S. R.; Lee, S.; Stambuli, J. P.; Hauck, S. I.; Hartwig, J. F. *Org. Lett.* **2000**, *2*, 1423–1426. (b) Viciu, M. S.; Germaneau, R. F.; Nolan, S. P. *Org. Lett.* **2002**, *4*, 4053–4056. (c) Viciu, M. S.; Navarro, O.; Germaneau, R. F.; Kelly, R. A., III; Sommer, W.; Marion, N.; Stevens, E. D.; Cavallo, L.; Nolan, S. P. *Organometallics* **2004**, *23*, 1629–1635.

TABLE 3. Anaerobic Oxidation of Secondary Alcohols



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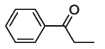
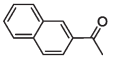
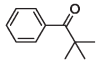
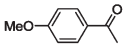
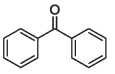
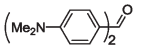
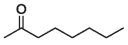
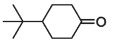
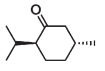
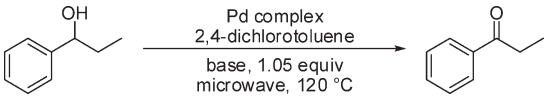
1		1	99[b]
2		1	98
3		5	94
4		0.25	92
5		1	99[b]
6		1.5	94[b]
7		1.5	90
8		0.5	99
9		1	88
10		2	70
11		1	99
12		4	94
13		0.5	77[b]
14		0.25	93[b]

[a]Average of two runs. [b]Reaction performed at 25 °C.

in short reaction times. Similar to when complex **1a** was used,[7] this improved system allows for the oxidation of a variety of secondary alcohols with aryl/alkyl (Table 3, entries 1−5, 8), aryl/aryl (Table 3, entries 6 and 7), and alkyl/alkyl substituents (Table 3, entries 9−14) at very mild temperature, affording yields of products comparable to those of the most common state-of-the-art Pd-catalyzed aerobic oxidations systems.[4,12] The differences in performance between our preliminary procedure and this improved system are clearly observed when comparing these results with those obtained with our previous system (Table 1): for a given substrate and at the same or lower temperature, reaction times have been reduced by 1 order of magnitude using only one-fourth of the palladium loading.

(12) For a substrate-dependent comparison of Pd-catalyzed aerobic systems, see: Schultz, M. J.; Hamilton, S. S.; Jensen, D. R.; Sigman, M. S. *J. Org. Chem.* **2005**, *70*, 3343–3352.

TABLE 4. Optimization of Catalytic Conditions for the Oxidation of 1-Phenylpropanol with (NHC)-Pd, Using Microwave Dielectric Heating



entry	complex (mol %)	base	time (min)	yield (%)[a,b]
1	**1a** (0.025)	NaO^{t}Bu	2	67
2	**1b** (0.025)	NaO^{t}Bu	2	70
3	**2a** (0.025)	NaO^{t}Bu	2	77
4	**2b** (0.025)	NaO^{t}Bu	2	67
5	**3a** (0.025)	NaO^{t}Bu	2	74
6	**3b** (0.025)	NaO^{t}Bu	2	66
7	**5** (0.025)	NaO^{t}Bu	2	68
8	**7** (0.025)	NaO^{t}Bu	2	84
9	**6b** (0.025)	NaO^{t}Bu	2	82
10	**6a** (0.025)	NaO^{t}Bu	2	93
11	**6a** (0.025)	KO^{t}Bu	2	37
12	**6a** (0.025)	NaOMe	2	49
13	**6a** (0.025)	CsF	2	< 5
14	**6a** (0.025)	Na_2CO_3	2	< 5
15	**6a** (0.05)	NaO^{t}Bu	5	100
16	**6a** (0.025)	NaO^{t}Bu	5	95
17	**6a** (0.01)	NaO^{t}Bu	5	81
18	**6a** (0.005)	NaO^{t}Bu	5	50

[a]Reaction conditions: alcohol (0.50 mmol), base (0.53 mmol), 2,4-dichlorotoluene (1 mL). [b]GC yields, average of two runs.

To our delight, this catalytic system is compatible with the use of microwave dielectric heating. The use of this technology has attracted much attention in the past decade since it allows for fast optimizations and short reaction times, making it a very useful tool in screening processes.[13] For our oxidation system, the use of the optimized conditions used in Table 3 led to incomplete reactions and/or the apparition of α-arylated ketones as byproduct. Therefore, a new optimization process was carried out (Table 4) with 1-phenylpropanol as substrate. We soon realized that the use of 2,4-dichlorotoluene as solvent/oxidant[8] precluded that extra coupling, leading to the exclusive formation of corresponding ketone. Interestingly, complexes **6** and **7** performed better at this high temperature than complexes **1**−**5**, even though they performed poorly at lower temperatures.[14] For this substrate, the use of commercially available complex **6a** allowed for complex loading and reaction time to be decreased by 1 order of magnitude when compared to the catalytic system at milder temperature. With these optimal conditions in hand, we proceeded to the microwave-assisted catalytic oxidation of a variety of secondary alcohols at 120 °C (Figure 2), being the results depicted in Table 5. High yields of the desired products were obtained in most cases, and substrates that did not oxidize under milder conditions (Table 5, entries 4 and 12) were smoothly oxidized to the corresponding ketones, bearing challenging functional groups such as thioether and alkene.

This catalytic system could be of utility for multistep synthesis because of its ability to perform oxidations using a mild oxidant that, after performing its task, becomes inert to starting material, product, and anything else that could be added to the reaction mixture for a second transformation.

(13) (a) Kappe, C. O. *Angew. Chem., Int. Ed.* **2004**, *43*, 6250–6284. (b) Dallinger, D.; Kappe, C. O. *Mol. Diversity* **2009**, *13*, 71–193.

(14) Using the same conditions as in Table 2, entry 14, yields and reaction times using these complexes are as follows: **6a**, 30%, 2.5 h; **6b**, 39%, 2.5 h; **7**, 52%, 2 h (all GC yields, average of two runs).

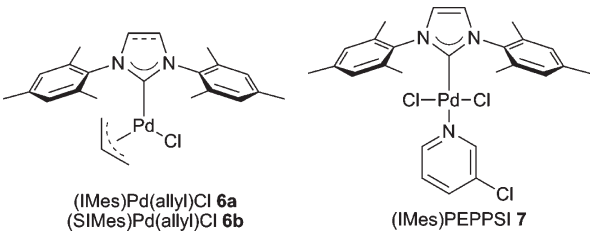
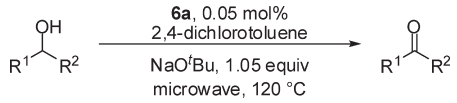
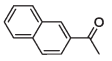
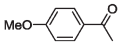
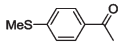
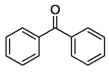
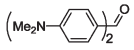
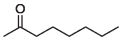


FIGURE 2. Additional (NHC)-Pd complexes screened for the anaerobic oxidation of secondary alcohols, using microwave dielectric heating.

TABLE 5. Anaerobic Oxidation of Secondary Alcohols with Microwave Dielectric Heating



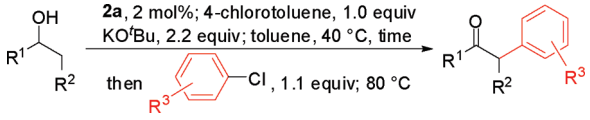
entry	product	time (min)	yield (%)[a]
1		5	96
2		10	92
3		5	97
4		10	94
5		5	95
6		5	90
7		5	96
8		10	88
9		5	85
10		10	67
11		10	86
12		10	93

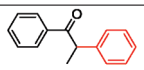
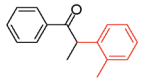
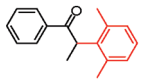
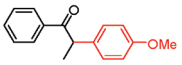
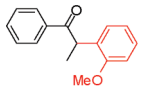
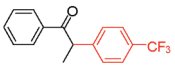
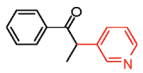
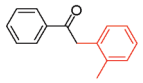
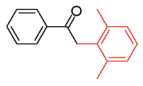
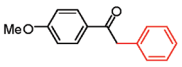
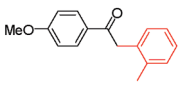
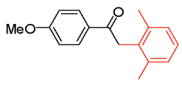
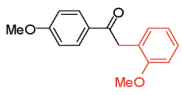
[a]Average of two runs.

To test this premise we decided to develop domino and one-pot oxidation/α-ketone arylation reactions[15] of aryl alcohols, since (NHC)-Pd complexes and imidazolium salt/Pd in situ systems have been extensively used in α-ketone arylation reactions.[9] There are two different possibilities when carrying out these reactions: we can either use a sacrificial, inexpensive aryl chloride for the oxidation and a more valuable chloride (because of price and/or availability) for the α-arylation (this sequence would be a one-pot reaction), or the same aryl chloride for both steps (this would be a domino reaction). The first approach was followed in Table 6 for a series of aryl alcohols, based on a single preliminary result presented in our preliminary report.[7] For these reactions, the oxidation step was carried out at 40 °C with 4-chlorotoluene as oxidant. Once it was determined by gas

(15) Very recent reviews on this reaction: (a) Bellina, F.; Rossi, R. *Chem. Rev.* **2010**, *110*, 1082–1146. (b) Johansson, C. C. C.; Colacot, T. J. *Angew. Chem., Int. Ed.* **2010**, *49*, 676–707.

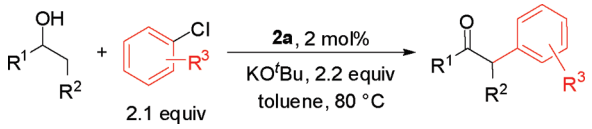
TABLE 6. One-Pot Oxidation/α-Ketone Arylation of Secondary Alcohols

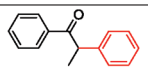
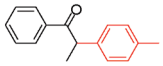
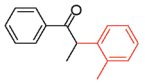
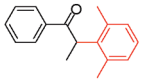
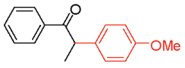
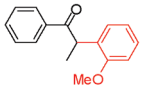
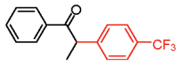
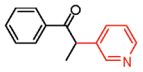
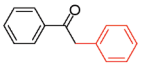
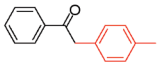
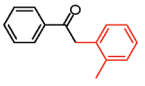
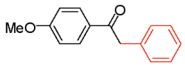
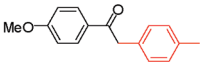
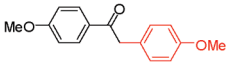


entry	product	time (h)[a]	yield (%)[b]
1		2.5 + 2	92
2		2.5 + 3	92
3		2.5 + 2	99
4		2.5 + 3	97
5		2.5 + 2	94
6		2.5 + 2	78
7		2.5 + 30	70
8		3 + 6	99
9		3 + 6	97
10		3 + 4	99
11		3 + 3	99
12		3 + 2	98
13		3 + 4	98

[a]Oxidation time + arylation time, monitored by gas chromatography. [b]Average of two runs.

TABLE 7. Domino Oxidation/α-Ketone Arylation of Secondary Alcohols



entry	product	time (h)	yield (%)[a]
1		1.5	99
2		2	94
3		4	98
4		0.5	88
5		1	96
6		1	96
7		1	76
8		24	81
9		3.5	92
10		3	95
11		3	90
12		4.5	90
13		4	96
14		6.5	86

[a]Average of two runs.

chromatography that the oxidation was completed, a second chloride was added and the temperature was raised to 80 °C to obtain the desired α-arylated product. Although this might seem pretty straightforward, initial attempts with complexes **1a** or **1b** in combination with NaO^{t}Bu derived, for some substrates, in the undesired apparition of the corresponding tolyl-arylated byproduct before the addition of the second chloride. To our delight, the use of complex (IPr)Pd(allyl)Cl (**2a**) in combination with KO^{t}Bu[16] led to clean oxidations with no signs of arylation with 4-chlorotoluene. Following this procedure, we were able to obtain the desired α-arylated products in a total of 9 h or less (with the exception of entry 7) in excellent yields and after two catalytic events using the same catalyst.

The domino approach, using the same aryl chloride for both oxidation and α-ketone arylation steps, was followed in Table 7. Since in this case we were not concerned about the possibility of multiple arylation products, the reactions were carried out at 80 °C with 2.2 equiv of aryl chloride. Following this procedure, the oxidation step was completed within 30 min and in most cases very good to excellent yields of the desired products were obtained.

In summary, we have presented a highly active, anaerobic (NHC)-Pd catalytic system for the oxidation of secondary alcohols at very mild temperatures. The use of complex **1b** as a precatalyst for this reaction allows for a drastic reduction of both catalyst loading and reaction times when compared to the unsaturated counterpart **1a**. We have also developed one-pot and domino procedures for the synthesis of α-arylated ketones from secondary aryl alcohols in very good yields.

Experimental Section

General Considerations. All reactions were carried out in oven-dried glassware and set up in a nitrogen-filled glovebox or with standard Schlenk techniques. All reagent-grade chemicals and solvents were obtained from commercial suppliers and were used as received unless otherwise noted. Toluene was refluxed over sodium and then distilled. Chromatography purifications were performed by column chromatography with silica gel (230−400 mesh). NMR spectra were recorded on either a 500 or 300 MHz spectrometer. Chemical shifts are given in ppm (δ) and were referenced to the internal solvent signal or to TMS used as an internal standard. Multiplicities are declared as follows: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), dd (doublet of doublet), ddd (doublet of doublet of doublet), dt (doublet of triplet), m (multiplet). Coupling constants J are given in hertz.

General Procedures for the Catalytic Oxidation of Secondary Alcohols. Conventional Heating. In a glovebox, (SIPr)Pd(cinnamyl)Cl (**1b**) (0.8 mg, 1.25×10^{-3} mmol, 0.25 mol %), sodium *tert*-butoxide (51 mg, 0.53 mmol), and dry toluene (1 mL) were loaded into a screw top vial with a septum equipped with a magnetic bar. Outside the glovebox the corresponding alcohol (0.50 mmol) and 4-chlorotoluene (63 μL, 0.53 mmol) were syringed in through the septum. When the alcohol was a solid, it was loaded in the glovebox. Then the mixture was allowed to stir at room temperature until the reaction reached completion or no further conversion was observed by gas chromatography. The combined mixtures from two vials were purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate, 1/0 to 8/2) affording the corresponding ketone.

Microwave Heating. In a glovebox, sodium *tert*-butoxide (51 mg, 0.53 mmol) and 25 μL of a 1 mol % standard solution of (IMes)Pd(ally)Cl (**6a**) (0.12 mg, 2.5×10^{-4} mmol, 0.05 mol %) in dry toluene were loaded into a 10 mL microwave vial with a septum cap and equipped with a magnetic bar. Outside the glovebox 2,4-dichlorotoluene (950 μL) and the corresponding alcohol (0.50 mmol) were syringed in through the septum.

(16) The use of complex **2a** in combination with NaO^{t}Bu also led to the formation of about 10% of the tolyl-arylated byproduct before the addition of the second aryl chloride.

When the alcohol was a solid, it was loaded in the vial in the glovebox. The mixture was then allowed to stir at room temperature for approximately 2 min and then placed in a CEM Discover microwave reactor with an IR sensor. The reaction parameters were as follows: temperature 120 °C, hold time 5−10 min, pressure 250 psi, power 300 W, power max off, and stirring on high. The combined mixtures from two vials were purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate, 1/0 to 8/2) affording the corresponding ketone.

Propiophenone (Table 3, entry 1 and Table 5, entry 1). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 90 min with conventional heating (129 mg, 0.95 mmol, 95%) and after 5 min with the microwave reactor (130 mg, 0.96 mmol, 96%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.23 (t, J = 7.2 Hz, 3H), 3.02 (q, J = 7.2 Hz, 2H), 7.43−7.49 (m, 2H), 7.53−7.59 (m, 1H), 7.96−7.99 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 8.1, 31.7, 127.9, 128.4, 132.8, 136.8, 200.7.

Acetophenone (Table 3, entry 2). The title compound was prepared according to the above general procedure from 1-ethyl-1-propanol (0.50 mmol, 66 μL) as a light yellow oil after 60 min at 40 °C (120 mg, 0.98 mmol, 98%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.61 (s, 3H), 7.45−7.48 (m, 2H), 7.55−7.59 (m, 1H), 7.96−7.97 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.5, 128.3, 128.6, 133.0, 137.3, 198.0.

2-Acetonaphthone (Table 3, entry 3 and Table 5, entry 2). The title compound was prepared according to the above general procedure from 1-(2-naphthyl)ethanol (0.50 mmol, 86 mg) as a off white solid after 300 min at 40 °C with conventional heating (160 mg, 0.94 mmol, 94%) and after 10 min with the microwave reactor (156 mg, 0.92 mmol, 92%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.73 (s, 3H), 7.54−7.62 (m, 2H), 7.89 (t, J = 8.2 Hz, 2H), 7.97 (d, J = 8.1 Hz, 1H), 8.04 (dd, J = 8.6 and 1.7 Hz, 1H), 8.47 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.7, 123.9, 126.8, 127.8, 128.4, 128.4, 129.5, 130.2, 132.5, 134.5, 135.6, 198.0.

2,2-Dimethylpropiophenone (Table 3, entry 4). The title compound was prepared according to the above general procedure from 2,2-dimethyl-1-phenyl-1-propanol (0.50 mmol, 83 mg) as an yellow oil after 15 min at 40 °C (150 mg, 0.92 mmol, 92%). ^{1}H NMR matched the values reported in the literature.[17] ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.35 (s, 9H), 7.36−7.48 (m, 3H), 7.66−7.69 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 27.9, 44.1, 127.7, 128.0, 130.7, 138.5, 209.1.

4-Methoxyacetophenone (Table 3, entry 5 and Table 5, entry 3). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as an yellow oil after 60 min with conventional heating (149 mg, 0.99 mmol, 99%) and after 5 min with the microwave reactor (145 mg, 0.97 mmol, 97%). ^{1}H NMR matched the values reported in the literature.[17] ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.56 (s, 3H), 3.87 (s, 3H), 6.94 (d, J = 9.0 Hz, 2H), 7.94 (d, J = 9.0 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.3, 55.4, 113.7, 130.4, 130.6, 163.5, 196.7.

Benzophenone (Table 3, entry 6 and Table 5, entry 5). The title compound was prepared according to the above general procedure from diphenylmethanol (0.50 mmol, 93 mg) as a off white solid after 90 min with conventional heating (172 mg, 0.94 mmol, 94%) and after 5 min with the microwave reactor (174 mg, 0.95 mmol, 95%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46−7.51 (m, 4H), 7.57−7.62 (m, 2H), 7.80−7.82 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 128.2, 129.9, 132.3, 137.4, 196.6.

(17) Schultz, M. J.; Hamilton, S. S.; Jensen, D. R.; Sigman, M. S. *J. Org. Chem.* **2005**, *70*, 3343–3352.

4,4′-Bis(dimethylamino)benzophenone (Table 3, entry 7 and Table 5, entry 6). The title compound was prepared according to the above general procedure from 4,4′-bis(dimethylamino)-diphenyl carbinol (0.50 mmol, 142 mg) as a light green solid after 90 min at 40 °C with conventional heating (242 mg, 0.90 mmol, 90%) and after 5 min with the microwave reactor (242 mg, 0.90 mmol, 90%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.06 (s, 12H), 6.69 (d, J = 9.0 Hz, 4H), 7.76 (d, J = 9.0 Hz, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 40.0, 110.4, 126.2, 132.1, 152.6, 193.9.

α-Tetralone (Table 3, entry 8 and Table 5, entry 7). The title compound was prepared according to the above general procedure from 1,2,3,4-tetrahydro-1-naphthol (0.50 mmol, 53 μL) as a brown oil after 30 min at 40 °C with conventional heating (148 mg, 0.99 mmol, 99%) and after 5 min with the microwave reactor (141 mg, 0.96 mmol, 96%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.12−2.18 (m, 2H), 2.64−2.69 (m, 2H), 2.97 (t, J = 6.1 Hz, 2H), 7.23−7.32 (m, 2H), 7.46 (dt, J = 7.5 and 1.4 Hz, 1H), 8.03 (dd, J = 7.8 and 1.4 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 23.3, 29.7, 39.2, 126.6, 127.2, 128.7, 132.6, 133.3, 144.5, 198.3.

2-Octanone (Table 3, entry 9 and Table 5, entry 8). The title compound was prepared according to the above general procedure from 2-octanol (0.50 mmol, 82 μL) as an yellow oil after 60 min at 40 °C with conventional heating (113 mg, 0.88 mmol, 88%) and after 10 min with the microwave reactor (113 mg, 0.88 mmol, 88%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.88 (t, J = 6.7 Hz, 3H), 1.27−1.31 (m, 6H), 1.52−1.62 (m, 2H), 2.14 (s, 3H), 2.42 (t, J = 7.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.0, 22.4, 23.8, 28.8, 29.8, 31.5, 43.7, 209.4.

Cyclohexanone (Table 3, entry 10). The title compound was prepared according to the above general procedure from cyclohexanol (0.50 mmol, 53 μL) as an yellow oil after 120 min at 40 °C (69 mg, 0.70 mmol, 70%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.69−1.76 (m, 2H), 1.87 (quint, J = 6.6 Hz, 4H), 2.34 (t, J = 6.6 Hz, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.9, 27.0, 41.9, 212.1.

2-Methylcyclohexanone (Table 3, entry 11 and Table 5, entry 9). The title compound was prepared according to the above general procedure from 2-methylcyclohexanol (0.50 mmol, 62 μL) as an yellow oil after 60 min at 40 °C with conventional heating (121 mg, 0.99 mmol, 99%) and after 5 min with the microwave reactor (95 mg, 0.85 mmol, 85%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.00 (d, J = 6.7 Hz, 3H), 1.31−1.40 (m, 1H), 1.58−1.70 (m, 2H), 1.78−1.85 (m, 1H), 2.01−2.09 (m, 2H), 2.23−2.30 (m, 1H), 2.33−2.41 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.7, 25.1, 27.9, 36.1, 41.8, 45.3, 213.5.

4-*tert*-Butylcyclohexanone (Table 3, entry 12). The title compound was prepared according to the above general procedure from 4-*tert*-butylcyclohexanol (0.50 mmol, 80 mg) as a off white solid after 240 min at 40 °C (145 mg, 0.94 mmol, 94%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.92 (s, 9H), 1.41−1.50 (m, 3H), 2.07−2.10 (m, 2H), 2.28−2.34 (m, 2H), 2.39−2.41 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 27.6, 32.5, 41.3, 46.7, 212.6.

Menthone (Table 3, entry 13 and Table 5, entry 10). The title compound was prepared according to the above general procedure from menthol (0.50 mmol, 80 mg) as an yellow oil after 30 min with conventional heating (119 mg, 0.77 mmol, 77%) and after 10 min with the microwave reactor (103 mg, 0.67 mmol, 67%). ^{1}H NMR matched that of a commercial sample (mixture of diastereoisomers, dr = 75:25). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.85 (two d, J = 6.6 and 6.8 Hz, 3H), 0.91 and 0.93 (two d, J = 6.8 and 6.6 Hz, 3H), 0.99 and 1.00 (two d, J = 6.7 and 6.4 Hz, 3H), 1.32−1.42 (m, 1.5H), 1.46−1.52 (m, 0.25H), 1.67−1.76 (m, 0.5H), 1.81−2.17 (m, 5.75H), 2.29−2.32 (m, 0.25H), 2.35 (ddd,

J = 13.0, 2.3, and 2.2 Hz, 0.75H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.7, 19.8, 20.9, 21.2, 21.3, 22.2, 26.0, 26.8, 27.0, 28.0, 29.6, 34.0, 34.2, 35.4, 48.1, 50.9, 56.0, 57.2, 212.1.

Camphor (Table 3, entry 14 and Table 5, entry 11). The title compound was prepared according to the above general procedure from borneol (0.50 mmol, 79 mg) as a white solid after 15 min with conventional heating (142 mg, 0.93 mmol, 93%) and after 10 min with the microwave reactor (136 mg, 0.86 mmol, 86%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.84 (s, 3H), 0.91 (s, 3H), 0.96 (s, 3H), 1.31−1.43 (m, 2H), 1.65−1.71 (m, 1H), 1.82−1.86 (m, 1H), 1.91−1.99 (m, 1H), 2.09 (t, J = 4.5 Hz, 1H), 2.35 (dt, J = 18.3 and 3.9 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 9.2, 19.2, 19.8, 27.1, 30.0, 43.2, 43.3, 46.8, 57.7, 219.3.

4-(Methylthio)acetophenone (Table 5, entry 4). The title compound was prepared according to the above general procedure from α-methyl-4-(methylthio)benzenemethanol (0.50 mmol, 84 mg) as a yellow solid after 10 min with the microwave reactor (156 mg, 0.94 mmol, 94%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.53 (s, 3H), 2.57 (s, 3H), 7.87 (d, J = 8.6 Hz, 2H), 7.27 (d, J = 8.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.9, 26.7, 125.1, 128.9, 133.7, 146.1, 197.4.

(−)-Carvone (Table 5, entry 12). The title compound was prepared according to the above general procedure from (−)-carveol (0.50 mmol, 82 μL) as a light yellow oil after 10 min with the microwave reactor (140 mg, 0.93 mmol, 93%). ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.75 (s, 3H), 1.79 (s, 3H), 2.23−2.34 (m, 2 H), 2.40−2.48 (m, 1H), 2.57−2.73 (m, 2H), 4.78 (d, J = 15 Hz, 2H), 6.76 (d, J = 4.4 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 15.7, 20.5, 31.1, 42.4, 43.0, 110.4, 135.4, 144.6, 146.7, 199.8.

General Procedure for the Catalytic Tandem Oxidation/α-Ketone Arylation of Secondary Alcohols. Conditions A, Domino Reaction. In the glovebox, (IPr)Pd(allyl)Cl (**2a**) (5.7 mg, 0.01 mmol, 2 mol %), potassium *tert*-butoxide (123 mg, 1.10 mmol), and dry toluene (1.5 mL) were loaded into a screw top vial with a septum equipped with a magnetic bar. Outside the glovebox the corresponding alcohol (0.50 mmol) and aryl halide (1.05 mmol) were syringed in through the septum. Then the mixture was allowed to stir at 80 °C until the reaction reached completion or no further conversion was observed by gas chromatography. The combined mixtures from two vials were purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate, 1/0 to 8/2) affording the corresponding product.

Conditions B, One-Pot Reaction. In the glovebox, (IPr)-Pd(allyl)Cl (**2a**) (5.7 mg, 0.01 mmol, 2 mol %), potassium *tert*-butoxide (123 mg, 1.10 mmol), and dry toluene (1.5 mL) were loaded into a screw top vial with a septum equipped with a magnetic bar. Outside the glovebox the corresponding alcohol (0.50 mmol) and 4-chlorotoluene (60 μL, 0.50 mmol) were syringed in through the septum. Then the mixture was allowed to stir at 40 °C until the reaction reached completion or no further conversion was observed by gas chromatography. Then the second aryl halide (0.55 mmol) was syringed in through the septum and the mixture was allowed to stir at 80 °C until the reaction reached completion or no further conversion was observed by gas chromatography. The combined mixtures from two vials were purified by flash chromatography on silica gel (eluent: hexane/ethyl acetate, 1/0 to 8/2) affording the corresponding product.

1-Phenyl-2-(phenyl)-1-propanone (Table 6, entry 1 and Table 7, entry 1). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 1.5 h (209 mg, 0.99 mmol, 99%) with conditions A and 2.5 + 2 h (194 mg, 0.92 mmol, 92%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.53 (d, J = 6.9 Hz, 3H), 4.68 (q, J = 6.9 Hz, 1H), 7.16−7.22 (m, 1H), 7.28−7.29 (m, 4H), 7.36−7.39 (m, 2H), 7.45−7.48 (m, 1H), 7.94−7.95 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.5, 47.9, 126.9, 127.7, 128.4, 128.7, 128.9, 132.7, 136.5, 141.4, 200.3.

2-(4-Methylphenyl)-1-phenyl-1-propanone (Table 7, entry 2). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 2 h (210 mg, 0.94 mmol, 94%) with conditions A. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.52 (d, J = 6.9 Hz, 3H), 2.29 (s, 3H), 4.65 (q, J = 6.9 Hz, 1H), 7.10 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.1 Hz, 2H), 7.38 (t, J = 7.7 Hz, 2H), 7.45−7.48 (m, 1H), 7.94−7.96 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.5, 21.0, 47.5, 127.6, 128.4, 128.7, 129.7, 132.6, 136.5, 138.5, 200.4.

2-(2-Methylphenyl)-1-phenyl-1-propanone (Table 6, entry 2 and Table 7, entry 3). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 4 h (220 mg, 0.98 mmol, 98%) with conditions A and 2.5 + 3 h (206 mg, 0.92 mmol, 92%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.47 (d, J = 6.8 Hz, 3H), 2.50 (s, 3H), 4.75 (q, J = 6.8 Hz, 1H), 7.02−7.03 (m, 1H), 7.06−7.12 (m, 2H), 7.19−7.20 (m, 1H), 7.34 (t, J = 7.7 Hz, 2H), 7.44 (t, J = 7.4 Hz, 1H), 7.81−7.83 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 18.0, 19.6, 44.6, 126.8, 126.9, 127.0, 128.5, 130.9, 132.6, 134.5, 136.6, 140.1, 200.9.

2-(2,6-Dimethylphenyl)-1-phenyl-1-propanone (Table 6, entry 3 and Table 7, entry 4). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a off white solid after 0.5 h (210 mg, 0.88 mmol, 88%) with conditions A and 2.5 + 2 h (235 mg, 0.99 mmol, 99%) with conditions B. ^{1}H NMR matched the reported literature values.[18] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.51 (d, J = 6.8 Hz, 3H), 2.29 (s, 6H), 4.53 (q, J = 6.8 Hz, 1H), 6.96−7.01 (m, 3H), 7.27−7.30 (m, 2H), 7.39−7.43 (m, 1H), 7.69−7.72 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.8, 20.5, 46.2, 126.7, 128.2, 128.2, 128.3, 129.5, 132.5, 135.6, 136.8, 139.8, 202.2.

2-(4-Methoxyphenyl)-1-phenyl-1-propanone (Table 6, entry 4 and Table 7, entry 5). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 1 h (230 mg, 0.96 mmol, 96%) with conditions A and 2.5 + 3 h (232 mg, 0.97 mmol, 97%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.50 (d, J = 6.9 Hz, 3H), 3.75 (s, 3H), 4.63 (q, J = 6.9 Hz, 1H), 6.82 (d, J = 8.7 Hz, 2H), 7.19 (d, J = 8.8 Hz, 2H), 7.37 (t, J = 7.8 Hz, 2H), 7.46 (t, J = 7.4 Hz, 1H), 7.93−7.95 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.5, 47.0, 55.2, 114.4, 128.4, 128.7, 128.8, 132.7, 133.5, 136.6, 158.5, 200.5.

2-(2-Methoxyphenyl)-1-phenyl-1-propanone (Table 6, entry 5 and Table 7, entry 6). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 1 h (230 mg, 0.96 mmol, 96%) with conditions A and 2.5 + 2 h (226 mg, 0.94 mmol, 94%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.46 (d, J = 6.8 Hz, 3H), 3.87 (s, 3H), 5.07 (q, J = 6.8 Hz, 1H), 6.84−6.87 (m, 2H), 7.10−7.12 (m, 1H), 7.15−7.19 (m, 1H), 7.35 (t, J = 7.6 Hz, 2H), 7.44 (t, J = 7.4 Hz, 1H), 7.94−7.96 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 17.6, 40.4, 55.5, 110.8, 121.0, 128.0, 128.1, 128.3, 128.5, 130.2, 132.5, 136.6, 155.8, 201.3.

1-Phenyl-2-[4-(trifluoromethyl)phenyl]-1-propanone (Table 6, entry 6 and Table 7, entry 7). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 1 h (209 mg, 0.76 mmol, 76%) with conditions A and 2.5 + 2 h (217 mg, 0.78 mmol,

(18) Grasa, G. A.; Colacot, T. J. *Org. Lett.* **2007**, *9*, 5489–5492.

78%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.56 (d, J = 6.9 Hz, 3H), 4.77 (q, J = 6.9 Hz, 1H), 7.41−7.43 (m, 4H), 7.52−7.57 (m, 3H), 7.92−7.96 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.4, 47.4, 124.0 (q, J = 271.9 Hz), 125.8 (d, J = 3.5 Hz), 128.1, 128.6, 128.7, 129.2 (q, J = 32.5 Hz), 133.1, 136.0, 145.3, 199.6.

1-Phenyl-2-(pyridin-3-yl)-1-propanone (Table 6, entry 7 and Table 7, entry 8). The title compound was prepared according to the above general procedure from 1-phenyl-1-propanol (0.50 mmol, 72 μL) as a light yellow oil after 24 h (170 mg, 0.81 mmol, 81%) with conditions A and 2.5 + 30 h (147 mg, 0.70 mmol, 70%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.57 (d, J = 6.9 Hz, 3H), 4.75 (q, J = 6.9 Hz, 1H), 7.22 (dd, J = 7.9 and 4.5 Hz, 1H), 7.42 (t, J = 7.7 Hz, 2H), 7.50−7.54 (m, 1H), 7.61 (dt, J = 7.9 and 1.9 Hz, 1H), 7.94−7.96 (m, 2H), 8.47 (dd, J = 4.7 and 1.3 Hz, 1H), 8.61 (d, J = 1.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.4, 44.9, 123.8, 128.7, 133.2, 135.0, 136.0, 136.9, 148.4, 149.5, 199.6.

1,2-Diphenylethanone (Table 7 entry 9). The title compound was prepared according to the above general procedure from 1-ethyl-1-propanol (0.50 mmol, 66 μL) as a light yellow oil after 3.5 h (180 mg, 0.92 mmol, 92%) with conditions A. ^{1}H NMR matched the reported literature values.[19] ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.29 (s, 2H), 7.28−7.36 (m, 4H), 7.46−7.59 (m, 4H), 8.02 (d, J = 7.9 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 45.7, 127.1, 128.8, 129.7, 133.4, 134.8, 136.8, 197.9.

2-(4-Methylphenyl)-1-phenylethanone (Table 7, entry 10). The title compound was prepared according to the above general procedure from 1-ethyl-1-propanol (0.50 mmol, 66 μL) as a light yellow oil after 3 h (210 mg, 0.95 mmol, 95%) with conditions A. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.32 (s, 3H), 4.25 (s, 2H), 7.12−7.18 (m, 4H), 7.43−7.48 (m, 2H), 7.53−7.58 (m, 1H), 8.00−8.02 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.0, 45.1, 128.6, 129.3, 129.4, 131.4, 133.1, 136.4, 136.6, 197.8.

2-(2-Methylphenyl)-1-phenylethanone (Table 6, entry 8 and Table 7, entry 11). The title compound was prepared according to the above general procedure from 1-ethyl-1-propanol (0.50 mmol, 66 μL) as a light yellow oil after 3 h (190 mg, 0.90 mmol, 90%) with conditions A and after 3 + 6 h (210 mg, 0.99 mmol, 99%) with conditions B. ^{1}H NMR matched the reported literature values.[11b] ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.26 (s, 3H), 4.30 (s, 2H), 7.09−7.25 (m, 4H), 7.46−7.49 (m, 2H), 7.55−7.59 (m, 1H), 8.01−8.03 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.8, 43.5, 126.1, 127.2, 128.3, 128.6, 130.3, 130.3, 133.1, 133.4, 136.9, 136.9, 197.4.

2-(2,6-Dimethylphenyl)-1-phenylethanone (Table 6, entry 9). The title compound was prepared according to the above general procedure from 1-ethyl-1-propanol (0.50 mmol, 66 μL) as a off white solid after 3 + 6 h (218 mg, 0.97 mmol, 97%) with conditions B. ^{1}H NMR matched the reported literature values.[18] ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.21 (s, 6H), 4.36 (s, 2H), 7.04−7.08 (m, 3H), 7.47−7.50 (m, 2H), 7.56−7.59 (m, 1H), 8.06−8.07 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.3, 39.6, 126.8, 127.9, 128.0, 128.6, 132.4, 133.1, 136.9, 137.1, 196.8.

1-(4-Methoxyphenyl)-2-phenylethanone (Table 6, entry 10 and Table 7, entry 12). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as a light yellow oil after 4.5 h (203 mg, 0.90 mmol, 90%) with conditions A and after 3 + 4 h (223 mg, 0.99 mmol, 99%) with conditions B. ^{1}H NMR matched the reported literature values.[20] ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.85 (s, 3H), 4.23 (s, 2H), 6.92 (d, J = 8.9 Hz, 2H), 7.23−7.34 (m, 5H), 7.99 (d, J = 8.9 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 45.2, 55.4, 113.8, 126.7, 128.6, 129.4, 130.9, 132.4, 134.9, 163.5, 196.2.

1-(4-Methoxyphenyl)-2-(4-methylphenyl)ethanone (Table 7, entry 13). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as a light yellow oil after 4 h (230 mg, 0.96 mmol, 96%) with conditions A. ^{1}H NMR matched the reported literature values.[21] ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.31 (s, 3H), 3.85 (s, 3H), 4.19 (s, 2H), 6.90 (d, J = 8.9 Hz, 2H), 7.10−7.17 (m, 4H), 7.99 (d, J = 8.9 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.0, 44.9, 55.4, 113.7, 129.2, 129.3, 129.6, 130.9, 131.8, 136.3, 163.4, 196.4.

1-(4-Methoxyphenyl)-2-(2-methylphenyl)ethanone (Table 6, entry 11). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as a light yellow oil after 3 + 3 h (237 mg, 0.99 mmol, 99%) with conditions B. ^{1}H NMR matched the reported literature values.[22] ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.26 (s, 3H), 3.86 (s, 3H), 4.24 (s, 2H), 6.93−6.95 (m, 2H), 7.11−7.20 (m, 4H), 7.99−8.01 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.8, 43.1, 55.5, 113.8, 126.0, 127.1, 130.0, 130.2, 130.3, 130.6, 133.8, 136.8, 163.5, 196.0.

2-(2,6-Dimethylphenyl)-1-(4-methoxyphenyl)ethanone (Table 6, entry 12). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as a light yellow oil after 3 + 2 h (250 mg, 0.98 mmol, 98%) with conditions B. ^{1}H NMR matched the reported literature values.[18] ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.22 (s, 6H), 3.90 (s, 3H), 4.33 (s, 2H), 6.97−6.99 (m, 2H), 7.05−7.17 (m, 3H), 8.06−8.08 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.4, 39.3, 55.5, 113.8, 126.8, 127.9, 130.3, 132.8, 137.0, 163.5, 195.4.

Desoxyanisoin (Table 7, entry 14). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as a light yellow oil after 6.5 h (220 mg, 0.86 mmol, 86%) with conditions A. ^{1}H NMR matched that of a commercial sample. ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.78 (s, 3H), 3.86 (s, 3H), 4.17 (s, 2H), 6.86 (d, J = 8.7 Hz, 2H), 6.92 (d, J = 8.9 Hz, 2H), 7.18 (d, J = 8.7 Hz, 2H), 7.99 (d, J = 8.9 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 44.4, 55.2, 55.4, 113.7, 114.1, 126.9, 130.3, 130.9, 152.2, 158.4, 163.4, 196.5.

2-(2-Methoxyphenyl)-1-(4-methoxyphenyl)ethanone (Table 6, entry 13). The title compound was prepared according to the above general procedure from 4-methoxy-α-methylbenzyl alcohol (0.50 mmol, 71 μL) as a light yellow oil after 3 + 4 h (250 mg, 0.98 mmol, 98%) with conditions B. ^{1}H NMR matched the reported literature values.[23] ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.79 (s, 3H), 3.85 (s, 3H), 4.22 (s, 2H), 6.87−6.93 (m, 4H), 7.17−7.18 (m, 1H), 7.22−7.24 (m, 1H), 8.01−8.03 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 39.5, 55.4, 55.4, 110.7, 113.7, 120.7, 124.2, 128.2, 130.7, 130.9, 157.2, 163.4, 196.6.

Acknowledgment. This work was supported by the University of Hawaii at Manoa. The authors wish to thank Prof. D. Horgan and J. Reinicke (Hawaii Pacific University) for allowing us access to their NMR instrument during renovations in our facilities.

Supporting Information Available: Compound characterization data. This material is available free of charge via the Internet at http://pubs.acs.org.

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