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Studies in aza-Claisen rearrangement: synthesis of dimedone-annelated unusual heterocycles

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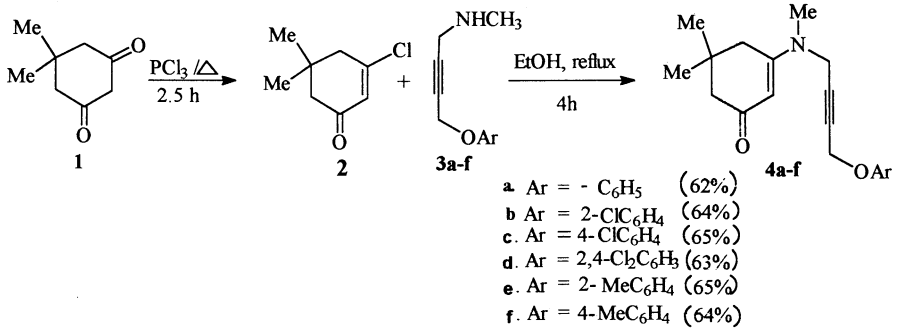
Abstract—A number of 3-*N*-(4′-aryloxybut-2′-ynyl)*N*-methyl amino-5,5-dimethyl cyclohex-2-enones are synthesized in 62–65% yield by refluxing 3-chloro-5,5-dimethyl cyclohex-2-enone with a number of 4-aryloxy-4-chlorobut-2-ynes in ethanol for 4 h. The amines are then heated in refluxing chlorobenzene to give 7,7-dimethyl-1,2,3,6,7,8-hexahydro-4-aryloxymethylene-1-methyl quinoline-5-one in 75–80% yields.

Since its discovery[1] in 1912, the Claisen rearrangement of allyl vinyl ethers has become a powerful tool for carbon–carbon bond formation.[2] Much of its current popularity is due to the subsequent development of a series of new variants[3] of this [3,3] sigmatropic rearrangement as well as its versatile application in the synthesis of various heterocycles using oxygen,[4] nitrogen,[5] and sulfur[6] Claisen rearrangements. Our recent work on the synthesis of various heterocycles by the application of [3,3] sigmatropic rearrangement[7] prompted us to undertake a study on the synthesis of dimedone-annelated heterocycles. Here we report the results of this investigation.

1. Results and discussion

The starting materials (**4a**–**f**) for this study were synthesized in 62–65% yield by the reaction of 3-chloro-5,5-dimethyl-cyclohex-2-enone[8] with a number of 1-aryloxy-4-*N*-methyl-amino but-2-ynes (**3a**–**f**) in refluxing absolute ethanol for 4 h. (Scheme 1). 3-Chloro-5,5-dimethyl cyclohex-2-enone (**2**) in turn was obtained from the reaction of dimedone with phosphorous trichloride.

The substrates (**4a**–**f**) are unique in the sense that the molecules provide two different sites for the occurrence of [3,3] sigmatropic rearrangement, the propargyl vinyl amine moiety and the aryl propargyl ether moiety. Our past observation in case of 5-*N*-[4-aryloxybut-2-ynyl],*N*-methyl amino-1,3-dimethyl pyrimidine-2,4-diones have shown that the [3,3] sigmatropic rearrangement is preferred at the aryl propargyl ether moiety over vinyl propargyl amine moiety.[9] However, with other substrates such as 4-*N*-(4-aryloxybut-2-ynyl),*N*-methyl amino-6-methyl pyran-2-one and 4-*N*-(4-aryloxybut-2-ynyl),*N*-methyl amino[1]benzopyran-2-one, the [3,3] sigmatropic rearrangement occurred at the



Scheme 1.

Keywords: aza-Claisen rearrangement; sigmatropic rearrangement; 3-chloro-5,5-dimethyl cyclohex-2-enone; 3,3-prototropic shift; 5-*N*-[4-aryloxybut-2-ynyl],*N*-methylamino-1,3-dimethylpyrimidine-2,4-dione; 7,7-dimethyl-1,2,3,6,7,8-hexahydro-4-aryloxymethylenemethyl quinoline-5-one.

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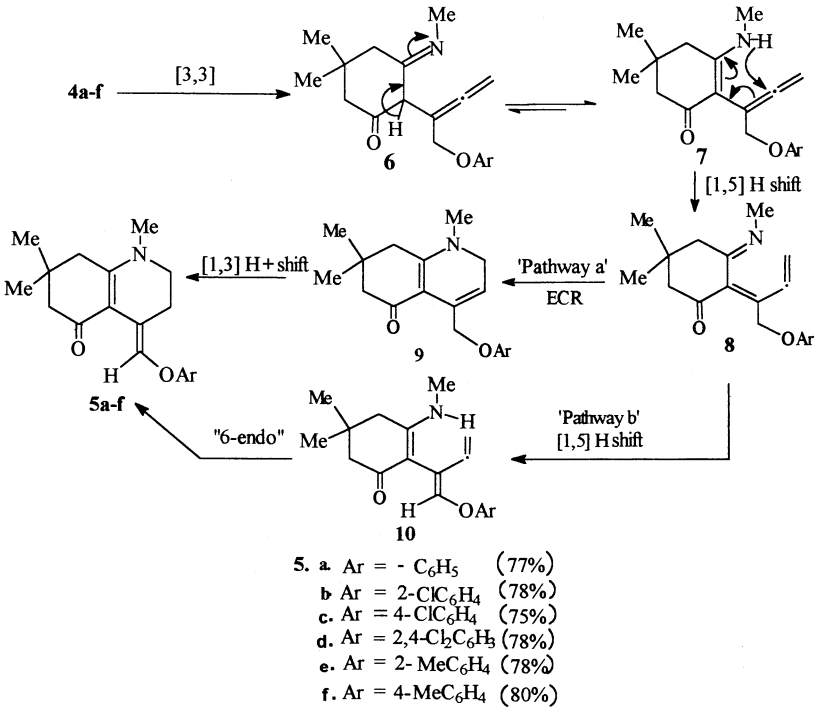

Scheme 2.

propargyl vinyl amine part of the substrates.[10] The present substrates (**4a–f**), therefore, provide scope for studying the competitive [3,3] sigmatropic rearrangement. With this end in view the substrate **4a** was heated in refluxing chlorobenzene. TLC monitoring indicated the formation of a new product. However, it took about 12 h to complete the reaction. The product was characterized from its elemental analysis and spectral data as 7,7-dimethyl-1,2,3,6,7,8-hexahydro-4-phenoxymethylene-1-methylquinoline-5-one (**5a**). Encouraged by this result other substrates were also similarly treated to give products **5b–f** (Scheme 2)

The formation of products **5a–f** from **4a–f** may be explained by an initial [3,3] sigmatropic shift at the propargyl vinyl amine moiety to give the allene intermediate **6**, followed by tautomerism, [1,5] H shift and electrocyclic ring closure to give unstable endocyclic intermediate **9** (not isolated). A [1,3] prototropic shift in **9** may give final product **5** ('pathway a'). Support in favour of this pathway came from our recent observation that the thermal rearrangement of 4-*N*-(4-aryloxybut-2-ynyl),*N*-methylamino-6-methyl-2-pyrone gave both exocyclic and endocyclic products and the endocyclic products are converted to exocyclic products under the same reaction condition.[10a] The TLC of the crude reaction mixture indicated the formation of the endocyclic products. However, we were unable to isolate them despite our best efforts. Another pathway may be via one more [1,5] H shift in **8** followed by a 6-endocyclization ('pathway b') to give **5** (Scheme 2).

It is interesting to note that the [3,3] sigmatropic rearrangement occurred at the propargyl vinyl amine part of all the substrates (**4a–f**) studied so far. The formation of unusual exocyclic products instead of normal endocyclic products is noteworthy.

2. Experimental

Melting points were measured on a sulfuric acid bath and are uncorrected. UV absorption spectra were recorded [Perkin–Elmer UV–vis Spectrophotometer, Lamda 20, 1 nm] in EtOH. IR spectra were run on KBr disks on a Perkin–Elmer 1330 apparatus. ^{1}H NMR spectra were determined for solutions in $CDCl_3$ with TMS as internal standard on a Bruker 300 (300 MHz) instrument. Elemental analyses and recording of mass spectra were carried out by RSIC(CDRI) Lucknow on a [JEOL D-300 (El)] instrument. Silica gel [(60–120 mesh), Spectrochem, India] was used for chromatographic separation. Petroleum ether refers to the fraction boiling between 60 and 80°C.

2.1. Preparation of 1-(aryloxy)-4-*N*-methylaminobut-2-yne

1-Aryloxy-4-chlorobut-2-yne[11] (1 g, 5.5 mmol) in 20 mL of EtOH was added dropwise at room temperature to a well stirred solution of (23 mL, 296.8 mmol) of methylamine (40% aqueous solution) for a period of 10 min. Stirring was continued for another 2 h at room temperature. Methylamine, water and EtOH were removed under reduced pressure and the residual mass was dissolved in $CHCl_3$ (25 mL). This was washed with brine (2×25 mL) and dried over anhydrous Na_2SO_4. Removal of $CHCl_3$ afforded a gummy mass. Attempts to purify this gummy mass by column chromatography failed. It was therefore used directly in the subsequent reactions.

2.2. General procedures for the synthesis of compounds 4(a–f)

A mixture of 1-aryloxy-4-*N*-methylaminobut-2-yne

(5 mmol) and 3-chloro-5,5-dimethylcyclohex-2-enone (0.50 g, 3.2 mmol) in anhydrous EtOH (50 mL) was refluxed for 4 h on a water bath. Alcohol was removed by distillation and the residual mass was dissolved in $CHCl_3$ (50 mL). This was washed with brine (2×25 mL) and dried over anhydrous Na_2SO_4. Evaporation of $CHCl_3$ afforded a gummy mass which was subjected to column chromatography over silica gel. Elution of the column with benzene–ethyl acetate (1:1) gave compounds **4(a–f)**.

2.2.1. 3-*N*-(4′-Phenoxybut-2′-ynyl)*N*-methyl amino-5,5-dimethyl cyclohex-2-enone (4a). Yield 62%; viscous liquid; λ_{max}: (log ϵ) 219 (3.94), 297 (4.36) nm; IR (KBr) ν_{max}: 1100, 1240, 1410, 1540, 1610, 2960 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.14 (s, 2H, CH_2CO), 2.26 (s, 2H, =CCH_2), 2.92 (s, 3H, N*Me*), 4.02 (brs, 2H, NCH_2), 4.70 (brs, 2H, OCH_2), 5.21 (s, 1H, =C*H*), 6.92–6.99 (m, 3H, Ph), 7.26–7.32 (m, 2H, Ph); MS *m*/*z* 297 (M^+); Anal. calcd for $C_{19}H_{23}NO_2$: C, 76.76; H, 7.74; N, 4.71; found C, 76.98; H, 7.89; N, 4.87%.

2.2.2. 3-*N*-[4′-(2′-Chlorophenoxy)but-2′-ynyl]*N*-methyl amino-5,5-dimethyl cyclohex-2-enone (4b). Yield 64%; viscous liquid; λ_{max}: (log ϵ) 220 (3.98), 295 (4.41) nm; IR (KBr) ν_{max}: 1100, 1230,1510, 1700, 2910 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.04 (s, 2H, CH_2CO), 2.26 (s, 2H, =CCH_2), 2.9 (s, 3H, N*Me*), 4.01 (t, *J*=1.5 Hz, 2H, NCH_2), 4.79 (t, *J*=1.5 Hz, 2H, OCH_2), 5.19 (s, 1H, =C*H*), 6.94–7.03 (m, 1H, Ph), 7.18–7.21 (m, 1H, Ph), 7.35–7.39 (m, 2H); MS *m*/*z* 331, 333 (M^+); Anal. calcd for $C_{19}H_{22}ClNO_2$: C, 68.77; H, 6.63; N, 4.22; found C, 68.92; H, 6.81; N, 4.31%.

2.2.3. 3-*N*-[4′-(4′-Chlorophenoxy)but-2′-ynyl]*N*-methyl amino-5,5-dimethyl cyclohex-2-enone (4c). Yield 65%; viscous liquid; λ_{max}: (log ϵ) 221 (3.98), 294 (4.08) nm; IR (KBr) ν_{max}: 1100, 1240, 1410, 1550, 1610, 2960 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.15 (s, 2H, CH_2CO), 2.25, (s, 2H, =CCH_2), 2.92 (s, 3H, N*Me*), 4.01 (brs, 2H, NCH_2), 4.67 (brs, 2H, OCH_2), 5.21 (s, 1H, =C*H*), 6.85–6.88 (m, 2H, Ph), 7.22–7.26 (m, 2H, Ph). MS *m*/*z* 331, 333 (M^+); Anal. calcd for $C_{19}H_{22}ClNO_2$: C, 68.77; H, 6.63; N, 4.22; found C, 68.97; H, 6.78; N, 4.33%.

2.2.4. 3-*N*-[4′-(2′,4′-Dichlorophenoxy)but-2′-ynyl]*N*-methyl amino-5,5-dimethyl cyclohex-2-enone (4d). Yield 63%; white crystal mp 108°C; λ_{max}: (log ϵ) 222 (3.90), 294 (4.08) nm; IR (KBr) ν_{max}: 1100, 1240, 1410, 1540, 1600, 2940 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.15 (s, 2H, CH_2CO), 2.25 (s, 2H, =CCH_2), 2.92 (s, 3H, N*Me*), 4.01 (brs, 2H, NCH_2), 4.77 (brs, 2H, OCH_2), 5.21 (s, 1H, =C*H*), 6.93–6.96 (m, 1H, Ph), 7.17–7.20 (m, 1H, Ph), 7.37–7.38 (m, 1H, Ph); MS *m*/*z* 365, 369, 367 (M^+); Anal. calcd for $C_{19}H_{21}Cl_2NO_2$: C, 62.29; H, 5.73; N, 3.82; found C, 62.52; H, 5.96; N, 3.86%.

2.2.5. 3-*N*-[4′-(2′-Methylphenoxy)but-2′-ynyl]*N*-methyl amino-5,5-dimethyl cyclohex-2-enone (4e). Yield 65%; viscous liquid; λ_{max}: (log ϵ) 221 (4.84), 296 (5.37) nm; IR (KBr) ν_{max}: 1100, 1240, 1400, 1550, 1600, 2950 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 1.93 (s, 2H, CH_2CO), 2.19 (s, 3H, *Me*Ph), 2.27 (s, 2H, =CCH_2), 2.92 (s, 3H, N*Me*), 4.01 (brs, 2H, NCH_2), 4.71 (brs, 2H, OCH_2), 5.20 (s, 1H, =C*H*), 6.8–6.9 (m, 2H, Ph), 7.1–7.2 (m, 2H, Ph); MS *m*/*z* 311 (M^+); Anal. calcd for $C_{20}H_{25}NO_2$: C, 77.17; H, 8.03; N, 4.50; found C, 77.35; H, 8.29; N, 4.62%.

2.2.6. 3-*N*-[4′-(4′-Methylphenoxy)but-2′-ynyl]*N*-methyl amino-5,5-dimethyl cyclohex-2-enone (4f). Yield 64%; viscous liquid; λ_{max}: (log ϵ) 221 (4.98), 296 (5.40) nm; IR (KBr) ν_{max}: 1100, 1240, 1410, 1550, 1610, 2960 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.14 (s, 2H, CH_2CO), 2.26 (s, 2H, =CCH_2), 2.28 (s, 3H, *Me*Ph), 2.92 (s, 3H, N*Me*), 4.01 (brs, 2H, NCH_2), 4.66 (brs, 2H, OCH_2), 5.20 (s, 1H, =C*H*), 6.81–6.84 (m, 2H, Ph), 7.06–7.09 (m, 2H, Ph); MS *m*/*z* 311 (M^+); Anal. calcd for $C_{20}H_{25}NO_2$: C, 77.17; H, 8.03; N, 4.50; found C, 77.48; H, 8.25; N, 4.67%.

2.3. General procedures for the synthesis of compounds 5(a–f)

Compounds **4a–f** (0.2 g, 0.67 mmol) were refluxed in chlorobenzene (2 mL) for 12 h. The reaction mixture was then cooled and directly subjected to column chromatography over silica gel. Chlorobenzene was eluted out with petroleum ether. All the products **5(a–f)** were obtained when the column was eluted with benzene–ethyl acetate (4:1). Unchanged starting materials **4(a–f)** were also carefully eluted out with benzene–ethyl acetate (1:1). The yields were calculated on the basis of actual conversion of starting materials.

2.3.1. 7,7-Dimethyl-1,2,3,6,7,8-hexahydro-4-phenoxymethylene-1-methyl quinoline-5-one (5a). Yield 77%; white crystal, mp 138–140°C; λ_{max}: (log ϵ) 219 (3.97), 273 (4.06) nm; IR (KBr) ν_{max}: 1230, 1510, 1700, 2910 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (s, 6H, 2*Me*), 2.17 (s, 2H, CH_2CO), 2.36 (s, 2H, =CCH_2), 2.71 (t, *J*=6 Hz, 2H, NCH$_2$CH_2), 3.06 (s, 3H, N*Me*), 3.32 (t, *J*=6 Hz, 2H, NCH_2), 6.95–7.08 (m, 3H, Ph), 7.26–7.30 (m, 2H, Ph), 8.00 (s, 1H, =C*H*OAr); MS *m*/*z* 297 (M^+); Anal. calcd for $C_{19}H_{23}NO_2$: C, 76.76; H, 7.74; N, 4.71; found C, 76.98; H, 7.87; N, 4.83%.

2.3.2. 7,7-Dimethyl-1,2,3,6,7,8-hexahydro-4-(2′-chlorophenoxy)methylene-1-methylquinoline-5-one (5b). Yield 78%; viscous liquid; λ_{max}: (log ϵ) 220 (4.08), 275 (3.73) nm; IR (KBr) ν_{max}: 1240, 1520, 1700, 2920 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (s, 6H, 2*Me*), 2.17 (s, 2H, CH_2CO), 2.36 (s, 2H, =CCH_2), 2.66 (t, *J*=6 Hz, 2H, NCH$_2$CH_2), 3.07 (s, 3H, N*Me*), 3.34 (t, *J*=6 Hz, 2H, NCH_2), 6.91–7.26 (m, 4H, Ph), 8.03 (s, 1H, =C*H*OAr); MS *m*/*z* 331, 333 (M^+); Anal. calcd for $C_{19}H_{22}ClNO_2$: C, 68.77; H, 6.63; N, 4.22; found C, 68.98; H, 6.81; N, 4.33%.

2.3.3. 7,7-Dimethyl-1,2,3,6,7,8-hexahydro-4-(4′-chlorophenoxy)methylene-1-methyl quinoline-5-one (5c). Yield 75%; viscous liquid; λ_{max}: (log ϵ) 223 (4.11), 276 (4.02) nm; IR (KBr) ν_{max}: 1240, 1490, 1700, 2930 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (s, 6H, 2*Me*), 2.26 (s, 2H, CH_2CO), 2.36 (s, 2H, =CCH_2), 2.68 (t, *J*=6 Hz, 2H, NCH$_2$CH_2), 3.07 (s, 3H, N*Me*), 3.32 (t, *J*=6 Hz, 2H, NCH_2), 6.8–7.2 (m, 2H, Ph), 7.2–7.3 (m, 2H, Ph), 7.95 (s, 1H, =C*H*OAr); MS *m*/*z* 331, 333 (M^+); Anal. calcd for $C_{19}H_{22}ClNO_2$: C, 68.77; H, 6.63; N, 4.22; found C, 68.99; H, 6.85; N, 4.37%.

2.3.4. 7,7-Dimethyl-1,2,3,6,7,8-hexahydro-4-(2′,4′-dichlorophenoxy)methylene-1-methyl quinoline-5-one (5d). Yield 78%; viscous liquid; λ_{max}: (log ϵ) 219 (4.14), 282 (3.84) nm; IR (KBr) ν_{max}: 1230, 1410, 1550, 1600, 2940 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (s, 6H, 2*Me*), 2.17 (s, 2H, CH_2CO), 2.34 (s, 2H, =CCH_2), 2.73 (t, *J*=6 Hz, 2H, NCH_2CH_2), 3.08 (s, 3H, N*Me*), 3.34 (t, *J*=6 Hz, 2H, NCH_2), 6.94–7.26 (m, 2H, Ph), 7.31–7.34 (m, 1H, Ph), 8.02 (s, 1H, =C*H*OAr); MS *m/z* 365, 369, 367 (M^+); Anal. calcd for $C_{19}H_{21}Cl_2NO_2$: C, 62.29; H, 5.73; N, 3.82; found C, 62.47; H, 5.89; N, 3.91%.

2.3.5. 7,7-Dimethyl-1,2,3,6,7,8-hexahydro-4-(2′-methylphenoxy)methylene-1-methylquinoline-5-one (5e). Yield 78%; viscous liquid; λ_{max}: (log ϵ) 220 (4.04), 273 (3.84) nm; IR (KBr) ν_{max}: 1235,1510, 1700, 2930 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (s, 6H, 2*Me*), 2.19 (s, 2H, CH_2CO), 2.26 (s, 3H, *Me*Ph), 2.36 (s, 2H, =CCH_2), 2.71 (t, *J*=6 Hz, 2H, NCH_2CH_2), 3.06 (s, 3H, N*Me*), 3.33 (t, *J*=6 Hz, 2H, NCH_2), 6.81–6.86 (m, 1H, Ph), 6.87–7.04 (m, 3H, Ph), 7.99 (s, 1H, =C*H*OAr); MS *m/z* 311 (M^+); Anal. calcd for $C_{20}H_{25}NO_2$: C, 77.17; H, 8.03; N, 4.50; found C, 77.41; H, 8.19; N, 4.71%.

2.3.6. 7,7-Dimethyl-1,2,3,6,7,8-hexahydro-4-(4′-methylphenoxy)methylene-1-methylquinoline-5-one (5f). Yield 80%; viscous liquid; λ_{max}: (log ϵ) 221 (4.20), 272 (4.00) nm; IR (KBr) ν_{max}: 1230, 1500, 1700, 2920 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (s, 6H, 2*Me*), 2.19 (s, 2H, CH_2CO), 2.28 (s, 3H, *Me*Ph), 2.41 (s, 2H, =CCH_2), 2.70 (t, *J*=6 Hz, 2H, NCH_2CH_2), 3.06 (s, 3H, N*Me*), 3.32 (t, *J*=6 Hz, 2H, NCH_2), 6.7–7.08 (m, 4H, Ph), 7.92 (s, 1H, =C*H*OAr); MS *m/z* 311 (M^+); Anal. calcd for $C_{20}H_{25}NO_2$: C, 77.17; H, 8.03; N, 4.50; found C, 77.39; H, 8.27; N, 4.63%.

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