JOC*Note*

Easy Access to *N*,*N*-Bis(but-3-enyl)-, *N*-Allyl-*N*-(but-3-enyl)-, and *N*-(But-3-ynyl)-*N*-(but-3-enyl)-amines

Alan R. Katritzky,* Satheesh K. Nair, and Alina Silina

Center for Heterocyclic Compounds, Department of Chemistry, University of Florida, Gainesville, Florida 32611-7200

katritzky@chem.ufl.edu

Received February 20, 2002

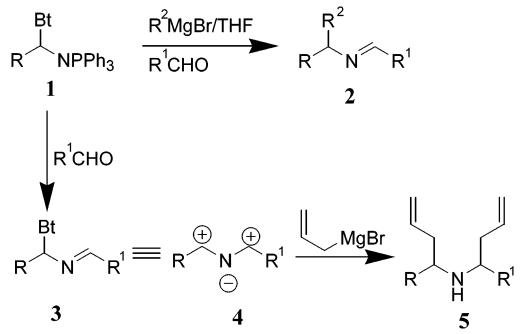
Abstract: *N*,*N*-Bis(but-3-enyl)amines **5a**−**i** were prepared in overall 74% yield from 1-(triphenylphosphoroylideneaminoalkyl)benzotriazole using an aza-Wittig reaction with aldehydes followed by a double Grignard reaction with allylmagnesium bromide. Use of vinyl or 1-propynylmagnesium bromide and allylmagnesium bromide in a sequential fashion also formed the expected doubly unsaturated amines **9a,b** and **12**, respectively.

Ring-closing metathesis (RCM) has emerged as a powerful tool for the construction of carbocycles and heterocycles.[1] *N*,*N*-Bis(3-butenyl)amines, diallylamines,[2] and other dialkenylamines[3] are versatile candidates for RCM syntheses of nitrogen-containing heterocycles.[4] Bis(butenyl)amines can be prepared by the allylation of sulfinylamines and used in the synthesis of piperidines;[5] *N*-allyl-*N*-(3-butenyl)amines have been used to prepare pyrrolidine and piperidine alkaloids.[6]

Previously, *N*-butenylamines have been prepared by multistep sequences.[5,6] Substituted bis(butenyl)amines were obtained from commercially available 4-(2-propenyl)-*β*-lactams and allyltrimethylsilane[7] and used in RCMs.[8] Pearson et al. made *N*,*N*-bis(3-butenyl)amines for tetrahydroazepines ring closures[9] from 2-(azaallyl)-stannanes, obtained in a two-step process starting from tin bearing phthalides.[10] A more general synthetic route to bisbutenyl and related amines with fewer steps should facilitate their application. We herein report such easy access to *N*,*N*-(bisbutenyl)amines from readily available 1-(triphenylphosphoroylideneaminoalkyl)benzotriazoles.

Synthetic applications of iminophosphoranes[11] are well documented; they possess structural and chemical characteristics similar to those of phosphorus ylides and react with carbonyl compounds to form Schiff bases.[11a] Iminophosphoranes also react with carbon dioxide and carbon disulfide to yield isocyanates and isothiocyanates, respectively.[12] We have reported earlier applications of 1-(triphenylphosphoroylideneaminomethyl)benzotriazole to the synthesis of carbodiimides, imines, isothiocyanates, aziridines, secondary and primary amines,[13] and *N*-(vinylimino)phosphoranes.[14] Grignard displacements of the benzotriazole group followed by the aza-Wittig reaction with aldehydes give 83% yields of the corresponding Schiff bases **2** (Scheme 1).[13a]

SCHEME 1



We envisioned that aza-Wittig reactions on **1** should afford imines **3** with the benzotriazole unit intact. Intermediates **3** could be synthetic equivalents to amine α,α′-dications **4**. Since nucleophilic addition to imines is known[15] to give amines and since benzotriazole is a good leaving group,[16] a double Grignard addition to **3** with allylmagnesium bromide should give bis(butenyl)amines **5**.

1-(Triphenylphosphoroylidenaminomethyl)benzotriazoles **1a**−**c** were prepared following known methods.[13] Aza-Wittig coupling of **1a**−**c** with aldehydes **6a**−**g** occurred in THF at room temperature; when **1a** was stirred with 1 equiv of benzaldehyde in THF for 10 h, TLC indicated complete reaction. However, attempts to purify the imine using chromatographic techniques failed, and vacuum distillation of the imine following the literature reports[13] resulted in charring and significant loss of material. Fortunately, the crude imine **3a** in a Grignard reaction with 2.2 equiv of allylmagnesium bromide in THF at 20 °C formed bis(butenyl)amine **5a** in 78% yield. Flash chromatography over silica gel provided an analytically pure sample of **5a** characterized

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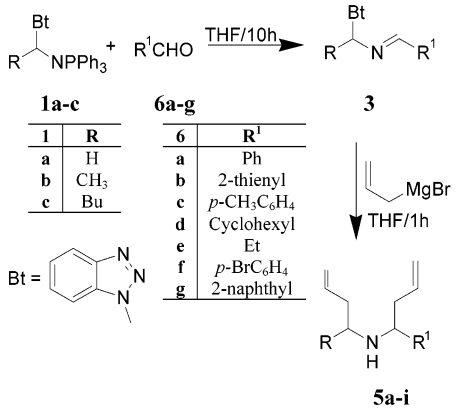
10.1021/jo020118t CCC: $22.00
Published on Web 09/21/2002

TABLE 1. ***N,N*-Bis(3-butenyl)amines 5 Prepared**

	R	R^1	yield (%)[a]
5a	H	Ph	78
5b	CH_3	Ph	62 (1:2.3)[b]
5c	H	2-thienyl	71
5d	CH_3	*p*-$CH_3C_6H_4$	58 (1:3.5)[b]
5e	H	cyclohexyl	82
5f	butyl	Ph	67 (1:1)[b]
5g	H	Et	82
5h	H	*p*-BrC_6H_4	82
5i	H	2-naphthyl	80

[a] All yields refer to pure isolated product. [b] Diastereomeric ratio.

SCHEME 2



by NMR spectra. 1-(Triphenylphosphoroylidenomethyl)-benzotriazoles **1b** and **1c** similarly yielded respectively amines **5b** and **5f**, which were isolated as mixtures of diastereomers as indicated by ^{1}H NMR. The method was extended to other aldehydes as summarized in Table 1. Attempts to use ketones in the aza-Wittig reaction failed even in refluxing toluene. Sequential addition of two different Grignard reagents to the intermediate imine **3** would make the approach more versatile. However, our results showed that there is not much selectivity between the imine carbon and the carbon bearing the benzotriazole, and initial attempts resulted in a mixture of products that were difficult to separate and characterize (e.g., when **1a** was treated with benzaldehyde followed by sequential addition of single equivalents of vinyl- and allylmagnesium bromide, TLC indicated an intractable mixture). Adding the first Grignard reagent to displace the benzotriazole before the aza-Wittig reaction solved this problem. Subsequent coupling with an aldehyde and addition of the second Grignard reagent (allylmagnesium bromide) succeeded.

Benzotriazole derivative **1a** was treated in THF with 1 equiv of vinylmagnesium bromide. The resulting intermediate **7a** was subjected to aza-Wittig with benzaldehyde. Imine **8a** was then reacted with 1 equiv of allylmagnesium bromide. The unsymmetrical bisalkenylamine **9a** was isolated in 87% yield after workup and purification (Scheme 3).

We used propynylmagnesium bromide in the sequential addition in an extension of this approach to the preparationof *N*-(2-butynyl)-1-phenyl-3-buten-1-amine **12** in 71% yield (Scheme 4). The only example of an amine of type **12** reported in the literature is 4-[(1-allyl-1-methoxy-3-butenyl)amino]-2-butyn-1-ol, which is prepared in three steps starting from the corresponding bisallyl aminoether.[17] Our general route to the preparation of these intermediates provides an easy access to these compounds with their potential applications in RCM.

Advantageously, all three steps of Schemes 3 and 4 can be carried out sequentially in one-pot reactions to provide good yields of the amines. Thus 1-(triphenylphosphoroylideneaminoalkyl)benzotriazoles serve as common precursors for the preparation of bis(butenyl)-, allylbutenyl-, and *N*-butynyl-3-butenyl-amines depending on the organometallic reagents used.

In conclusion, convenient synthetic methods are described for the preparation of amines of types **5**, **9**, and **12**, which should expedite the synthesis of these intermediates and their applications particularly in the construction of heterocycles.

Experimental Section

Melting points were determined using a hot-stage microscope and are uncorrected. ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR spectra were recorded on a 300 MHz NMR spectrometer in chloroform-*d* solution. Column chromatography was performed on silica gel. All Grignard reagents were procured as solution in THF. THF was distilled from sodium-benzophenone ketal prior to use. All reactions were performed under a nitrogen atmosphere and in flame dried glasswares. 1-(Triphenylphosphoroylideneaminoalkyl)benzotriazoles **1** were prepared following the literature method.[13]

General Procedure for the Preparation of Bis(butenyl)-amines 5a−i. 1-(Triphenylphosphoroylideneaminoalkyl)benzotriazole **1** (10 mmol) was taken up in dry THF (50 mL) and was allowed to react with the corresponding aldehyde (10 mmol) at room temperature for 5 h. After TLC analysis indicated complete consumption of the aldehyde, allylmagnesium bromide (20 mmol, 1 M solution in THF) was added dropwise at room temperature. After the addition, the reaction mixture was stirred for 2 h at room temperature, poured into cold water, and extracted with ether (3 × 100 mL). The combined etherial layer was washed with 2 N NaOH (2 × 50 mL) followed by water (2 × 50 mL). The amine **5** was purified by acid−base workup. Further purification was done by flash column chromatography over silica gel using hexane as the eluent.

***N*-(But-3-enyl)-1-phenyl-3-buten-1-amine (5a)**: Obtained as a pale yellow oil (78%). ^{1}H NMR δ 1.42 (bs, 1 H), 2.08−2.15 (m, 2 H), 2.28−2.36 (m, 2 H), 2.39−2.44 (m, 2 H), 3.56 (t, J = 6.6, 1 H), 4.90−5.02 (m, 4 H), 5.55−5.71 (m, 2 H), 7.12−7.23 (m, 5 H). ^{13}C NMR δ 34.1, 42.9, 46.4, 62.4, 116.1, 117.4, 126.8, 127.1, 128.2, 135.4, 136.43, 143.9. Anal. Calcd for $C_{14}H_{19}N$: C, 83.53; H, 9.51; N, 6.96. Found: C, 83.51; H, 9.60; N, 7.45.

***N*-(1-Methyl-3-butenyl)-*N*-(1-phenyl-3-butenyl)amine (5b).** Obtained (mixture of diastereomers, 2.3:1) as a pale yellow oil (62%). ^{1}H NMR δ 0.84 (d, J = 6.6 Hz, 2.1 H), 0.92 (d, J = 6 Hz, 0.88 H), 1.51 (bs, 1 H), 1.99−2.12 (m, 2 H), 2.26−2.34 (2 H), 2.35−2.55 (m, 1 H), 3.68−3.73 (m, 1 H), 4.92−5.02 (m, 4 H), 5.55−5.62 (m, 2 H), 7.19−7.31 (m, 5 H). ^{13}C NMR δ 19.6, 21.3, 40, 42.4, 43.2, 43.3, 48.8, 49.8, 59.3, 59.8, 116.8, 117.1, 117.4, 126.7, 127, 128.2, 135.3, 135.5, 135.9, 144.0, 144.6. HRMS (FAB) calcd for $C_{15}H_{22}N$ (M + H) 216.1757, found 216.1757.

***N*-(But-3-enyl)-1-(2-thienyl)-3-buten-1-amine (5c).** Obtained as a pale yellow oil (71%). ^{1}H NMR δ 1.62 (bs, 1 H), 2.18−2.25 (m, 2 H), 2.45−2.52 (m, 2 H), 2.54−2.68 (m, 2 H), 3.96 (t, J = 6.9 Hz, 1 H), 4.99−5.14 (m, 4 H), 5.67−5.81 (m, 2 H), 6.89−6.94 (m, 2 H), 7.18−7.20 (m, 1 H). ^{13}C NMR δ 34.0, 43.4, 46.3, 57.9, 116.3, 117.8, 123.7, 123.8, 126.3, 134.7, 136.3, 149.0. Anal. Calcd for $C_{12}H_{17}N$: C, 69.51; H, 8.26; N, 6.76. Found: C, 69.52; H, 8.79; N, 6.73.

***N*-(1-Methyl-3-butenyl)-*N*-(1-(4-methylphenyl)-3-butenyl)amine (5d).** Obtained (mixture of diastereomers, 3.5:1) as a pale yellow oil (58%). ^{1}H NMR δ 0.83 (d, J = 6.6 Hz, 2.4 H),

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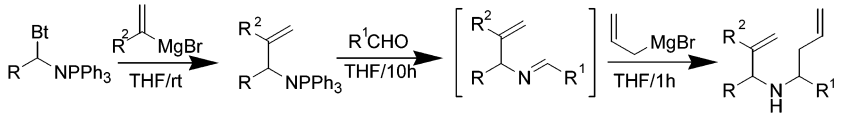
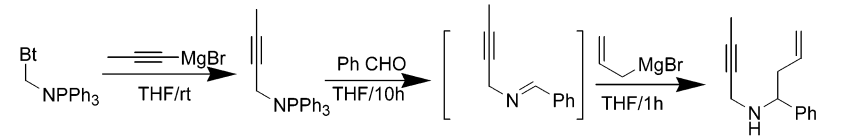
SCHEME 3



1a **7a-b** **8** **9a-b**

9	R	R^1	R^2	Yield (%)
a	H	Ph	H	87
b	H	*p*-BrC_6H_4	CH_3	84

SCHEME 4



1a **10** **11** **12 (71%)**

0.85 (d, J = 6.0 Hz, 0.62H), 1.39 (bs, 1 H), 1.96−2.19 (m, 2.6 H), 2.25−2.3 (m, 5.4 H), 2.32−2.56 (m, 1 H), 3.67 (t, J = 6.6 Hz, 1 H), 4.90−5.02 (m, 4 H), 5.53−5.73 (m, 2 H), 7.04 (d, J = 6.0 Hz, 2 H), 7.08 (d, J = 6.0 Hz, 2 H). ^{13}C NMR δ 19.6, 21.0, 21.3, 39.9, 42.4, 43.2, 43.3, 48.7, 49.7, 59.0, 59.4, 116.8, 117.0, 117.1, 117.3, 126.9, 128.9, 135.3, 135.6, 136.0, 136.2, 140.8, 141.5. Anal. Calcd for $C_{16}H_{23}N$: C, 83.78; H, 10.11; N, 6.11. Found: C, 81.01; H, 9.16; N, 5.91.

***N*-(But-3-enyl)-1-cyclohexyl-3-buten-1-amine (5e).** Obtained as a pale yellow oil (82%). ^{1}H NMR δ 0.87−1.21 (m, 6 H), 1.31−1.39 (m, 1 H), 1.61−1.69 (m, 5 H), 1.93−2.03 (m, 1 H), 2.10−2.28 (m, 4), 2.56 (t, J = 6.9 Hz, 1 H), 4.93−5.03 (m, 4 H), 5.64−5.77 (m, 2 H).^{13}C NMR 26.5, 26.6, 26.7, 28.7, 29.4, 34.5, 35.4, 40.6, 46.9, 62.0, 116.0, 116.6, 136.7. Anal. Calcd for $C_{14}H_{25}N$: C, 81.09; H, 12.15; N, 6.75. Found: C, 80.84; H, 12.17; N, 6.98.

***N*-(1-Phenyl-3-butenyl)-*N*-(1′-propyl-3-butenyl)amine (5f).** Obtained (mixture of diastereomers, 1:1) as a pale yellow oil (67%). ^{1}H NMR δ 0.76 (t, J = 6.6 Hz, 1.42 H), 0.871 (t, J = 6.9 Hz, 1.43 H), 0.94−0.99 (m, 0.36 H), 1.12−1.52 (m, 5 H), 1.55−2.25 (m, 2 H), 2.27−2.40 (m, 3 H), 3.69−3.81 (m, 1 H), 4.96−5.08 (m, 4 H), 5.58−5.78 (m, 2 H), 7.19−7.31 (m, 5H). ^{13}C NMR 14.0, 14.4, 18.4, 19.0, 35.6, 37.2, 37.3, 39.3, 41.5, 43.143.5, 53.2, 53.5, 59.4, 59.6, 59.8, 116.7, 117.1, 117.3, 126.7, 126.8, 127.1, 127.2, 128.0, 128.1, 135.1, 135.2, 135.6, 135.7, 136.2, 144.5, 144.6. Anal. Calcd for $C_{17}H_{25}N$: C, 83.89; H, 10.35; N, 5.75. Found: C, 83.73; H, 10.80; N, 5.82.

***N*-(But-3-enyl)-1-ethyl-3-buten-1-amine (5g).** Obtained as a pale yellow oil (82%). ^{1}H NMR δ 0.89 (t, J = 7.5 Hz, 3 H), 1.35−1.48 (m, 2 H), 2.08−2.27 (m, 4 H), 2.44−2.50 (m, 1 H), 2.61−2.69 (m, 2 H), 5.01−5.11 (m, 4 H), 5.70−5.82 (m, 2 H). ^{13}C NMR δ 9.9, 26.3, 34.4, 37.9, 46.0, 58.2, 116.2, 117.0, 135.8, 136.5. Anal. Calcd for $C_{11}H_{23}N$: C, 78.03; H, 13.69; N, 8.27. Found: C, 78.26; H, 11.96; N, 8.27.

***N*-(But-3-enyl)-1-(4-bromophenyl)-3-buten-1-amine (5h).** Obtained as a pale yellow oil (82%). ^{1}H NMR δ 1.53 (bs, 1 H), 2.15−2.22 (m, 2 H), 21.32−2.34 (m, 2 H), 2.36−2.49 (m, 2 H), 3.61 (t, J = 6.9 Hz, 1 H), 4.99−5.09 (m, 4 H), 5.61−5.79 (m, 2 H), 7.19 (d, J = 8.4 Hz, 2 H), 7.44 (d, J = 8.4 Hz, 2 H). ^{13}C NMR δ 34.1, 42.9, 46.5, 61.9, 116.3, 117.8, 120.5, 128.9, 131.3, 134.9, 136.3, 143.0. Anal. Calcd For $C_{14}H_{18}BrN$: C, 60.01; H, 6.47; N, 5.00. Found: C, 60.27; H, 6.63; N, 5.27.

***N*-(But-3-enyl)-1-(2-naphthyl)-3-buten-1-amine (5i).** Obtained as a reddish oil (80%). ^{1}H NMR δ 1.63 (bs, 1 H), 2.09−2.16 (m, 2 H), 2.36−2.46 (m, 4 H), 3.72 (t, J = 6.9 Hz, 1 H), 4.90−5.04 (m, 4 H), 5.57−5.71 (m, 2 H), 7.32−7.40 (m, 3 H), 7.65−7.74 (m, 4 H). ^{13}C NMR 34.2, 42.9, 62.6, 116.3, 117.6, 125.2, 125.4, 125.8, 125.9, 127.6, 127.7, 128.1, 182.8, 133.3, 135.3, 136.4, 141.3. Anal. Calcd for $C_{18}H_{21}N$: C, 86.01; H, 8.42; N, 5.57. Found: C, 86.29; H, 8.71; N, 5.58.

General Procedure for the Preparation of 9a,b and 12. 1-(Triphenylphosphoroylideneaminoalkyl)benzotriazole **1** (10 mmol) was taken up in dry THF (50 mL), and the corresponding vinylic or propargyl Grignard reagent (10.2 mmol) was added dropwise at room temperature. The mixture was allowed to stir at room temperature for 3 h, diluted with ether (200 mL), and filtered. The filtrate was dried over sodium sulfate and concentrated, and the oily residue was redissolved in THF (50 mL). The resulting solution was treated with the appropriate aldehyde (10 mmol) at room temperature for 5 h. After TLC analysis indicated complete consumption of the aldehyde, allylmagnesium bromide (10 mmol, 1 M solution in THF) was added dropwise at room temperature. After the addition, the reaction mixture was stirred for 2 h at room temperature, poured into cold water, and extracted with ether (3 × 100 mL). The combined etherial layer was washed with 2 N NaOH (2 × 50 mL) followed by water (2 × 50 mL). The amine was purified by acid−base workup. Further purification was done by flash column chromatography over silica gel using hexane as the eluent.

***N*-Allyl-1-phenyl-3-buten-1-amine (9a).** Obtained as a colorless liquid (87%). ^{1}H NMR δ 1.49 (bs, 1 H), 2.37−2.42 (m, 2 H), 3.05 (dddd, J = 20.7, 19.5, 6.6, 5.4 Hz, 2 H), 3.66−3.70 (t, J = 7.2 Hz, 1 H), 5.63−5.74 (m, 1 H), 5.77−5.90 (m, 1 H), 7.16−7.40 (m, 5 H). ^{13}C NMR δ 42.80, 49.8, 61.5, 115.4, 117.3, 126.8, 127.0, 128.1, 135.2, 136.7, 143.5. Anal. Calcd for $C_{13}H_{17}N$: C, 83.37; H, 9.15; N, 7.48. Found: C, 83.36; H, 9.77; N, 7.68.

***N*-(2-Methyl-2-propenyl)-1-(4-bromophenyl)-3-buten-1-amine (9b).** Obtained as a pale yellow oil (84%). ^{1}H NMR δ 1.58 (bs, 1 H), 1.74−1.75 (m, 3 H), 2.29−2.39 (m, 2 H), 2.89−3.00 (m, 2 H), 3.61 (t, J = 7.8 Hz, 1 H), 4.84−4.88 (m, 2 H), 5.09−5.18 (m, 2 H), 5.63−5.77 (m, 1 H), 7.20 (d, J = 8.4 Hz, 2 H), 7.42 (d, J = 8.4 Hz, 2 H). ^{13}C NMR δ 20.7, 43.0, 53.0, 60.6, 110.7, 117.8, 120.5, 128.9, 131.3, 135.0, 143.0, 143.7. Anal. Calcd for $C_{14}H_{18}BrN$: C, 60.01; H, 6.47; N, 5.00. Found: C, 60.31; H, 6.79; N, 5.24.

***N*-(But-2-ynyl)-*N*-(1-phenyl-3-butene-1-amine (12).** Obtained as a pale yellow oil (71%). ^{1}H NMR 1.63 (bs, 1 H), 1.80 (t, J = 4.5, Hz, 3 H), 2.35−2.49 (m, 2 H), 3.04 (dd, J = 16.5, 2.1 Hz, 1 H), 3.27 (dd, J = 14.1, 2.4 Hz, 1 H), 3.84 (dd, J = 7.8, 6.0 Hz, 1 H), 5.01−5.13 (m, 2 H), 5.65−5.79 (m, 1 H), 7.2−7.32 (m, 5 H). ^{13}C NMR δ 3.4, 36.2, 42.6, 60.6, 78.7, 117.6, 127.0, 127.3, 128.2, 128.3, 135.2, 142.8. HRMS (FAB) calcd for $C_{14}H_{18}N$ (M + H) 200.1458, found 200.1458.

Supporting Information Available: Copies of NMR spectra for **5d** and **8a**. This material is available free of charge via the Internet at http://pubs.acs.org.

JO020118T