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Observations on the Synthesis of Functionalised Methyleneaziridines

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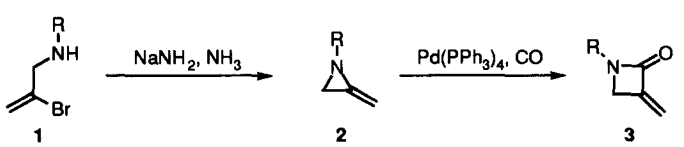
Abstract: *N*-Triphenylmethyl-2-methyleneaziridine **8** was synthesised from *N*-(2-bromo-2-propenyl)-amine **7** by treatment with sodium amide in liquid ammonia and its structure established using x-ray crystallography. Using modified conditions, (*S*)-*N*-(1-phenylethyl)-2-methyleneaziridine **9** was prepared in enantiomerically enriched form. Studies directed towards the synthesis of *N*-tosyl and *N*-Boc methyleneaziridines **14** and **15** respectively reveal limitations with this methodology.

Methyleneaziridines were first prepared by Pollard and Parcell by treatment of *N*-(2-bromoallyl)-alkylamines **1** with sodium amide in liquid ammonia.[1] However, it was several years later before Bottini and Johnson correctly identified the product from these reactions as the corresponding 1-alkyl-2-methyleneaziridines **2** (Scheme 1).[2] While less forcing reaction conditions (*n*-BuLi, THF, -70°C) have been described for the synthesis of the related 2-isopropylideneaziridines,[3] the original procedure still remains the only practical route to simple 1-alkyl-2-methyleneaziridines.[4] Investigations into the chemistry of methyleneaziridines have established that they undergo a variety of interesting and potentially useful chemical reactions.[5,6] For example, Alper and Hamel have shown that these strained heterocycles undergo palladium catalysed ring expansion reactions in the presence of carbon monoxide yielding the corresponding α-methylene-β-lactams **3**.[5]

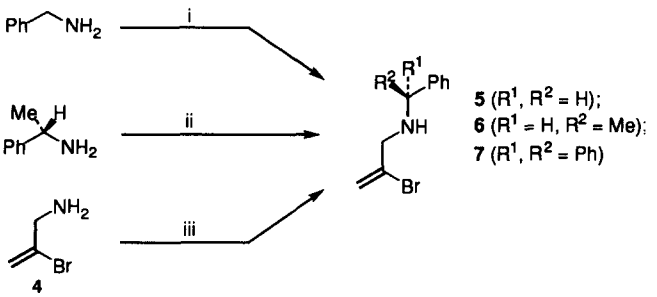


1 **2** **3**

Scheme 1

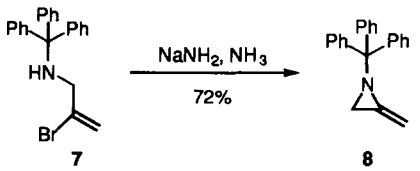
As a consequence of the forcing reaction conditions employed for the synthesis of methyleneaziridines, only derivatives containing relatively simple alkyl substituents on the aziridine nitrogen atom (*eg* Et, Bu^t, 1-adamantyl) have been prepared to date.[7] During work directed towards the development of new synthetic methods based upon methyleneaziridines, we required access to more highly functionalised compounds possessing substituents on the ring nitrogen atom that have the potential to be readily removed at a later stage in a reaction sequence. In this paper, we disclose some of our observations on the synthesis of such functionalised methyleneaziridines using the Pollard and Parcell methodology.

Our initial investigations focused on the preparation of *N*-benzyl substituted methyleneaziridines. This decision was made on the basis of the known stability of the *N*-benzyl group to a wide range of reaction conditions and the ease with which the benzylic C-N bond can be cleaved by hydrogenation.[8] Thus, three related methyleneaziridine precursors **5-7** were made differing only in the nature of the substituents attached to the benzylic carbon atom. Amine precursors **5** and **6** were prepared by alkylation of benzylamine and (*S*)-1-phenylethylamine respectively with commercially available 2,3-dibromopropene. While the *N*-trityl derivative **7** could not be prepared in this way using the more sterically encumbered triphenylmethylamine, an alternative approach involving coupling of *N*-(2-bromoallyl)-amine **4**[9] and trityl chloride readily furnished this precursor in good yield (Scheme 2).

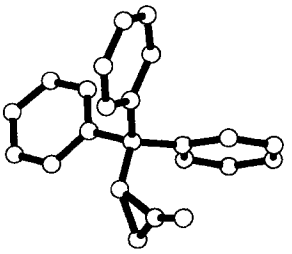


Scheme 2. *Reagents & Conditions:* (i) 2,3-dibrompropene, K_2CO_3, THF, reflux, 68% (**5**); (ii) 2,3-dibromopropene, K_2CO_3, Et_2O, reflux, 76% (**6**); (iii) Ph_3CCl, Et_3N, CH_2Cl_2, 88% (**7**).

With the requisite precursors in hand, we were ready to examine the conversion of these materials into the corresponding methyleneaziridines employing the procedure described by Pollard and Parcell.[1] Treatment of the *N*-benzyl derivative **5** with differing amounts of sodium amide (1.1-15 equiv) for varying reaction times (5-120 min) lead to the formation of complex mixtures of products from which no appreciable quantities of the desired methyleneaziridine could be isolated. Since closely related compounds such as *N*-ethyl methyleneaziridine can be made using this chemistry, we reasoned that the increased acidity of the hydrogens on the benzylic carbon atom may be causing the detrimental side reactions. To test this hypothesis, we next chose to examine the cyclisation of *N*-trityl derivative **7** which has no such acidic hydrogens. In this instance, we were delighted to discover that treatment of this substrate with 15 equivalents of sodium amide in liquid ammonia for 6 hours smoothly lead to the formation of desired methyleneaziridine **8** in 72% yield (Scheme 3). The structure of this compound was unambiguously established using x-ray crystallography. While Quast *et al* have published an x-ray crystallographic study of 1-(1-adamantyl)-2-isopropylidineaziridine,[10] we believe this represents the first crystallographic study of a simple methyleneaziridine.[11]

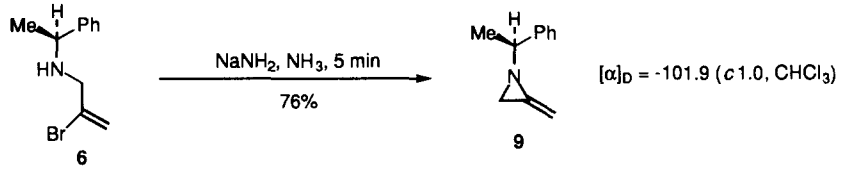


Scheme 3



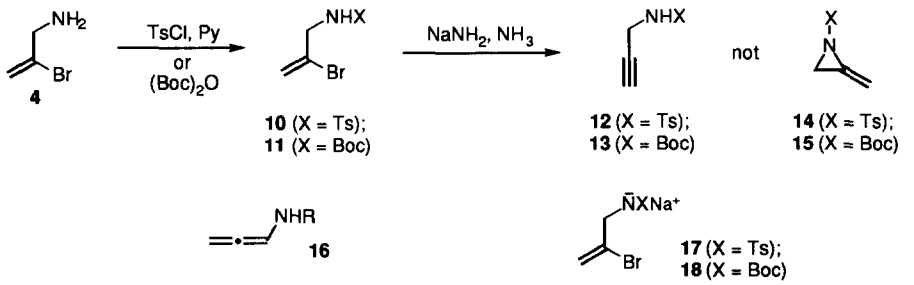
X-Ray Crystal Structure of *N*-(triphenylmethyl)-2-methyleneaziridine **8**

Using modified reaction conditions, amine **6**, derived from (*S*)-1-phenylethylamine, can be cyclised to methyleneaziridine **9** in 76% yield. The reaction time is critical, such that if this cyclisation is allowed to proceed for more than 5 minutes dramatically lower yields of the product are obtained. Importantly, chiral shift NMR studies performed using (+)-[Eu(hfc)$_3$] established that this cyclisation proceeds without detectable racemisation at the asymmetric centre. The successful cyclisation of **6** into **9** is particularly notable in view of the problems encountered with the closely related precursor **5** (*Vide Supra*). We speculate that the differing reactivity exhibited by these compounds may, in part, be due to the reduced acidity and accessibility of the benzylic hydrogen atom of **6** compared with those contained within **5**.



Scheme 4

We were interested in establishing whether the Pollard and Parcell chemistry could be used to access other types of methyleneaziridines possessing potentially cleavable groupings on the aziridine nitrogen atom such as **14** and **15** (Scheme 5). The requisite precursors **10** and **11** were prepared using standard chemistry from *N*-(2-bromoallyl)-amine **4**.[9] Unfortunately, our attempts to facilitate cyclisation of **10** to methyleneaziridine **14** were unsuccessful. Using just 1.1 equivalent of sodium amide in liquid ammonia the starting amine **10** was recovered unchanged, while use of excess sodium amide (2.1-15 equivalents) facilitated clean conversion to the corresponding acetylene **12**. Similar behaviour was exhibited by the *N*-Boc derivative **11** which was converted to acetylene **13** rather than the desired methyleneaziridine **15**. The generation of acetylenes as minor side products in this type of cyclisation reaction has been noted previously,[2] although the failure to form any of the desired methyleneaziridines in these reactions was surprising. Bottini and Olsen have presented evidence that suggests that methyleneaziridines are formed in this cyclisation reaction by an elimination-addition mechanism involving allenes such as **16**.[12] We believe that the increased acidity of the NH group within amines **10** and **11** means that they are rapidly and essentially irreversibly deprotonated to the corresponding sodium anions **17** and **18** respectively. We suggest that electrostatic repulsion deters proton abstraction from the adjacent carbon atom in these anions, a requirement for the formation of **16** (R = *p*-$SO_2C_6H_4CH_3$ or $CO_2{}^tBu$). Instead, abstraction occurs at the remote alkene terminus producing the corresponding acetylenes. These findings suggest that the Pollard and Parcell methodology cannot be used to make methyleneaziridines possessing electron withdrawing groups on the nitrogen atom.



Scheme 5

In summary, we have devised an efficient approach to methyleneaziridines **8** and **9** which possess potentially cleavable groups on the aziridine nitrogen atom. Since 1-phenylethylamine is cheap and readily available in both enantiomerically pure forms, we envisage that homochiral methyleneaziridine **9** may represent a useful building block for asymmetric synthesis. Studies to explore the potential of methyleneaziridines **8** and **9** in organic synthesis are ongoing and will be disclosed in due course.

EXPERIMENTAL

General. ^{1}H and ^{13}C nmr spectra were recorded at 250 MHz and 62.5 MHz on a Bruker ACF-250 instrument, at 270 MHz and 67.5 MHz on a Jeol GSX- 270 instrument, and at 400 MHz and 100 MHz on a Bruker DPX 400 instrument with either tetramethylsilane or residual protic solvent as the internal standard. Infrared spectra were recorded on a Nicolet FT-205 spectrometer. Mass Spectra were recorded on a Kratos MS80 or VG analytical ZAB-E instrument under EI conditions unless otherwise stated. All solvents and reagents were purified by standard means. All reactions were performed using oven dried glassware under an atmosphere of nitrogen unless otherwise stated.

***N*-(2-bromo-2-propenyl)-benzylamine (5).** To a stirred solution of benzylamine (5.0 g, 46.7 mmol) in THF (40 ml) was added 2,3-dibromopropene (4.66 g, 23.3 mmol) dropwise and the resulting solution heated under reflux for 12 hours. Potassium carbonate (12.8 g, 91.4 mmol) was then added and the mixture heated for a further 12 hours. On cooling, the mixture was filtered and the precipitate washed with diethyl ether. The filtrate was washed with 10% sodium hydroxide (2 x 10 ml) and extracted with diethyl ether (3 x 25 ml). The combined organic extracts were washed with water, dried over $MgSO_4$ and solvent removed under reduced pressure. Column chromatography (5% ethyl acetate / petroleum ether using silica pretreated with triethylamine) gave **5** (3.60 g, 68%) as an orange oil. ν_{max} (film) 3950, 2900, 1626, 1454, 736 cm^{-1}; δ_H (270 MHz, $CDCl_3$) 7.35-7.25 (5H, m, Ph), 5.81 (1H, d, 1.6 Hz, =CH), 5.61 (1H, d, 1.6 Hz, =CH), 3.75 (2H, s, CH_2Ph), 3.48 (2H, s, CH_2N), 1.76 (1H, bs, NH); δ_C (62.5 MHz, $CDCl_3$) 140.1 (s), 133.9 (s), 129.0 (d), 128.7 (d), 127.7 (d), 118.3 (t), 57.0 (t), 51.9 (t); *m/z* 227/225 (M^+), 146 (M^+-Br), 120, 91, 77; Observed (M^+): 225.0153; $C_{10}H_{12}BrN$ requires 225.0154.

(*S*)-*N*-(2-bromo-2-propenyl)-1-phenylethylamine (6). To a suspension of (*S*)-1-phenylethylamine (10.0 g, 82.5 mmol) and potassium carbonate (5.60 g, 41.0 mmol) in diethyl ether (100 ml) was added 2,3 dibromopropene (8.20 g, 40.5 mmol) dropwise and the mixture refluxed for 21 hours. On cooling, the mixture was filtered and the resulting solution washed with 10% sodium hydroxide solution, dried over $MgSO_4$ and concentrated under reduced pressure. Column chromatography (25% diethyl ether / petroleum ether) gave **6** (7.50 g, 76%) as a pale yellow oil. $[\alpha]_D$ = -30.8 (*c* 1.0, $CHCl_3$); ν_{max} (film) 3316, 3085, 2825, 1603, 699, 659 cm^{-1}; δ_H (250 MHz; $CDCl_3$) 7.98-7.19 (5H, m, Ph), 5.65 (1H, d, 1.5 Hz, =CH), 5.54 (1H, d, 1.5 Hz, =CH), 3.85 (1H, q, 7.5 Hz, CH), 3.35 (1H, d, 15.6 Hz, NCH_2), 3.24 (1H, d, 15.6 Hz, NCH_2), 1.78 (1H, s, NH), 1.39 (3H, d, 7.5 Hz, CH_3); δ_C (62.9 MHz; $CDCl_3$) 144.8 (s), 133.8 (s), 128.4 (d), 127.0 (d), 126.7 (d) 117.7 (t), 55.5 (d), 55.0 (t), 24.2 (q); *m/z* 242/240 (MH^+), 224, 160 (M^+-Br), 144, 120, 105. Observed (MH^+): 240.0388, requires $C_{11}H_{15}BrN$ requires 240.0388.

***N*-(2-bromo-2-propenyl)-triphenylmethylamine (7).** To a stirred solution of 2-bromoallylamine[9] (2.43 g, 19.93 mmol) in dichloromethane (40 ml) was added triphenylmethyl chloride (5.56 g, 19.93 mmol) and triethylamine (2.8 ml, 19.93 mmol). After 48 hours, water (20 ml) was added and the reaction mixture extracted with dichloromethane (3 x 15 ml). The combined organic extracts were washed with water (2 x 15 ml), dried over Na_2SO_4 and the solvent removed under reduced pressure. Recrystallisation from petroleum ether / ethyl acetate gave **7** (6.61 g, 88%) as a white solid (m.p. 99-103°C). ν_{max} (film) 3400, 3050, 1420, 1275, 750 cm^{-1}; δ_H (270 MHz, $CDCl_3$) 7.57-7.42 (6H, m, Ph), 7.34-7.16 (9H, m, Ph), 6.08 (1H, d, 1.5 Hz, =CH), 5.56 (1H, d, 1.5 Hz, =CH), 2.97 (2H, s, NCH_2), 2.07 (1H, bs, NH); *m/z* 380/378 (MH^+), 298 (M^+-Br), 243 (Ph_3C^+); Observed (M^+): 377.0829; $C_{22}H_{21}BrN$ requires 377.0779.

***N*-(Triphenylmethyl)-2-methyleneaziridine (8).** To a three necked flask fitted with dry ice condenser, calcium chloride drying flask and gas inlet was added sodium amide (11.16 g, 286 mmol) and the system flushed with ammonia. A dry ice / acetone mixture was added to the condenser and ammonia (100 ml) condensed into the flask, **7** (7.19 g, 19.07 mmol) was added dropwise to this mixture and the resulting solution stirred for 6 hours. The reaction mixture was diluted with diethyl ether (15 ml) and quenched by the dropwise addition of water (15 ml) (CAUTION). After the ammonia had evaporated, water (10 ml) and diethyl ether (20 ml) were added and the mixture stirred for two minutes. The organic phase was separated and the aqueous phase extracted with diethyl ether (3 x 20 ml). The combined organic extracts were washed with 10% sodium hydroxide (2 x 10 ml), then water (2 x 10 ml), dried over $MgSO_4$ and the solvent removed under reduced pressure. Recrystallisation from petroleum ether / ethyl acetate gave **8** (4.10 g, 72%) as a white crystalline solid (m.p. 119-124°C). ν_{max} (film) 3050, 2900, 1770, 1450, 1275 cm^{-1}; δ_H (270 MHz, $CDCl_3$) 7.52-7.40 (6H, m, Ph), 7.39-7.19 (9H, m, Ph), 4.44 (1H, d, 1.2 Hz, =CH), 4.33 (1H, m, =CH), 1.81 (2H, d, 0.5 Hz, aziridine CH_2); *m/z* 298 (MH^+), 243 (Ph_3C^+); Found: C 88.69, H 6.69, N 4.36%; $C_{22}H_{19}N$ requires: C 88.85, H 6.44, N 4.71%.

(*S*)-*N*-(1-Phenylethyl)-2-methyleneaziridine (9). A three neck flask was fitted with a dry ice condenser and a gas inlet tube. The system was purged with dry ammonia then sodium amide (11.5 g, 310 mmol) was added and the system again flushed with ammonia. After ammonia (100 ml) was condensed into the flask, **6** (5.0 g, 20.7 mmol) was added dropwise. During this addition the reaction mixture changed colour from grey to dark brown. After 5 minutes, the mixture was diluted with diethyl ether (20 ml) and quenched by the dropwise addition of water (CAUTION). After standing overnight, diethyl ether (50 ml) was added to the residue and the mixture stirred for 2 minutes. The organic layer was separated, washed with 10% sodium hydroxide solution, dried over $MgSO_4$ and concentrated under reduced pressure. Purification by bulb-to-bulb distillation (*ca* 95°C / 5mmHg) gave **9** (2.50 g, 76%) as a clear oil. $[\alpha]_D$ = -101.9 (*c* 1.0, $CHCl_3$); ν_{max} (film) 3076, 1749, 1599, 1449, 699 cm^{-1}; δ_H (250 MHz; $CDCl_3$) 7.38-7.25 (5H, m, Ph), 4.64-4.62 (2H, m, $=CH_2$), 2.93 (1H, q, 6.6 Hz, CH), 2.05 (1H, d, 2.5 Hz, aziridine CH), 1.87 (1H, d, 2.5 Hz, aziridine CH), 1.01 (3H, d, 6.5 Hz, CH_3); δ_C (62.5 MHz; $CDCl_3$) 143.7 (s) 132.9 (s), 128.0 (d), 127.2 (d), 126.7 (d), 82.9 (t), 68.4 (d), 29.8 (t), 23.5 (q); *m/z* 159 (M^+), 118, 105, 51, 31; Observed (M^+): 159.1048; $C_{11}H_{13}N$ requires 159.1048.

X-ray crystal data for *N*-triphenylmethyl-2-methyleneaziridine (8). $C_{22}H_{19}N$, M = 297.40, hexagonal, a = 9.903(2), c = 29.326(2)Å, V = 2490.9(9)Å^3, space group $P6_5$ (#170), Z = 6, D_{calc} = 1.189gcm^{-3}, Cu radiation, λ = 1.54178Å, μ(Cu-K$_\alpha$) = 5.21 cm^{-1}, $F(000)$ = 948. Data were measured on a Rigaku AFC7S diffractometer with Cu-K$_\alpha$ radiation (graphite monochromator) using ω-scans. A crystal of dimensions 0.13 x 0.10 x 0.28 mm was used. A total of 1525 reflections were measured, 1287 (R_{int} = 0.022) were unique, of which 920 were considered observed ($I > 2.00\sigma(I)$). The data were corrected for Lorentz, polarisation and absorption factors. The structure was solved by direct methods. The non-hydrogen atoms were refined anisotropically. The positions of the hydrogen atoms were idealised, C-H = 0.95Å, and assigned isotropic thermal parameters. Refinement was by full-matrix least-squares to R = 0.046, R_w = 0.040 [w^{-1} = $\sigma^2(F_o)$]. The maximum and minimum residual electron density in the final ΔF map were 0.16 and -0.17 eÅ^{-3} respectively. The maximum shift/error in the final refinement was 0.13. Computations were carried out using the teXsan crystal structure analysis package (Molecular Structure Corporation, 1985 & 1992).

Chiral analysis of (*S*)-*N*-(1-phenylethyl)-2-methyleneaziridine (9). ^{1}H NMR analysis at 400MHz of (±)-**9** (3.0 mg, 18.8 μmol) in the presence of (+)-[Eu(hfc)$_3$] (0.35 molar equivalents) in d-chloroform produced two overlaping doublets due to the methyl group at δ_H 1.59 (d, 6.6 Hz) and 1.58 (d, 6.6 Hz). Repetition of this procedure using (*S*)-**9** indicated the presence of only one enantiomer, addition of (±)-**9** to this sample resulted in the appearance of the second set of resonances.

4-Methyl-*N*-(2-bromo-2-propenyl)-benzenesulphonamide (10). To a stirred solution of 2-bromoallylamine[9] (1.5 g, 12.30 mmol) and pyridine (1.45 ml, 18.07 mmol) in chloroform (15 ml) at 0°C was added *p*-toluenesulphonyl chloride (2.29 g, 12.01 mmol) portionwise. After 24 hours, the mixture was diluted with dichloromethane (20 ml) and washed with aqueous hydrochloric acid. The organic phase was separated and the aqueous layer re-extracted with dichloromethane. The combined organic extracts were washed with water (2 x 10 ml), then sodium hydrogen carbonate (3 x 15 ml), dried over $MgSO_4$ and the solvent removed under reduced pressure. Recrystallisation from n-hexane / ethyl acetate gave **10** (2.55 g, 75 %) as a yellow solid (m.p. 68-71°C). ν_{max} (film) 3300, 1640, 1600, 1350, 1150 cm^{-1}; δ_H (270 MHz, $CDCl_3$) 7.77-7.72 (2H, m, Ph), 7.32-7.26 (2H, m, Ph), 5.78 (1H, m, =CH), 5.47 (1H, m, =CH), 4.83 (1H, bt, 4.5 Hz, NH), 3.86 (2H, m, CH_2), 2.43 (3H, s, CH_3); δ_C (67.5 MHz, $CDCl_3$): 145.0 (s), 138.1 (s), 130.0 (s), 128.5 (d), 128.1 (d), 119.1 (t), 51.0 (t), 22.3 (q); *m/z* 292/290 (MH^+), 155, 134, 91; Found: C 41.29, H 4.08, N 4.71%, $C_{10}H_{12}BrNSO_2$ requires C 41.39, H 4.17, N 4.83%.

Attempted preparation of *N*-(4-methylbenzenesulphonyl)-2-methyleneaziridine (14). To a three necked flask fitted with dry ice condenser, calcium chloride drying flask and gas inlet was added sodium amide (2.02 g, 51.79 mmol) and the system flushed with ammonia. A dry ice / acetone mixture was added to the condenser and ammonia (30 ml) condensed into the flask, **10** (1.0 g, 3.44 mmol) was added dropwise to this mixture and the resulting solution stirred for 5 hours. The reaction mixture was diluted with diethyl ether (15 ml) and quenched by the dropwise addition of water (15 ml) (CAUTION). After the ammonia had evaporated, water (10 ml) and diethyl ether (20 ml) were added and the mixture stirred for two minutes. The organic phase was separated and the aqueous phase extracted with diethyl ether (3 x 20 ml). The organic

extracts were washed with 10% sodium hydroxide (2 x 10 ml), then water (2 x 10 ml), dried over $MgSO_4$ and the solvent removed under reduced pressure to give 4-methyl-*N*-(2-propynyl)-benzenesulphonamide **12** (0.59 g, 81%) as an orange oil. ν_{max} (film) 3350, 3075, 2400, 2300, 1270, 745 cm^{-1}; δ_H (270 MHz, $CDCl_3$) 7.80-7.64 (2H, m, Ph), 7.36-7.27 (2H, m, Ph), 4.51 (1H, t, 4.5 Hz, NH), 3.83 (2H, dd, 6.1, 2.7 Hz, $HNCH_2$), 2.43 (3H, s, CH_3) 2.19 (1H, t, 2.7 Hz, C≡CH); δ_C (67.5 MHz, $CDCl_3$): 143.8 (s), 136.4 (s), 129.7 (d), 127.3 (d), 76.5 (s), 73.0 (d), 32.9 (t), 21.5 (q); *m/z* 210 (MH^+), 155, 139, 91, 54; Observed (M^+): 209.0511; $C_{10}H_{11}NO_2S$ requires 209.0511.

***N-tert*-Butoxycarbonyl-*N*-(2-bromo-2-propenyl)-amine (11).** To a solution of 2-bromoallylamine[9] (0.15 g, 1.13 mmol) and triethylamine (0.12 g, 1.21 mmol) in dichloromethane (10 ml) was added di-*tert* butyl dicarbonate (0.24 g, 1.10 mmol). After stirring for 24 hours, the reaction was quenched by addition of water and extracted with dichloromethane (3 x 15 ml). The combined organic extracts were washed with water then sodium hydrogen carbonate, dried with $MgSO_4$ and the solvent removed under reduced pressure. Column chromatography (5% ethyl acetate / petroleum ether) gave **11** (0.20 g, 77%) as a white crystalline solid (m.p. 62-65°C). ν_{max} (film) 3400, 3350, 1740, 1640 cm^{-1}; δ_H (270 MHz, $CDCl_3$) 5.81 (1H, d, 1.5 Hz, =CH), 5.53 (1H, m, =CH), 4.97 (1H, bs, NH), 3.95 (2H, m, $NHCH_2$), 1.45 (9H, s, ^{t}Bu); δ_C (67.5 MHz, $CDCl_3$) 155.3 (s), 130.5 (s), 116.9 (s), 79.9 (t), 48.5 (t), 28.2 (q); *m/z* 238/236 (MH^+); Observed (M^+-Me): 219.9968; $C_7H_{11}BrNO_2$ requires 219.9973.

Attempted preparation of *N-tert*-butoxycarbonyl-2-methyleneaziridine (15). To a three necked flask fitted with dry ice condenser, calcium chloride drying flask and gas inlet was added sodium amide (0.19 g, 5.10 mmol) and the system flushed with ammonia. A dry ice / acetone mixture was then added to the condenser and the ammonia condensed into the flask (30 ml), **11** (80 mg, 0.34 mmol) was added dropwise to this mixture and the resulting solution stirred for 5 minutes. The reaction mixture was diluted with diethyl ether (15 ml) and quenched by the dropwise addition of water (15 ml) (CAUTION). After the ammonia had evaporated, water (10 ml) and diethyl ether (20 ml) were added and the mixture stirred for two minutes. The organic phase was separated and the aqueous phase extracted with diethyl ether (3 x 10 ml). The combined organic extracts were washed with 10% sodium hydroxide (2 x 10 ml), then water (2 x 10 ml), dried over $MgSO_4$ and the solvent removed under reduced pressure to give *N-tert*-butoxycarbonyl-2-propynylamine **13** (40 mg, 76%) as a pale yellow oil. δ_H (270 MHz, $CDCl_3$) 4.70 (1H, bs, NH), 3.92 (2H, d, 2.4 Hz, NCH_2), 2.22 (1H, t, 2.5 Hz, C≡CH), 1.45 (9H, s, ^{t}Bu); *m/z* 156 (MH^+), 117. Data consistent with those reported for this compound in the literature.[13]

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