β-Lithiations of Carboxamides. Synthesis of β-Substituted-α,β-Unsaturated Amides

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Dedicated to the memory of Roland K. Robins

N-Methyl-2-methyl-3-(benzotriazol-1-yl)propanamide, on treatment with butyllithium forms a dianion which on treatment with alkyl and benzyl halides, aldehydes and ketones affords monosubstituted products; with ethyl *p*-toluate, a lactam is formed. The alkylated derivatives eliminate benzotriazole in the presence of base to afford trisubstituted α,β-unsaturated amides.

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Introduction.

Synthetic strategies involving carbon-carbon bonds arising from protonation followed by reaction of the resulting carbanion with an electrophile are widely employed in organic syntheses. There are many factors that govern the regio- and stereo-selectivities and specificities. Thermodynamically favored carbanions and more recently, kinetic deprotonations are known and afford approaches to a variety of novel synthetic routes [1,2].

Tertiary amides normally undergo α-deprotonation in the presence of base to afford enolates. However, the rarer β-deprotonations are also known to occur when the β-position is activated by a phenyl, phenylthio, phenylsulfonyl or a vinyl group [1,3,4]. This β-deprotonation affords a homoenolate carbanion that is usually available in a masked form or from a β-halocarbonyl precursor [5]. These β-lithio α-amides can be thought of as a β-lithio α-alkyl carboxylic acid synthons. Some of the more common approaches to them involve metal insertion into a carbon-halogen bond [6]; use of trimethyltin in the presence of a Lewis acid [7]; the use of β-lithio acetals [8] or of dilithiated amides [9]. Another method for the generation of a homoenolate dianion of a secondary amide involves tin/lithium exchange used by Goswami and Corcoran [10,11] to generate β-substituted amides in good yields. The dianions obtained from *N*-monosubstituted-3-(phenylsulfonyl)propanamides react smoothly with aldehydes and ketones to afford γ-hydroxy amides, which cyclize to furanones [4].

Studies on β-homoenolate anions have also involved unsaturated systems. Watt *et al.* [12] have used β-haloacrylate orthoesters as unsaturated homoenolate equivalents, while Funk and Bolten [13] have used 4*H*-1,3-dioxin as a β-acyl vinyl anion equivalent. We refer later to the work of Tanaka *et al.* [14] with 2-(phenylsulfonylmethyl)propenamide. The use of β-(tri-*n*-butylstannyl)acrylamide as a β-lithioacrylamide synthon has been demonstrated by Quayle and coworkers [15]: transmetallation with butyllithium affords dianions which are then treated with carbonyl compounds to give, depending upon the workup conditions, either hydroxyamides or butenolides. Nájera and Yus [16] have used (*E*)-*N*-isopropyl-3-tosylacrylamide as their β-acyl vinyl anion equivalent and have also generated butenolides. Similarly, the lithio derivative of 2-bromo-1-cyclohexenecarboxamide reacts with carbonyl compounds to afford bicyclic $\Delta^{\alpha,\beta}$-butenolides [17]. Other examples of 3-carbon (d^3) synthons in unsaturated systems include β-alkyl- [18], β-bromo- [19], β-thiophenoxy- [20], and β-pyrrolidino- [21,22] acrylic acid derivatives.

Work in our laboratory has amply demonstrated the versatility of benzotriazole in various synthetic transformations [23]. It has a strong electron withdrawing nature which enables α-deprotonations in a variety of systems. Depending upon the environment, it can be displaced readily by acid, base, nucleophiles *etc.* Consequently, when appropriately positioned, it should assist in the β-lithiation of carboxamides and subsequent transformations, and then when required, be removed.

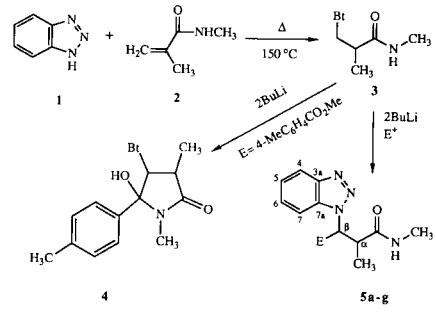
Results and Discussion.

Lithiation of *N*-Methyl-2-methyl-3-(benzotriazol-1-yl)propanamide (**3**) with Butyllithium and Treatment of the Dianion with Electrophiles.

Benzotriazole underwent a Michael addition with *N*-methylmethacrylamide (**2**) at 150° to afford, after column chromatography, amide **3** as colorless microcrystals (45%). The amide **3** was then treated with two equivalents of butyllithium at −78°: after addition of the first equivalent, a dark red color appeared and persisted, indicating that whilst the monolithium salt is colorless, the dianion is deeply colored. Methyl iodide was added at −78° and the reaction mixture stirred overnight at room temperature. Workup afforded a solid, the nmr spectrum of which indicated it to be the dimethylated (*C*- and *N*-methylated)

product. On the other hand, when the workup was carried out at −78° only the *C*-methylated product **5a** was obtained (58%). Reaction with other alkyl halides and benzyl bromide afforded the corresponding *C*-alkylated products **5b-e** (56-73%) (Scheme 1). The results are summarized in Table 1. Treatment of the dianion with an equivalent of *p*-tolualdehyde or benzophenone afforded the corresponding secondary and tertiary alcohols **5g** (46%) and **5f** (56%) respectively. Treatment of the dianion of **3** with methyl *p*-toluate afforded the 5-hydroxylactam **4** (63%). Cyclizations on similar systems have been observed [4,15]. In those examples, stirring the γ-hydroxy amide overnight at room temperature after acid quench or heating it in the presence of base afforded butenolides by elimination of the amino group.

Scheme 1



The products **4** and **5a-g** were characterized by their ^1H and ^{13}C nmr spectra (see Tables 2 and 3) and by their microanalyses data (Table 1). In the ^1H nmr spectrum of

Table 1

Reaction of Amide **3** with Butyllithium and Treatment with Electrophiles

Compound No.	Electrophile	E	Yield (%)	Mp (°C)	Molecular Formula	Analysis (%) Calcd. C	H	N	Found C	H	N
5a	CH_3I	CH_3	58	151-153	$C_{12}H_{16}N_4O$	62.05	6.94	24.12	62.23	7.01	24.37
5b	BuI	Bu	56	104-106	$C_{15}H_{22}N_4O$	65.67	8.08	20.42	65.49	8.09	20.30
5c	HexI	Hex	64	oil	$C_{17}H_{26}N_4O$	67.52	8.67	18.53	67.42	8.81	18.59
5d	OctI	Oct	62	oil	$C_{19}H_{30}N_4O$	69.06	9.15	16.95	69.08	9.28	16.99
5e	$PhCH_2Br$	$PhCH_2$	73	190-192	$C_{18}H_{20}N_4O$	70.11	6.54	18.17	70.22	6.62	18.23
5f	4-MeC_6H_4CHO	4-$MeC_6H_4CH(OH)$	46	208-210	$C_{19}H_{22}N_4O_2$	67.44	6.55	16.56	67.69	6.75	16.41
5g	Ph_2CO	$Ph_2C(OH)$	56	232-233	$C_{24}H_{24}N_4O_2$	71.98	6.04	13.99	72.13	6.07	14.00
4	4-$MeC_6H_4CO_2Me$		63	214-215	$C_{19}H_{20}N_4O_2$	67.84	5.99	16.65	67.78	6.00	16.75

Table 2

^1H NMR Spectra of Lithiation Products **5a-g**

Compound No.	Benzotriazole	Bt-CβH-E (m, 1H)	CαH-(CH_3) (m, 1H)	CαH-(CH_3) [a]	NH(CH_3) (m, 1H)	NH-(CH_3) [a]	E
5a	7.63 [b], 7.97 [b] 7.3-7.5 [c]	5.13	3.20	1.39 (6.9)	6.25	2.31 (3.48)	1.74 (d J = 6.8 Hz, 3H)
5b	8.00 [b], 7.58 [b] 7.3-7.5 [c]	4.90	3.17	1.39 (6.9)	5.74	2.25 (4.85)	0.76 (t, J = 7.3 Hz. 3H, 0.8-1.35 (m, 4H) 2.0-2.15 [c]
5c	7.99 [b], 7.61 [b] 7.3-7.5 [c]	4.93	3.23	1.40 (6.95)	6.12	2.26 (4.65)	0.7-1.35 (m, 11H) 2.0-2.15 [c]
5d	8.00 [b], 7.59 [b] 7.3-7.5 [c]	4.83	3.20	1.39 (6.84)	5.94	2.26 (4.83)	0.7-1.35 (m, 15H) 2.0-2.15 [c]
5e	7.8-7.9 (m, 1H) 6.75-7.30 (m, 8H)	5.08	3.40 [d]	1.55 (6.84)	5.92	2.29 (4.39)	[e]
5f	7.86 (d, J = 7.83, 1H) 7.2-7.3 [d] 6.85-6.9 (m, 4H)	5.14	3.45	1.28 (7.08)	6.48	2.59 (4.83)	5.38 (m, 1H) 5.27 (m, 1H) 2.16 (s, 3H)
5g	7.75-7.9 (m, 4H) 7.20-7.55 (m, 7H) 6.75-6.95 [d]	6.15 [f]	3.47	0.95 (7.02)	5.40	2.19 (4.83)	5.66 (s, 1H)

[a] {d, J (Hz), 3H}. [b] (d, J = 8.4 Hz, 1H). [c] (m, 2H). [d] (m, 3H). [e] Overlaps with benzotriazole signals and with CαH. [f] (1H, d, J = 8.06 Hz).

Table 3
^{13}C NMR Spectra of Lithiation Products **5a-g**

Compound	Benzotriazole											
No.	C-7	C-4	C-5	C-6	C-7a	C-3a	$C_\beta E$	C_α	$C_\alpha CH_3$	C-O	N(CH_3)	E
5a	110.2	118.9	124.2	127.2	132.9	144.6	57.0	47.2	15.3	173.9	25.8	18.3
5b	110.0	119.2	123.9	127.3	134.0	145.0	61.6	46.8	15.3	174.0	25.8	31.5, 27.7 22.1, 13.7
5c	110.0	119.3	123.9	127.3	134.0	145.0	61.6	46.9	15.3	174.0	25.9	31.8, 31.4, 28.7 25.6, 22.4, 13.9
5d	110.1	119.2	123.9	127.3	134.0	144.9	61.5	46.7	15.3	174.0	25.8	31.7, 31.6, 29.1 29.0 28.9, 25.5, 22.4, 13.9
5e	109.6	118.8	123.6	127.0 [a]	134.0	144.7	63.5	46.4	15.7	174.0	25.9	136.7, 128.6, 128.2 126.5 [a], 38.6
5f	110.1	119.0	123.88	127.2	134.0	144.6	66.7	43.0	14.9	174.3	26.3	137.5, 136.9, 128.7, 126.0, 73.9, 20.9
5g	110.6	119.4	124.1 [a]	127.2 [a]	134.1	145.4	66.9	45.1	17.3	173.6	26.0	144.3, 143.6, 128.5 127.9, 127.8, 126.5 [a] 125.1, 124.0 [a], 80.6

[a] Tentative assignments.

Table 4
Synthesis of α,β-Unsaturated Amides **6a-c, 7**

Starting Material	Product	Yield (%)	Mp (°C)	Molecular Formula	Analysis (%) Calcd.			Found		
					C	H	N	C	H	N
5b	**6a**	76	oil	$C_9H_{17}NO$	69.67	11.09	9.02	69.66	11.04	9.36
5c	**6b**	74	oil	$C_{11}H_{21}NO$	72.08	11.55	7.64	71.73	11.69	7.53
5d	**6c**	70	oil	$C_{13}H_{25}NO$	73.88	11.92	6.63	73.61	12.21	6.69
5e	**7**	63	76-77	$C_{12}H_{15}NO$	76.16	7.99	7.40	76.13	8.02	7.39

the starting **3**, the methylene protons are non-equivalent and appear as double doublets at 4.65 and 4.90 ppm *i.e.* they form along with the C*H*Me protons, an $ABXY_3$ pattern. In the products. the remaining proton on the carbon atom α to benzotriazole collapses into a multiplet and is observed slightly further downfield. For compounds **5a-g**, the *N*-methyl group was observed as a doublet between 2.2 and 2.6 ppm while for compound **4** it appeared as a singlet further corroborating our suspicions that the product was indeed a lactam. In the ^{13}C nmr spectra of the products, the C_α carbon was shifted downfield by about 6-16 ppm. For compound **4**, a signal at 80.5 ppm and the presence of only one carbonyl resonance in the ^{13}C nmr spectrum once again indicated that the acyl derivative was not formed.

Elimination of Benzotriazole. Formation of α,β-Unsaturated Amides.

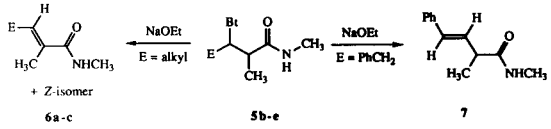
Work in our laboratory has demonstrated that base-induced elimination of benzotriazole from *N*-(α-aminoalkyl)benzotriazoles affords enamines in good yields [24]. This strategy has been extended to the synthesis of dienamines [25] and to enol ethers [26]. In this system, for both electronic and steric reasons, the proton α to the carbonyl group should be more susceptible to deprotonation than the proton α to the benzotriazole moiety. Thus, elimination of benzotriazole could afford a novel route for the synthesis of β-substituted-α,β-unsaturated amides.

Heating the butyl derivative **5b** with two equivalents of sodium ethoxide in ethanol afforded, after purification by column chromatography, the expected amide **6a** as a pale yellow oil. The ^1H nmr spectrum of **6a** displayed a doublet of triplets at 6.35 ppm and a more complex pattern between 5.4-5.6 implying that the trisubstituted olefin was obtained as a mixture of the *E*- and *Z*-isomer. The ratio of the integrals indicated a predominance of the *E*-isomer by a factor of 5:2. The *C*-methyl protons appeared as a singlet (indicating no adjacent proton) at 1.8 ppm. This was about 0.4 ppm downfield from the corresponding signal of its precursor **5b**, indicating that this methyl group was adjacent to an sp^2 carbon atom. Under similar conditions, the hexyl (**5c**) and the octyl (**5d**) derivatives afforded the corresponding trisubstituted olefins **6b,c**. As in the prev-

ious case, the ratio of the *E*- to *Z*-isomer was 5:2. In all cases, the yields of the oils were about 80% (Table 4).

With the benzyl derivative **5e** the corresponding α,β-unsaturated amide was not obtained. The ^{1}H nmr spectrum of the solid indicated the presence of two vinylic protons. Elimination had indeed occurred but it was not the proton α to the carbonyl that was abstracted by the base. It seems that in this system, the benzylic proton was more acidic and consequently abstracted affording the β,γ-unsaturated amide **7** (Scheme 2). The large coupling constant (15.8 Hz) of the doublet at 6.5 ppm indicated the product to be the *trans* isomer. The β-hydrogen appeared as a double doublet at 6.25 ppm and displayed the characteristic *trans* and *geminal* coupling pattern. The *C*-methyl protons appear as a doublet at 1.4 ppm implying a proton on an adjacent carbon atom.

Scheme 2



Conclusion.

α,β-Unsaturated amides similar to the ones described above have previously been prepared [14] from the dianion of *N*-phenyl-2-(phenylsulfonylmethyl)propenamide. Regiospecific monoalkylation α to the phenylsulfonyl group followed by stereoselective reduction of the alkylated products with sodium borohydride affords the trisubstituted olefins in good yields. The isomerization of a saturated cyanohydrin in the presence of acid to afford the α,β-unsaturated amide has been described only in preliminary format [27]. We know of no prior precedent of the conversion of a saturated amide into an α,β-unsaturated amide system. The closest analogy is the reaction of the dianion from $PhSO_2CH_2CH_2CONHR$ with carbonyl compounds. This is followed by the loss of the amino moiety and thus presents a synthesis of butenolides [4].

EXPERIMENTAL

Melting points were determined on a Bristoline hot-stage microscope and are uncorrected. The ^{1}H (300 MHz) nmr spectra were recorded on a Varian VXR-300 (FT mode) spectrometer with TMS as the internal standard. The ^{13}C nmr spectra were recorded at 75 MHz on the same instrument using the solvent peak (deuteriochloroform, δ 77.0) as reference. High Resolution Mass Spectrometry was carried out on a Finnigan Mat 95. Elemental analyses (C,H,N) were carried out using a Carlo Erba 1106 elemental analyzer. Flash chromatography was run on EM Science silica gel (230-400 mesh). *N*-Methyl methacrylamide was purchased from Monomer-Polymer Laboratories (MPL) and used without further purification.

N-Methyl-2-methyl-3-(benzotriazol-1-yl)propanamide (**3**).

A mixture of benzotriazole (11.9 g, 0.1 mole) and *N*-methylmethacrylamide (10 ml, 0.1 mole) was heated at 150° for 6 days. The reaction mixture was dissolved in a small amount of chloroform and purified by column chromatography (diethyl ether) to afford the pure 1-substituted isomer **3** as colorless microcrystals, 10 g (45%), mp 104-105°; ^{1}H nmr: δ 8.35 (d, J = 8.35 Hz, 1H, Bt-C7), 7.63 (d, J = 8.4 Hz, 1H, Bt-C4), 7.47 (t, J = 6.9 Hz, 1H, Bt-C6(or C5)) 7.33 (t, J = 8.0 Hz, 1H, Bt-C5(or C6)), 6.8 (bs, 1H, N-H), 4.88 (dd, J = 8.7 and 13.9 Hz, 1H, Bt-CH_2), 4.67 (dd, J = 5.7 and 13.9 Hz, 1H, Bt-CH_2), 3.3-3.1 (m, 1H, C(CH_3)-H), 2.44 (d, J = 4.7 Hz, 3H, N-CH_3), and 1.27 (d, J = 6.9 Hz, 3H, C(H)CH_3); ^{13}C nmr: δ 173.95, 145.0, 133.2, 127.3, 123.9, 119.0, 110.0, 50.8, 41.8, 26.0, and 15.8.

Anal. Calcd. for $C_{11}H_{14}N_4O$: C, 60.53; H, 6.47; N, 25.67. Found: C, 60.35; H, 6.44; N, 25.70.

General Procedure for the Lithiation of **3** and Treatment with Electrophiles.

A solution of the amide **3** (1.25 g, 5.7 mmoles) in tetrahydrofuran (50 ml) was cooled to −78° in a nitrogen atmosphere and butyllithium (2.5 *M* in hexanes, 5 ml, 12.5 mmoles) was added dropwise ensuring that the temperature stayed below −70°. After 1 hour, the appropriate electrophile (6 mmoles) in tetrahydrofuran (5 ml) was added dropwise to the dark red solution. The reaction temperature was kept below −70° and the reaction monitored by tlc. When all the starting material had disappeared, (*ca.* 3-6 hours), the reaction mixture was poured into water (25 ml) and the organic material extracted with chloroform (3 x 50 ml). The organic layer was dried (magnesium sulfate) and the solvent removed under reduced pressure to afford the crude product. Purification by column chromatography (diethyl ether) afforded the pure products **4** and **5a-g** (see Tables 1-3).

4(Benzotriazol-1-yl)-*N*,3-dimethyl-5-hydroxy-5-(4-methylphenyl)-2-pyrrolidone (**4**).

This compound was obtained as colorless needles (benzene), (see Table 1); ^{1}H nmr: δ 8.07 (d, J = 8.7 Hz, 1H), 7.35-7.15 (m, 6H), 6.41 (d, J = 8.4 Hz, 1H), 4.99 (d, J = 10.0 Hz, 1H, C4-H), 4.87 (s, 1H, OH), 4.11 (m, 1H, C3-H), 2.78 (s, 3H, N-CH_3), 2.40 (s, 3H, ArCH_3), and 1.37 (d, J = 7.1 Hz, 3H, C3-CH_3); ^{13}C nmr: δ 174.8, 145.9, 138.9, 136.6, 134.0, 129.7, 127.4, 126.2, 124.4, 119.9, 109.6, 90.3, 72.0, 38.7, 25.9, 21.2, and 13.8.

General Procedure for the Base Induced Elimination of Benzotriazole from **5b-e**. Formation of Unsaturated Amides **6a-c** and **7**.

The appropriate amide **5b-e** (5 mmoles) was heated under reflux with 0.2 *M* ethanolic sodium ethoxide (50 ml, 10 mmoles) for 1 day. The solvent was removed under reduced pressure and water (25 ml) was added. The organic material was extracted with chloroform (3 x 25 ml) and the organic fraction was washed with water (3 x 20 ml), dried (magnesium sulfate) and the solvent removed under reduced pressure to afford the unsaturated amides **6a-c** and **7**. The crude products were purified by column chromatography (methylene chloride as eluent for **6a-c**, diethyl ether as eluent for **7** (see Table 4).

2,*N*-Dimethylhept-2-enamide (**6a**).

This compound was obtained as a pale yellow oil; ^{1}H nmr (*E*)-isomer only: δ 6.34 (dt, J = 1.4, 7.3 Hz, 1H, CH=), 6.1-6.0 (bs, 1H, N-H), 2.85 (d, J = 4.8 Hz, 3H, N-CH_3), 2.2-2.1 (m, 2H,

$=CCH_2$), 1.84 (s, 3H, $=CCH_3$), 1.5-1.3 (m, 4H, alkyl), and 0.90 (t, J = 7.0 Hz, 3H, alkyl-CH_3); ^{13}C nmr: δ 170.1, 136.0, 130.5, 30.8, 27.8, 26.4, 22.3, 13.8, and 12.5.

2,*N*-Dimethylnon-2-enamide (**6b**).

This compound was obtained as a pale yellow oil; ^1H nmr (*E*)-isomer only: δ 6.34 (dt, J = 1.1, 7.1 Hz, 1H, CH=), 6.1-5.9 (bs, 1H, N-H), 2.86 (d, J = 4.6 Hz, 3H, N-CH_3), 2.2-2.1 (m, 2H, $=CCH_2$), 1.84 (s, 3H, $=CCH_3$), 1.5-1.2 (m, 8H, alkyl), and 0.88 (m, 3H, alkyl-CH_3); ^{13}C nmr: δ 170.1, 136.0, 130.4, 34.5, 28.9, 28.6, 28.1, 22.4, 13.9, and 12.5.

2,*N*-Dimethylundec-2-enamide (**6c**).

This compound was obtained as a pale yellow oil; ^1H nmr (*E*)-isomer only: δ 6.34 (t, J = 7.5 Hz, 1H, =C-H), 6.14 (bs, 1H, N-H), 2.86 (d, J = 4.8 Hz, 3H, N-CH_3), 2.18-2.06 (m, 2H, =C-CH_2), 1.83 (s, 3H, =C-CH_3), 1.5-1.25 (m, 12H, alkyl chain), and 0.88 (t, J = 6.8 Hz, terminal CH_3); ^{13}C nmr: δ 170.1, 136.0, 130.4, 31.7, 29.3, 29.2, 29.1, 28.7, 28.1, 26.4, 22.5, 13.9, and 12.5.

2,*N*-Dimethyl-4-phenylbut-3-enamide (**7**).

This compound was purified by column chromatography (diethyl ether) to give **7** as colorless microcrystals; ^1H nmr: δ 7.4-7.2 (m, 5H, phenyl), 6.50 (d, J = 15.9 Hz, 1H, PhCH=), 6.25 (dd, J = 8.2 and 15.9 Hz, =CH), 5.80 (bs, 1H, N-H), 3.14 (m, 1H, C(CH_3)-H), 2.80 (d, J = 4.88 Hz, 3H, N-CH_3), and 1.37 (d, J = 7.1 Hz, 3H, C-CH_3); ^{13}C nmr: δ 175.5, 136.5, 131.8, 129.6, 128.6, 127.7, 126.2, 44.8, 26.4, and 17.4.

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