Improved catalytic procedures for the copper(I)-promoted reactions of β-amido zinc reagents

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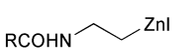
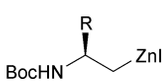
Copper-catalysed cross coupling of the β-aminoalkylzinc reagents **1a** and **2** with unsaturated alkyl halides gives β-unsaturated ethylamines in 47–78% yield (4 examples) and enantiomerically pure β-unsaturated propylamines in 55–69% yield (3 examples). This method is more efficient for simple β-aminoalkylzinc reagents than that using stoichiometric copper.

Zinc organometallics are important nucleophilic reagents having considerable synthetic potential.[1,2] The improved stability of β- and γ-amino acid-derived organozinc reagents in DMF,[3] and the fact that Knochel and co-workers have prepared the β-benzamido organozinc reagent **1b**, and the corresponding zinc–copper reagent, in a DMSO–THF solvent mixture,[4] led us to develop the β-amido organozinc reagent **1a** and similarly the enantiomerically pure analogues **3** and **4**.[4]

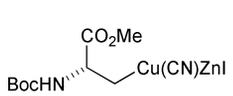


1a, R = ^{t}BuO
1b, R = Ph



2, R = Me
3, R = ^{i}Pr
4, R = Bn

Subsequent reaction of these reagents with substituted aryl iodides under palladium catalysis gave the corresponding arylated products in moderate to good yields.[5] We wished to explore further the potential of the simple β-amido zinc reagent **1a** and the previously unreported organozinc reagent **2** in the context of copper-mediated cross coupling processes. The extensive work by Knochel and co-workers in the area of zinc–copper reagents has led us to develop the β-amido zinc–copper reagents **5**, **6** and **7**, derived from serine, aspartic acid and glutamic acid, respectively. Reaction of these reagents with a range of electrophiles allowed the synthesis of a variety of enantiomerically pure unsaturated α-, β- and γ-amino acids, not available from the corresponding zinc reagents.[6,7]



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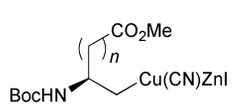
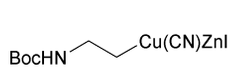


6, *n* = 1
7, *n* = 2

Our previous efforts to prepare simple analogues of the zinc–copper reagents **5**–**7** lacking the ester function, using THF as solvent, were frustrated by low yields. These poor yields appeared to be a reflection of the instability of the corresponding zinc–copper reagents in THF. It was clearly important to define conditions in which analogues with more easily deprotected *N*-protecting groups could be employed. We now report conditions that allow the use of the simple zinc–copper reagents **8** and **9** in an effective and reliable manner.



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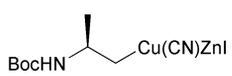

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Results and discussion

The necessary alkyl iodide precursors **10** and **11** were prepared in two simple steps from 2-aminoethanol and (*S*)-alaninol using the general methods previously described.[5]

Reactivity of zinc–copper reagents 8 and 9

The organozinc reagent **1a** was generated from the iodide **10** using activated zinc dust in DMF. An equimolar amount of a DMF solution of CuCN·2LiCl[8] was added to a solution of **1a** at −55 °C. The reaction was then allowed to warm briefly to 0 °C to ensure formation of the cuprate, whereupon it was re-cooled to −55 °C and allyl bromide added (Scheme 1).

Standard work-up and purification *via* flash chromatography gave the allylated compound **12** in moderate yield (33%). The mass balance was accounted for by protonated zinc reagent and $BocNH_2$, a degradation product formed *via* β-elimination of the carbamate protecting group. Analogous treatment of iodide **11** gave the compound **16** in similar yield (36%). A representative selection of unsaturated alkyl halide electrophiles was employed and moderate yields of coupled products were obtained in most cases (Table 1). While these results were a significant improvement on our previous work, the instability of the zinc–copper reagents **8** and **9** was clearly having a deleterious effect on the efficiency of the process. In an attempt to address this issue, we therefore turned to the catalytic use of copper.

Copper-catalysed cross coupling of zinc reagents 1a and 2

Hiemstra and co-workers recently reported that it is possible to couple a pyroglutamic acid-derived β-amido organozinc

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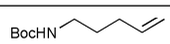
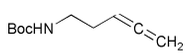
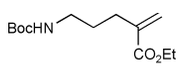
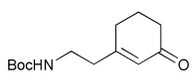
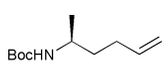
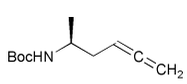
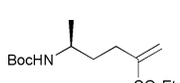
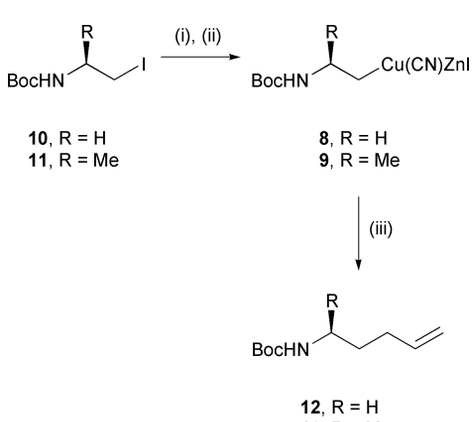
Table 1 Preparation of compounds **12**–**18** using stoichiometric or catalytic copper

Zn reagent	Electrophile	Product	Yield (%)[a] Stoichiometric CuCN·2LiCl	Catalytic CuBr·DMS
1a	Allyl bromide	 **12**	33	51
1a	Propargyl chloride	 **13**	30	63
1a	Ethyl 2-bromomethylacrylate	 **14**	60	78
1a	3-Iodocyclohex-2-enone	 **15**	38	47
2	Allyl bromide	 **16**	36	55
2	Propargyl chloride	 **17**	29	60
2	Ethyl 2-bromomethylacrylate	 **18**	59	69

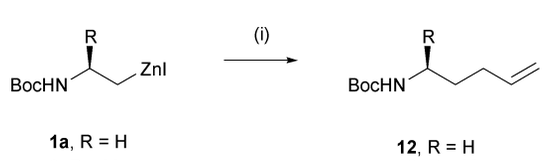
[a] Based on starting iodide **10** or **11**.



Scheme 1 *Reagents and conditions*: i, Zn* (prepared from Zn dust using Me_3SiCl, in DMF), 5–15 min, 0 °C; ii, CuCN·2LiCl in DMF, 5 min, −55 to 0 °C; iii, allyl bromide (1.33 equiv.), −55 °C to room temperature, 14 h.

reagent with propargylic‡ tosylates in good yield provided a catalytic quantity of $CuBr{\cdot}SMe_2$ was used in the reaction,[9] a process we have recently applied to amino acid synthesis.[10] In the latter case, the use of catalytic copper gave good results, albeit slightly inferior to those obtained using stoichiometric CuCN·2LiCl.

The organozinc reagent **1a** was generated as described above. The excess zinc dust was allowed to settle and the supernatant was then removed by syringe and added to a pre-mixed DMF solution of $CuBr{\cdot}SMe_2$ (5 mol%) and allyl bromide at −10 °C (Scheme 2). After subsequent purification the allylated compound was isolated in a much improved 51% yield, which was obtained consistently on a 0.75 mmol scale. Reaction of zinc reagents **1a** and **2** with the other electrophiles in the presence of catalytic $CuBr{\cdot}SMe_2$ brought about increased yields in all cases compared to the use of stoichiometric CuCN·2LiCl (Table 1). The work-up for reactions using catalytic amounts of copper is substantially more straightforward, especially given the need for special precautions for the disposal of aqueous cyanide waste.



Scheme 2 *Reagents and conditions*: i, allyl bromide and $CuBr{\cdot}SMe_2$ (5 mol%) in DMF, −10 °C to room temperature, 14 h.

The main decomposition pathway for reagents such as **8** and **9** involves a β-elimination process, which leads to ethene and propene respectively. It is likely that the higher yields using catalytic amounts of copper simply reflect the greater stability of the organozinc reagents **1a** and **2**, compared with the zinc–copper reagents **8** and **9**.

The enantiomeric excess of the representative product **18** was established as greater than 98% by preparation of a racemic sample, followed by chiral phase HPLC analysis. This established that no significant racemisation had occurred during the reaction of zinc reagent **2** with ethyl 2-bromomethylacrylate.

Conclusions

We have shown that the use of a catalytic quantity of

‡ The IUPAC name for propargyl is prop-2-ynyl.

$CuBr{\cdot}SMe_2$ instead of a stoichiometric amount of $CuCN{\cdot}2LiCl$ is beneficial in promoting the reactions of the β-amido zinc reagents **1a** and **2** with unsaturated electrophiles. In addition, we have provided evidence that for the zinc reagent **2**, complete retention of stereochemical integrity occurs during the coupling process.

Experimental

Dry DMF was distilled from calcium hydride and stored over 4 Å molecular sieves. Dry dichloromethane was distilled from calcium hydride. Dry THF was distilled from potassium–benzophenone ketyl. Petroleum ether refers to the fraction with a boiling point between 40 and 60 °C. Specific rotations were measured at 20 °C, unless otherwise stated and values are given in 10^{-1} deg cm^2 g^{-1}. ^1H NMR spectra were recorded in $CDCl_3$ solvent at 500 MHz, referenced to TMS. ^{13}C NMR spectra were recorded in $CDCl_3$ at 125 MHz and referenced to TMS. Chemical shifts are given in ppm. Coupling constants are given in hertz. Organic extracts were dried over $MgSO_4$ and the solvent removed on a rotary evaporator. The preparation of iodide **10** has been described,[5] and iodide **11** was prepared by the same method.

(2*S*)-*N*-*tert*-Butoxycarbonyl-2-amino-1-iodopropane 11 [11]

Isolated as a white solid, (2*S*)-*N*-*tert*-butoxycarbonyl-2-amino-1-iodopropane **11** was recrystallised from petroleum ether–ethyl acetate (9.21 g, 60%). Mp 60–62 °C (lit.[11] 58–59 °C) (Found M^+ 285.0211; $C_8H_{16}NO_2I$ requires 285.0226); $[a]_D^{14}$ −15.3 (*c* 1.00 in CH_2Cl_2) (Found: C, 33.8; H, 5.6; N, 4.8%; $C_8H_{16}NO_2I$ requires C, 33.7; H, 5.7; N, 4.9%); IR (KBr disc)/cm^{-1} 3285, 2976, 1678, 1533, and 1172; NMR δ_H 1.20 (3H, d, *J* 7, C(3)H_3), 1.45 (9H, s, C(CH_3)$_3$), 3.30 (1H, dd, *J* 10, 4, C(1)*H*), 3.38–3.43 (1H, m, C(1)*H′*), 3.50–3.56 (1H, m, C(2)*H*), 4.60–4.66 (1H, br s, N*H*); δ_C 16.02 (CH_3), 21.12 (CH_2), 28.52 (CH_3), 45.89 (CH), 79.67 (quat.), and 154.79 (CO); *m*/*z* (EI) 285 (M^+, 11%), 144 (61), 102 (32), 88 (15), and 57 (33).

A racemic sample *rac*-**11** of the above material was prepared in an identical manner. Compound *rac*-**11** exhibited identical spectroscopic data to the enantiomerically pure sample **11** but had melting point of 53–54 °C.

Generation of zinc reagents 1a and 2. General procedure

Zinc dust (325 mesh, 0.147 g, 2.25 mmol, 3.0 equivalents) was weighed into a 50 mL round-bottom flask with side arm, which was repeatedly evacuated (with heating using a hot air gun) and flushed with nitrogen. Dry DMF (0.5 mL) and trimethylsilyl chloride (6 μL, 0.046 mmol) were added, and the resultant mixture was stirred for 30 min at room temperature. The iodide **1a** or **2** (0.75 mmol) was dissolved in dry DMF (0.5 mL) under nitrogen. The iodide solution was transferred by syringe to the zinc suspension and stirred at 0 °C. TLC analysis (petroleum ether–ethyl acetate, 2 : 1) showed complete consumption of the iodide within 5–15 min.

Method (i): preparation of β-unsaturated ethylamines and propylamines *via* zinc–copper reagents 8 and 9

The pre-formed zinc reagent (**1a** or **2**) was cooled to −55 °C (cryostat temperature). A solution of $CuCN{\cdot}2LiCl$, prepared by dissolving copper(I) cyanide (0.067 g, 0.75 mmol) and vacuum-dried (at 180 °C, for 4 h) lithium chloride (0.064 g, 1.50 mmol) in dry DMF (0.5 mL), was transferred *via* syringe to the reaction mixture, which was then allowed to warm to 0 °C for 5 min. After re-cooling to −55 °C, the electrophile (1.00 mmol) was introduced, and then the mixture was stirred at this temperature for 4 h, then allowed to warm slowly to room temperature and stirred for a further 10 h. The reaction mixture was partitioned between ethyl acetate (30 mL) and saturated aqueous ammonium chloride (20 mL) and then filtered. The organic layer was washed with water (20 mL) and brine (20 mL) and then dried, and the solvent removed at reduced pressure. Flash column chromatography on silica gel, eluting with an appropriate petroleum ether–ethyl acetate gradient yielded the unsaturated products **12**–**15** and **16**–**18**.

Method (ii): preparation of β-unsaturated ethylamines and propylamines *via* $CuBr{\cdot}SMe_2$ catalysis

The organozinc reagent (**1a** or **2**) was prepared as described above. The excess zinc dust was allowed to settle for 5 minutes at 0 °C then the supernatant was transferred under nitrogen by syringe to a pre-mixed solution of $CuBr{\cdot}SMe_2$ (0.010 g, 5 mol%), and the electrophile (1.00 mmol, 1.3 equiv.) in DMF (0.5 mL) at −10 °C (ice–salt). The solution was then allowed to warm slowly to room temperature and stirred for a further 14 h. The reaction mixture was diluted with ethyl acetate (30 mL) and washed successively with water (20 mL) and brine (20 mL), dried and evaporated to dryness. Flash column chromatography over silica with an appropriate petroleum ether–ethyl acetate gradient furnished the products **12**–**15** and **16**–**18**.

***N*-Pent-4-enylcarbamic acid *tert*-butyl ester 12.** Treatment with allyl bromide (1.0 mmol) yielded pent-4-enylcarbamic acid *tert*-butyl ester **12** (0.071 g, 51%) isolated as a colourless oil (Found M^+ 185.1410; $C_{10}H_{19}NO_2$ requires 185.1416); IR (KBr disc)/cm^{-1} 3350, 2978, 1691, and 1641; NMR δ_H 1.44 (9H, s, C(CH_3)$_3$), 1.55–1.61 (2H, m, C(2)H_2), 2.06–2.11 (2H, m, C(3)H_2), 3.13 (2H, q, *J* 7, C(1)H_2), 4.54–4.58 (1H, br s, N*H*), 4.96–5.05 (2H, m, C(5)*H* and C(5)*H′*), 5.75–5.84 (1H, m, C(4)*H*); δ_C 28.43 (CH_3), 29.24 (CH_2), 30.44 (CH_2), 40.10 (CH_2), 79.09 (quat.), 115.09 (C(5)H_2), 137.86 (C(4)H_2), and 155.96 (CO); *m*/*z* (EI) 183 (M^+, 64%), 110 (88), 57 (100), and 53 (12).

***N*-(*tert*-Butoxycarbonyl)penta-3,4-dienylamine 13.** Treatment with propargyl chloride (1.0 mmol) yielded *N*-(*tert*-butoxycarbonyl)penta-3,4-dienylamine **13** (0.086 g, 63%) isolated as a colourless oil (Found M^+ 183.1260; $C_{10}H_{17}NO_2$ requires 183.1259); IR (KBr disc)/cm^{-1} 3347, 2954, and 1705; NMR δ_H 1.44 (9H, s, C(CH_3)$_3$), 2.15–2.22 (2H, m, C(2)H_2), 3.22 (2H, d, *J* 6, C(1)H_2), 4.64–4.69 (1H, br s, N*H*), 4.70–4.74 (2H, m, C(5)*H* and C(5)*H′*), 5.05–5.11 (1H, m, C(4)*H*); δ_C 28.38 (CH_3), 28.80 (CH_2), 39.81 (CH_2), 75.34 (C(5)), 79.17 (quat.), 87.09 (C(3)), 155.84 (CO), and 208.90 (C(4)); *m*/*z* (EI) 183 (M^+, 22%), 110 (88), 71 (42), and 57 (100).

Ethyl (5-*N*-*tert*-butoxycarbonylamino)-2-methylenepentanoate 14. Treatment with ethyl 2-bromomethylacrylate (1.0 mmol) yielded ethyl (5-*N*-*tert*-butoxycarbonylamino)-2-methylenepentanoate **14** (0.151 g, 78%), isolated as a colourless oil (Found MH^+ 258.1712; $C_{13}H_{23}NO_4$ requires 258.1705); IR (KBr disc)/cm^{-1} 3376, 2978, 1731, and 1631; NMR δ_H 1.31 (3H, t, *J* 7, $CO_2CH_2CH_3$), 1.44 (9H, s, C(CH_3)$_3$), 1.64–1.70 (2H, m, C(4)H_2), 2.33 (2H, t, *J* 7.5, C(3)H_2), 3.15 (2H, q, *J* 6, C(5)H_2), 4.21 (2H, q, *J* 7, $CO_2CH_2CH_3$), 4.65–4.70 (1H, br s, N*H*), 5.56 (1H, d, *J* 1, C methylene *H*), 6.17 (1H, d, *J* 1, C methylene *H′*); δ_C 14.21 (CH_3), 28.44 (CH_3), 28.96 (CH_2), 29.06 (CH_2), 39.96 (CH_2), 60.70 (CH_2), 79.04 (quat.), 125.09 (C methylene), 140.08 (C(2)H_2), 156.01 (carbamate), and 167.15 (ester); *m*/*z* (EI) 258 (MH^+, 26%), 202 (51), 184 (5), 112 (9), 85 (6), and 57 (100).

3-(2′-*N*-*tert*-Butoxycarbonylaminoethyl)cyclohex-2-en-1-one 15. Treatment with 3-iodocyclohex-2-enone (1.0 mmol) yielded 3-(2′-*N*-*tert*-butoxycarbonylaminoethyl)cyclohex-2-en-1-one **15** (0.084, 47%), isolated as a colourless oil. IR (KBr disc)/cm^{-1} 3344, 2933, 1694, and 1667; NMR δ_H 1.42 (9H, s, C(CH_3)$_3$), 1.98–2.03 (2H, m, C(5)H_2), 2.32–2.41 (6H, m, C(1)H_2, C(4′)H_2, C(6′)H_2), 3.33 (2H, q, *J* 6, C(2)H_2), 4.56–4.62 (1H, br s, N*H*), 5.87 (1H, s, C(3′)*H*); δ_C 22.72 (CH_2), 28.43 (CH_3), 29.44, 37.34,

37.95, 38.81, 79.64 (quat.), 127.24, 155.56 (CO), 162.96, and 199.69; *m*/*z* (EI) 74 (13%), and 57 (100).

(2*S*)-*N*-(Hex-5-en-2-yl)carbamic acid *tert*-butyl ester 16.[12] Treatment with allyl bromide (1.0 mmol) yielded (2*S*)-*N*-(hex-5-en-2-yl)carbamic acid *tert*-butyl ester **16** (0.082 g, 51%), isolated as a colourless oil (Found $M^+ - CH_3$ 184.1388; $C_{10}H_{18}NO_2$ requires 184.1337); $[\alpha]_D^{23}$ 1.8 (*c* 2.65 in CH_2Cl_2) (Found: C, 66.1; H, 11.0; N, 7.2%; $C_{11}H_{21}NO_2$ requires C, 66.3; H, 10.6; N, 7.0%); IR (KBr disc)/cm^{-1} 3339, 2977, 1686, and 1641; NMR δ_H 1.13 (3H, d, *J* 7, C(1)H_3), 1.45 (9H, s, C(CH_3)$_3$), 1.46–1.52 (2H, m, C(3)H_2), 2.05–2.11 (2H, m, C(4)H_2), 3.62–3.71 (1H, m, C(2)*H*), 4.25–4.32 (1H, br s, N*H*), 4.93–5.05 (2H, m, C(6)*H* and C(6)*H*′), 5.76–5.83 (1H, m, C(5)*H*); δ_C 21.47 (CH_3), 28.68 (CH_3), 30.57 (CH_2), 36.76 (CH_2), 46.39 (CH), 79.26 (quat.), 115.02, 138.36, and 155.19 (CO); *m*/*z* (EI) 157 ($M^+ - C_3H_6$, 32%), 144 (87), 142 (67), 126 (5), and 57 (100).

(2*S*)-*N*-(*tert*-Butoxycarbonyl)hexa-4,5-dien-2-ylamine 17. Treatment with propargyl chloride yielded (2*S*)-*N*-(*tert*-butoxycarbonyl)hexa-4,5-dien-2-ylamine **17** (0.089 g, 60%), isolated as a colourless oil (Found MH^+ 198.1489; $C_{11}H_{20}NO_2$ requires 198.1494); $[\alpha]_D^{25}$ 7.2 (*c* 0.970 in CH_2Cl_2); IR (KBr disc)/cm^{-1} 3364, 2977, and 1689; NMR δ_H 1.15 (3H, d, *J* 7, C(1)H_3), 1.45 (9H, s, C(CH_3)$_3$), 2.12–2.19 (2H, m, C(3)H_2), 3.70–3.77 (1H, br s, C(2)*H*), 4.41–4.48 (1H, br s, N*H*), 4.49–4.70 (2H, m, C(6)H_2), and 5.02–5.08 (1H, m, C(4)H_2); δ_C 20.42 (CH_3), 28.39 (CH_3), 35.96 (CH_2), 46.30 (CH_2), 79.06 (quat.), 85.93, 86.70, 155.23 (CO), and 209.56 (C(5)); *m*/*z* (CI, methane) 198 (M^+, 8%), 142 (27), 98 (100), and 57 (13).

Ethyl (5*S*)-5-(*N*-*tert*-butoxycarbonylamino)-2-methylenehexanoate 18. Treatment with ethyl 2-bromomethylacrylate (1 mmol) yielded ethyl (5*S*)-5-(*N*-*tert*-butoxycarbonylamino)-2-methylenehexanoate **18** (0.140 g, 69%) isolated as a colourless oil (Found MH^+ 272.2577; $C_{14}H_{26}NO_4$ requires 272.1861); $[\alpha]_D^{26}$ −5.5 (*c* 2.1 in $CHCl_3$); IR (KBr disc)/cm^{-1} 3368, 2977, 1716, and 1631; NMR δ_H 1.14 (3H, d, *J* 7, C(6)H_3), 1.31 (3H, t, *J* 7, $CO_2CH_2CH_3$), 1.45 (9H, s, C(CH_3)$_3$), 1.55–1.60 (2H, m, C(4)H_2), 2.27–2.41 (2H, m, C(3)H_2), 3.64–3.72 (1H, m, C(5)*H*), 4.20 (2H, q, *J* 7, $CO_2CH_2CH_3$), 4.37–4.42 (1H, br s, N*H*), 5.56 (1H, s, C methylene *H*), 6.15 (1H, s, C methylene *H*′); δ_C 14.21 (CH_3), 21.33 (CH_3), 28.43 (CH_3), 28.58 (CH_2), 36.05 (CH_2), 46.19 (CH_2), 60.65 (CH), 79.00 (quat.), 124.87, 140.29, 155.39 (carbamate), 167.13 (ester); *m*/*z* (EI) 272 (MH^+, 9%), 216 (17), 198 (7), 172 (47), 155 (13), 144 (4), 110 (7), 88 (7), 81 (9), and 57 (100).

A racemic sample *rac*-**18** of the above material was prepared in an identical manner using *rac*-**11** as the starting material. Compound *rac*-**18** exhibited identical spectroscopic data to the enantiomerically pure sample **18**. The racemic sample was analysed by chiral phase HPLC (Chiralpak AD, eluent 98 : 2 hexane–ethanol, flow rate 1 ml min^{-1}, detection at 215 nm), which gave baseline enantiomer separation. Analysis of **18** indicated an enantiomeric excess of 98%.

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