

Electrophilic amination of organozinc reagents with acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime and *O*-methylhydroxylamine

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Received (in Cambridge, UK) 27th July 1999, Accepted 8th September 1999

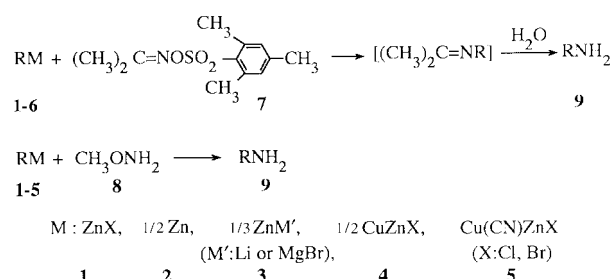
Reaction of diorganozincs and triorganozincates with acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime or *O*-methylhydroxylamine in the presence of CuCN provides a new one-flask method for electrophilic amination of organozinc reagents. Considering the lithium- or magnesium-to-zinc transmetallation, this method also extends the scope of electrophilic amination of organolithiums and Grignard reagents.

Introduction

The importance of amines and the commonplace use of organometallic reagents in organic synthesis has created the need for 'NH₂⁺'-containing reagents capable of C–N connection. There are detailed reviews on the amination of carbanions.¹ In addition, a number of new reagents have been recently developed, among them *N,O*-bis(trimethylsilyl)-hydroxylamine,² LiN(OTs)CO₂Bu^t (LiBTOC),³ *N*-protected oxaziridines,⁴ 1-chloro-1-nitrosocyclohexane,⁵ acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime,⁶ 4,4'-bis(trifluoromethyl)benzophenone *O*-methyloxime⁷ and bis(2,2,2-trichloroethyl) azodicarboxylate.⁸

Efficient methods are known for electrophilic amination of organolithiums,^{1a,3a–d,4,5,9} Grignard reagents,^{1a,6,7,9} organocoppers^{1a,2,3,8–10} and organoboranes.¹¹ However, to date, there have been only some unsuccessful attempts on the amination of ordinary organozinc reagents^{1a,3e,8,9,12,13} and a few successful amination examples for zinc enolates.^{3a,5,14} Recently, two papers have appeared on the amination of organozinc reagents. In the first,¹⁵ zinc cyanocuprates react with lithium amides and the formed zinc amidocyanocuprates, upon oxidation, decompose to provide amines. In the second,¹⁶ the addition of organozinc halides to di-*tert*-butyl azodicarboxylate (DBAD) is described to give hydrazino derivatives, which give amines after the removal of *tert*-butoxycarbonyl groups and then reductive cleavage of the N–N bond.

The importance of organozinc reagents^{17–20} due to their high reactivity, easy preparation by Li- and Mg-to-Zn transmetalation, and the use of electrophilic functional group-containing reagents prompted us to investigate the electrophilic amination of organozinc reagents. Since the major drawback related to the use of organozinc reagents is the use of active zinc, the work to be discussed here involves preparation of organozincs by transmetalation, except for some functional group-containing organozincs which are prepared by metallation of organic halides. In our study, the reagents of choice for amination of organozincs are acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime and *O*-methylhydroxylamine (Scheme 1). Acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime **7** has been developed in our laboratories for amination of Grignard reagents⁶ and reacts by displacement of the mesitylsulfonyloxy group giving an imine, which upon hydrolysis provides amines. *O*-Methylhydroxylamine and its methyllithium complex have long been used successfully for amination of organolithiums,^{9,21} Grignard reagents^{9,21–23} and organocoppers⁹ and once unsuccessfully for organozincs.⁹ Compound **7** can be more easily prepared and conveniently stored than can **8**.



Scheme 1 Amination of organozinc species **1–6** with acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime **7** and *O*-methylhydroxylamine **8**.

We have reported in an earlier paper²⁴ a simple and mild method for electrophilic amination of diarylzincs and triarylzincates with **7** and **8**. In this paper, we explore the use of this method.

Results and discussion

At the initial stage of this investigation, we focused on the amination of phenylzinc species with **7** and **8** for determining the optimal reaction conditions. Phenylzinc chloride **1a**, diphenylzinc **2a** and lithium (or bromomagnesium) triphenylzincate **3a** were prepared by transmetalation of the corresponding phenyllithium or phenylmagnesium bromide with ZnCl₂; chlorozinc diphenylcuprate **4a** and chlorozinc phenylcyanocuprate **5a** were prepared by transmetalation of *in situ* prepared lithium diphenylcuprate and lithium phenylcyanocuprate, respectively, with ZnCl₂.¹⁹ Amination yields of phenylzinc reagents **1a–6a** with **7** and **8** are given in Table 1.

We observed that the reaction of phenylzinc chloride **1a**, diphenylzinc **2a**, and triphenylzincate **3a** with **7** takes place only in the presence of CuCN, except a low-yield reaction of **3a** with **8** (entry 7).

The reaction conditions were optimized by adding 1 mol equiv. of aminating reagent **7** or **8** to 1.5 mol equiv. of phenylzinc reagents **1a**, **2a** and phenylzincate **3a** or 2 mol equiv. of zinc phenylcuprates **4a**, **5a** in diethyl ether or in diethyl ether–THF at room temperature, resulting in almost complete consumption of **7** or **8** within 3 h. Experiments indicated that the yield is strongly dependent on the type of phenylzinc species and on the amount of CuCN. The highest yield in the amination with **7** is obtained by treating triphenylzincate **3a** in the presence of 20 mol% CuCN (entry 9); however phenylzinc chloride **1a** and diphenylzinc **2a** are also effective in CuCN-mediated amination (entries 3 and 6). Carrying out the

Table 1 Reactions of phenylzinc reagents **1a–6a** with aminating reagents **7** and **8**^a

PhM + 7 or 8 1a–6a		1. THF, rt, 3 h 2. Hydrolysis PhNH ₂ 9		
Entry	PhM ^b	CuCN (mol%)	Yield of PhNH ₂ ^c	
			Aminating reagent	
			7	8
1	PhZnCl 1a		0	0
2	PhZnCl 1a	10	25(25 ^d)	
3	PhZnCl 1a	20	58	7
4	PhZnCl 1a	20	48 ^e	
5	Ph ₂ Zn 2a		0	
6	Ph ₂ Zn 2a	20	44	82(82 ^f)
7	Ph ₃ ZnLi 3a		0	33
8	Ph ₃ ZnLi 3a	10	52	
9	Ph ₃ ZnLi 3a	20	70	95(89, ^g 95 ^f)
10	Ph ₂ CuZnCl 4a		49	50(50 ^f)
11	PhCu(CN)ZnCl 5a		56	42(38 ^f)
12	PhZnCl 1a	20	65(49 ^f)	
13	Ph ₂ Zn 2a	20	68(51 ^f)	
14	Ph ₂ ZnMgBr 3a	20	85(76 ^f)	
15	Ph ₃ ZnLi 3a ^h	20	79(70 ^f)	
16	Me ₂ PhZnLi 6a	20	59	

^a Reactions were carried out on 1.5 or 3.0 mmol aminating reagent **7** or **8**. PhM:**7** = 1.5 and PhM:**8** = 2 for **1a**, **2a** and **3a**; PhM:**7** (or **8**) = 2 for **4a** and **5a**. ^b Phenylzinc reagents in entries 1–10, 15 and 16 were prepared by Li-to-Zn transmetallation and in entries 11–14 by Mg-to-Zn transmetallation. ^c Yield determined as average of two or more runs by GC using internal standard technique and based on **7** or **8**. ^d Yield of aniline purified by column chromatography. ^e PhZnCl:**7** = 4. ^f Yield of aniline isolated as benzanilide. ^g Yield of aniline purified by distillation. ^h 20 mol% MgCl₂ was added.

amination of phenylzinc chloride with a 4:1 molar ratio of **1a**:**7** did not give a comparable yield with **3a**. The use of dimethylphenylzincate **6a** instead of triphenylzincate was not found to be more successful for amination. These observations seem identical with the fact that tetracoordinated zincates prepared *in situ* from triorganozincates and anion species such as CN have higher reactivity compared with the conventional triorganozincates.²⁵

As seen, higher yields are obtained in the amination of Grignard reagent-derived organozincs rather than lithium reagent-derived organozincs (entries 3 and 12; 6 and 13; 9 and 14), supporting the role of MgCl₂ as a Lewis acid in electrophilic amination of carbanions with synthetic equivalents of the NH₂⁺ synthon.^{6,26} In fact, CuCN-catalyzed amination of lithium triphenylzincate with **7** led to a higher yield in the presence of MgCl₂ (20 mol%) (entries 9 and 15). Amination of stoichiometric zinc diphenylcuprate **4a** and zinc phenylcyanocuprate **5a** did not give higher yields than amination of CuCN-mediated zincates **3c** and even phenylzinc chloride **1a**.

Applying the same procedure to the amination of phenylzinc reagents with **8**, diphenylzinc **2a** afforded a surprisingly higher yield compared with the amination with **7** (entry 6) while amination of phenylzinc chloride **1a** did not take place. Lithium triphenylzincate **3a** was aminated almost in quantitative yield (entry 9).

In an effort to determine the most effective phenylmetal in the amination with **7** and **8**, we examined the relevant literature data and we also carried out amination of phenyl-lithium and -copper reagents with **7** in addition to our amination studies of phenyl Grignard reagents with **7** (Table 2). For comparison, only lithium phenylcyanocuprate (entry 8) gives a yield comparable with that of the lithium triphenylzincate/CuCN system, but not as high as the yield of bromomagnesium triphenyl-

Table 2 Amination reactions of phenylmetals with **7** and **8**

PhM + 7 or 8		1. THF, rt, 3 h 2. Hydrolysis PhNH ₂ 9	
Entry	M	Yield of PhNH ₂	
		Aminating reagent	
		7 ^a	8 ^{b,c}
1	Li	6 ^d	91
2	Li ^f /CuI ^g	22 ^e	
3	Li ^f /CuI ^g	46 ^e	
4	MgBr	58 ^d	37
5	MgBr ^f /CuI ^g	59 ^{d,f}	
6	Cu	56 ^e	
7	1/2 CuLi	43 ^e	83
8	Cu(CN)Li	76 ^e	
9	1/3 CuLi ₂	39 ^e	
10	1/2 Cu(CN)Li ₂	35 ^e	
11	ZnCl ^f /CuCN ^g	58 ^g (65 ^h)	7 ^a
12	1/2 Zn ^f /CuCN ^g	44 ^g (68 ^h)	82
13	1/3 ZnM ^f /CuCN ^g (M = Li, MgBr)	70 ^g (85 ^h)	95 ^g
14	1/2 CuZnCl	49 ^g	
15	Cu(CN)ZnCl	56 ^g	

^a GC yield. ^b Aminating reagent is **8**-methylithium complex (refs. 9 and 22). ^c Yield of benzanilide. ^d Ref. 6a. ^e Amination of phenyllithium (2 mol equiv.), phenylcopper (2 mol equiv.), lower- and higher-order homocuprates (2 mol equiv.) and lower- and higher-order cyanocuprates (1 mol equiv.) with **7** (1 mol equiv.) was carried out in diethyl ether at room temperature similar to amination of organozinc reagents as explained in the Experimental section. ^f Ref. 6a. The reaction time decreases in CuI-mediated reaction. ^g Organozinc reagent was prepared by Li-to-Zn metallation (Table 1, entries 3, 6, 9). ^h Organozinc reagent was prepared by Mg-to-Zn metallation (Table 1, entries 12–14).

zincate/CuCN (entry 13). Phenylmagnesium bromide/CuI (entry 5) is not as effective as bromomagnesium triphenylzincate/CuCN (entry 13). Concerning the available data for the amination with **8**, the most effective phenylmetals are phenyllithium (entry 1) and lithium triphenylzincate/CuCN (entry 13). Lithium diphenylcuprate and diphenylzinc/CuCN can also be aminated in comparable high yields (entries 7 and 12, respectively). From these results, it appears that transmetallation of organolithiums to triorganozincates instead of organocyanocuprates seems to provide an alternative method in the amination with **7**. However, transmetallation of Grignard reagents to organozincates rather than organocuprates seems to be the method of choice in the amination with either **7** or **8**.

Having established a viable route to aminate organozincs with acetone *O*-(2,4,6-trimethylphenylsulfonyl)oxime **7** and with *O*-methylhydroxylamine **8**; the scope and generality of the amination protocol was examined for a series of organozincs (Table 3). In amination with **7**, yields of monoorganozincs **1** were tabulated in addition to diorganozincs **2** and triorganozincates **3** since **1** and **2** are expected to give comparable yields with **3**; however, in amination with **8**, yields of only **2** and **3** were reported, except for **1a** and **1e**. These data show that amination proceeds with medium yields for aryl, alkyl, cycloalkyl and benzyl zinc reagents. However, to some degree, there are differences between the effectiveness of organozincs and aminating reagents. For example, amination of *p*-ethoxycarbonylphenylzinc bromide **1e** with **8** resulted in a very low yield (entry 13); and *n*-hexylzinc reagents **1f**, **2f**, **3f** could not be aminated with **7** (entries 14–16). The mildness of the conditions involved allow a certain tolerance for electron-rich functional groups on arylzincs; however, attempts to aminate functional-group-containing alkylzincs, for example, 4-chlorobutylzinc reagents failed and products possibly formed after amination of organozinc species were not identified.

work-up after amination with **7**, the mixture was stirred at room temperature with 12 M HCl (10 cm³) for 2 h. The aqueous phase was washed with diethyl ether, made basic with conc. NaOH and the free amine was extracted with diethyl ether (4 × 30 cm³). The organic layer was dried over Na₂SO₄. The crude material obtained after evaporation of solvent *in vacuo* was purified by distillation or flash chromatography on silica gel with diethyl ether–light petroleum (5:4) to give pure amine **9** whose GC and spectral data matched those of authentic material. After amination with **8**, hydrolysis of the mixture was carried out with saturated aq. NH₄Cl–NH₃ (5:1) (10 cm³). The aqueous phase was extracted with diethyl ether and the crude material was purified with column chromatography to give the amine as above. For convenience in isolation, the product amines were converted to their *N*-benzoyl derivatives by reaction with benzoyl chloride in the presence of NaOH.³⁵ Crude derivatives were recrystallized from ethanol or ethanol–water as white solids whose mp³⁶ matched that of previously synthesized material.

N-Phenylbenzamide **9a**: mp 162–164 °C (lit.,^{22,36} 160 °C); δ_H^{22,37} 7.88–7.92 (2H, m, NArH), 7.65–7.69 (2H, m, COArH), 7.48–7.62 (3H, m, NArH), 7.16–7.43 (3H, m, COArH); ν_{max}/cm⁻¹ 3345, 1655, 1599, 1531, 1439, 1250, 1070, 755, 715, 698.

N-*p*-Tolylbenzamide **9b**: mp 158 °C (lit.,^{22,36} 158 °C); δ_H^{22,37} 7.85–7.92 (2H, m, NArH), 7.52–7.58 (2H, m, COArH), 7.44–7.50 (3H, m, COArH), 7.15–7.20 (2H, m, NArH), 2.30 (s, ArCH₃); ν_{max}/cm⁻¹ 3308, 1647, 1599, 1531, 1404, 1265, 1024, 813, 697.

N-(*p*-Methoxyphenyl)benzamide **9c**: mp 154–157 °C (lit.,³⁶ 154 °C); δ_H 7.85–7.90 (m, 2H, NArH), 7.78–7.82 (1H, br s, NH), 7.52–7.60 (2H, m, COArH), 7.45–7.52 (3H, m, COArH), 6.90–6.96 (m, 2H, NArH), 3.82 (s, OCH₃); ν_{max}/cm⁻¹ 3329, 2850, 1647, 1519, 1412, 1250, 1032, 820, 654.

N-(*p*-Bromophenyl)benzamide **9d**: mp 203 °C (lit.,³⁶ 204 °C); δ_H 7.86–7.90 (2H, m, NArH), 7.78–7.87 (1H, br s, NH), 7.58–7.60 (2H, m, COArH), 7.48–7.55 (2H, m, COArH), 7.27–7.30 (2H, m, NArH); ν_{max}/cm⁻¹ 3332, 1647, 1578, 1522, 1491, 1385, 1250, 1070, 1005, 820, 715, 654, 508.

N-(*p*-Ethoxycarbonylphenyl)benzamide **9e**: mp 150 °C (lit.,³⁶ 148 °C); δ_H 7.96–8.07 (2H, m, NArH), 7.86–7.90 (2H, m, NArH), 7.30–7.60 (5H, m, COArH), 7.37 (2H, q, CO₂CH₂CH₃), 1.39 (3H, t, CO₂CH₂CH₃); ν_{max}/cm⁻¹ 3360, 1710, 1605, 1452, 1310, 1270, 1115, 712, 687, 650.

N-Hexylbenzamide **9f**: mp 40 °C (lit.,³⁶ 40 °C); δ_H 7.74–7.81 (2H, m, COArH), 7.36–7.48 (3H, m, COArH), 6.30–6.60 (1H, br s, NH), 3.38–3.45 (2H, m, NCH₂), 1.55–1.65 (2H, m, NCH₂CH₂), 1.25–1.40 (6H, m, NCH₂CH₂CH₂CH₂CH₂), 0.85–0.95 (3H, t, CH₂CH₃); ν_{max}/cm⁻¹ 3341, 2925, 2855, 1631, 1570, 1530, 1485, 1275, 1155, 1070, 718, 687.

N-Cyclohexylbenzamide **9g**: mp 149 °C (lit.,³⁶ 149 °C); δ_H 7.74–7.78 (2H, m, COArH), 7.38–7.50 (3H, m, COArH), 6.10–6.20 (1H, br s, NH), 3.84–4.05 (1H, br s, NCH), 1.72–2.10 (4H, m, 2-H and 6-H₂), 1.28–1.50 (6H, m, 3-, 4- and 5-H₂); ν_{max}/cm⁻¹ 3312, 2932, 2855, 1620, 1578, 1535, 1480, 1152, 1080, 690.

N-Benzylbenzamide **9h**: mp 106 °C (lit.,³⁶ 105 °C); δ_H³⁷ 7.76–7.84 (2H, m, COArH), 7.40–7.55 (3H, m, COArH), 7.32–7.40 (5H, m, NCH₂ArH), 6.50–6.52 (1H, br s, NH), 4.62–4.68 (2H, s, NCH₂); ν_{max}/cm⁻¹ 3287, 1637, 1605, 1552, 1415, 1261, 1070, 980, 785, 697.

Acknowledgements

We are grateful to the Turkish Scientific and Technical Research Council (Grant No. TBAG-1618), the Ankara University Research Fund (Grant No. 96250026) and NATO (Research Grant No. CRG971168) for financial support.

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