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ABOUT THE FACTORS WHICH GOVERN THE RING-OPENING OF α-LACTAMS WITH BENZYLAMINE: I. THE RELATIVE STABILITY OF THE α-LACTAM AND THE SUBSTITUENT ON NITROGEN

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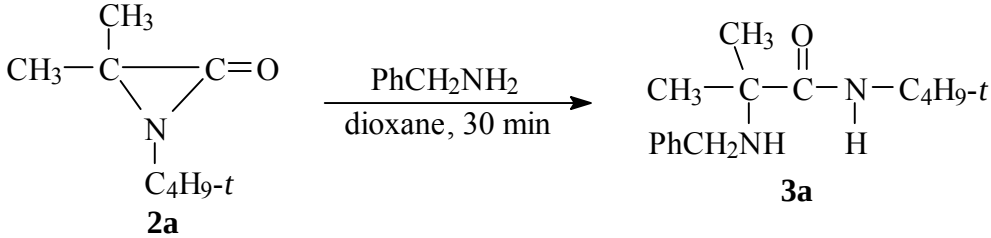
Abstract-Eight α-lactams (aziridinones) of varying stability, one of them previously unreported, were synthesized and reacted with benzylamine. Three of the α-lactams, 1-(1-adamantyl)-3,3-dimethylaziridinone **(2j)**, 3,3-dimethyl-1-triphenylmethylaziridinone **(2m)**, and 3-phenyl-1-triphenylmethyl-aziridinone **(2o)** gave α-benzylaminoamides **(3j**, **3m**, **3o)** as products, indicating C_3-N bond cleavage. Four α-lactams, 1,3-di-*tert*-butylaziridinone **(2g)**, 1-*tert*-butyl-3-triphenylmethylaziridinone **(2i)**, 1-(1-adamantyl)-3-*tert*-butylaziridinone **(2k)**, and 1-(1-adamantyl)-3-triphenylmethylaziridinone **(2*l*)** yielded *N*-benzylamides **(4g**, **4i**, **4k**, **4*l*)**, resulting from C_2-N bond cleavage. 3-(1-Adamantyl)-1-triphenylmethylaziridinone **(2n)** gave a mixture of both types of adduct **(3n, 4n)**. Based on these experimental results, two important factors which govern the ring-opening of α-lactams are the relative stability of the α-lactam and the substituent on nitrogen.

INTRODUCTION

A great deal of effort has been expended in trying to unravel the factors that govern the regioselectivity in nucleophilic ring-opening of stable α-lactams (aziridinones),[1-4] as well as α-lactam intermediates[5,6] generated *in situ*. However, no single theory has emerged to date that would satisfactorily explain all the experimental results.

For example,[7] the reaction of 1-*tert*-butyl-3,3-dimethylaziridinone **(2a)** with benzylamine gave the α-benzylamino-*N*-*tert*-butylamide **(3a)**, as the only product, in good yield (Scheme 1), indicating a ring opening by cleavage of the C_3-N bond. It should be noted that this is the *general* ring-opening of α-lactams with protic non-ionic nucleophiles such as water, alcohols, thiols, and amines,[1,2] although some exceptions have been observed.[1-4]

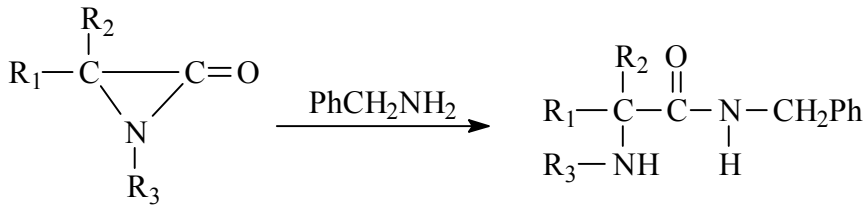
Scheme 1. The reaction of 1-*tert*-butyl-3,3-dimethylaziridinone **(2a)** with benzylamine.



2a **3a**

In 1997, Shimazu *et al.* reported[8] that four α-lactams **(2b-e)** substituted at C_3 and/or the nitrogen by the bulky α,α-dimethylbenzyl group, as well as 3-*tert*-butyl-1-triphenylmethylaziridinone **(2f)**, gave *N*-benzylamides **(4b-f)** with benzylamine, resulting from C_2-N bond cleavage (Scheme 2).

Scheme 2. The reaction of α-lactams **(2b-e)** with benzylamine.



2b-e **4b-e**

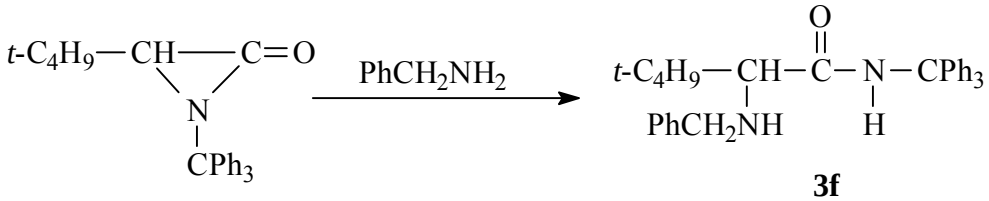
α-Lactam*	Product*	R_1	R_2	R_3
2b	4b	Dmb	H	*t*-C_4H_9
2c	4c	Dmb	H	Ad
2d	4d	*t*-C_4H_9	H	Dmb
2e	4e	Dmb	H	Dmb

*Ad denotes 1-adamantyl, $C_{10}H_{15}$-

Dmb denotes α, α-dimethylbenzyl, $C_6H_5C(CH_3)_2$-

In contradiction to the latter report, we have demonstrated[9] that the product of the reaction of 3-*tert*-butyl-1-triphenylmethylaziridinone **(2f)** with benzylamine is rather the α-benzylamino-*N*-tritylamide **(3f)** (Scheme 3).

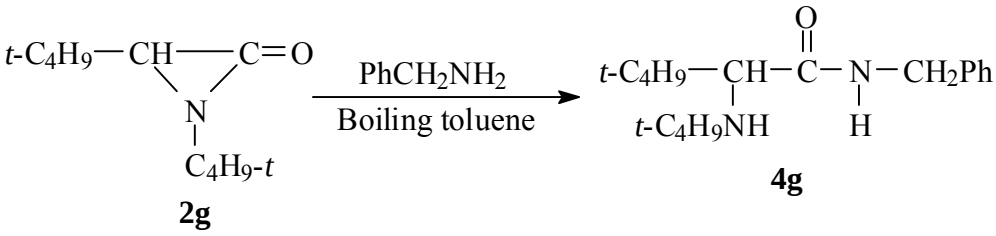
Scheme 3. The reaction of 3-*tert*-butyl-1-triphenylmethylaziridinone (**2f**) with benzylamine.



2f **3f**

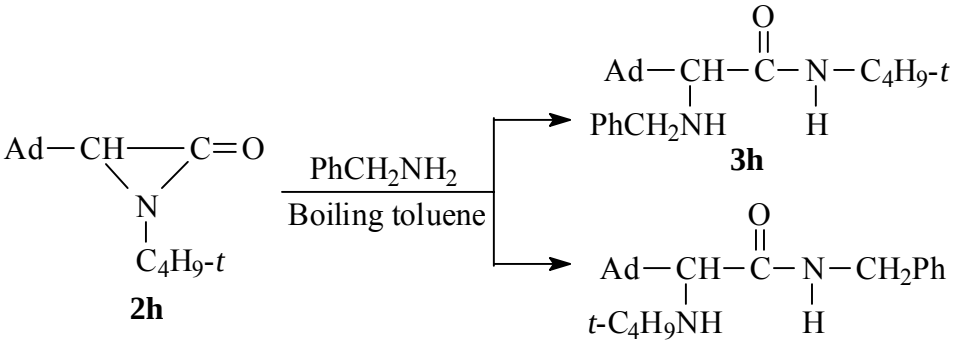
In 1998, Talaty *et al*. reported[10] that 1,3-di-*tert*-butylaziridinone **(2g)**[11] with benzylamine in boiling toluene gave the *N*-benzylamide adduct **(4g)** in 92% yield (Scheme 4), while 3-(1-adamantyl)-1-*tert*-butylaziridinone **(2h)**[12] gave a 1:4.88 mixture of the α-benzylaminoamide **(3h)** and *N*-benzylamide **(4h)**, respectively (Scheme 5), in 89% overall yield.

Scheme 4. The reaction of 1,3-di-*tert*-butylaziridinone **(2g)** with benzylamine.



2g **4g**

Scheme 5. The reaction of 3-(1-adamantyl)-1-*tert*-butylaziridinone **(2h)** with benzylamine.

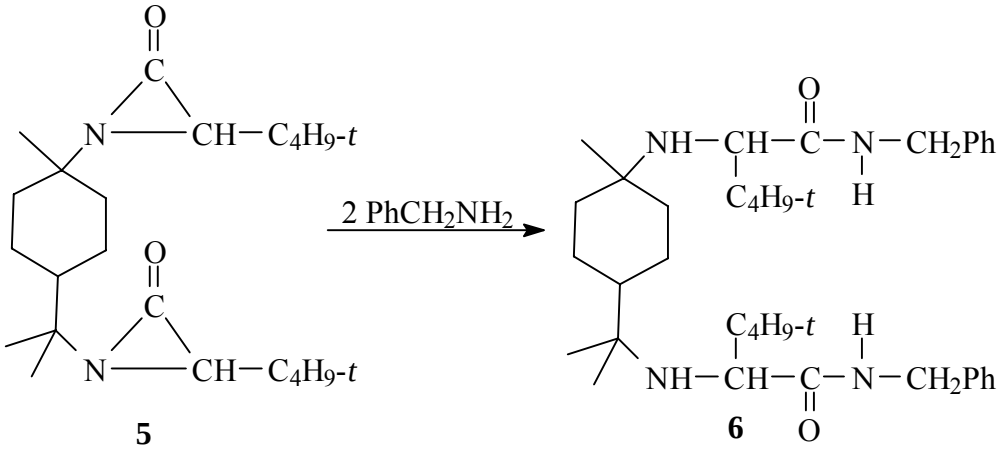


2h **3h**

4h (major product)

However, no important structure-proving spectral data for these products **(3h**, **4g-h)** were given. Lastly, in a recent report[13] it was revealed that the reaction of a *bis*-α-lactam, *cis*-1, 1'(*p*-menth-1,8-ylene)*bis*(3-*tert*-butyl-2-aziridinone) **(5)** with benzylamine also leads to the *bis*-*N*, *N*'-benzylamide type product **(6)** (Scheme 6).

Scheme 6. The reaction of *cis*-1, 1'(*p*-menth-1,8-ylene)*bis*(3-*tert*-butyl-2-aziridinone) **(5)** with benzylamine.



5 **6**

In summary, before this investigation, nine α-lactams **(2a-h**, **5)** have been reacted with benzylamine, with results that strike one as conflicting, contradictory, and confusing. Sometimes C_2-N bond cleavage occurs, while other times the C_3-N bond is cleaved.

RESULTS AND DISCUSSION

The present investigation was undertaken with the purpose to:

(a) gain an understanding of the causes why some α-lactams with benzylamine yield α-benzylaminoamides **(3)** while others give *N*-benzylamides **(4)**,

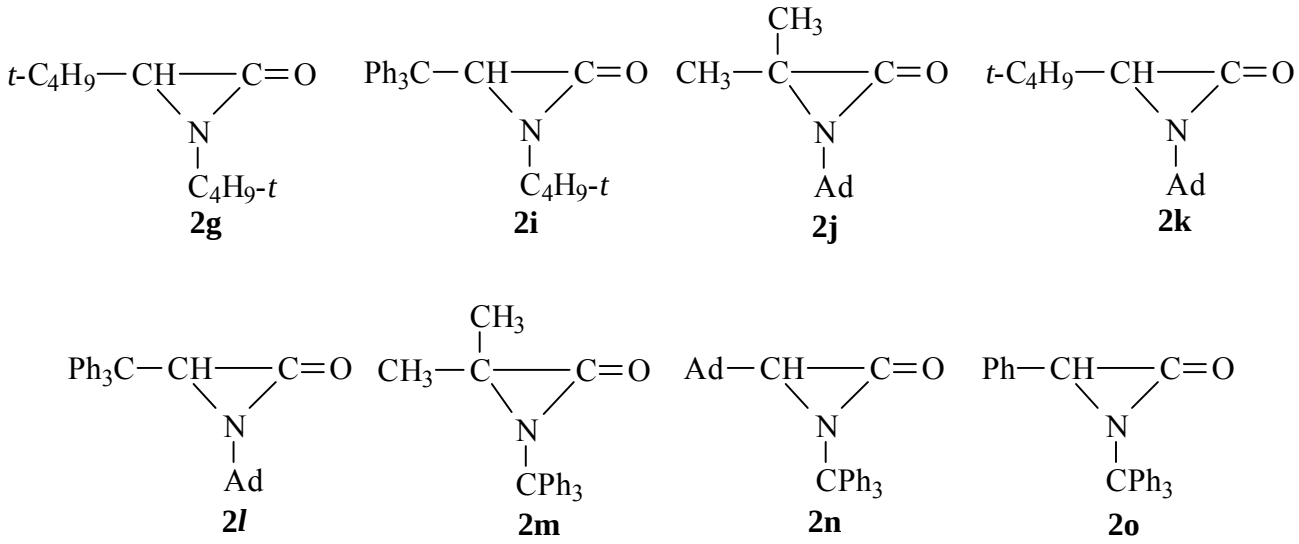
(b) elucidate the structural parameters that determine which product will form.

In order to achieve these goals, the reaction conditions, such as solvent (THF) and temperature (rt), as well as the amount of the nucleophilic reagent (benzylamine), unless otherwise indicated, were set constant for all of the α-lactam ring-opening reactions reported in this study. Only the reacting α-lactam was varied.

1. Synthesis of the α-lactams.

Eight aziridinones **(2g**, **i-o**, Table 1**)**, one **(2o)** of them new (previously unreported), were synthesized.

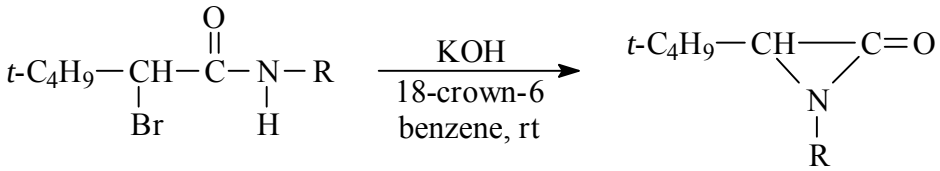
Table 1. The eight aziridinones synthesized for this investigation*.



*Ad denotes 1-adamantyl.

1,3-Di-*tert*-butylaziridinone[11] **(2g)** and 1-(1-adamantyl)-3-*tert*-butylaziridinone[14] **(2k)** were synthesized by the phase-transfer catalysis (PTC) method introduced by Scrimin *et al.*[15] (Scheme 7).

Scheme 7. The PTC synthesis of α-lactams (**2g** and **2k**).

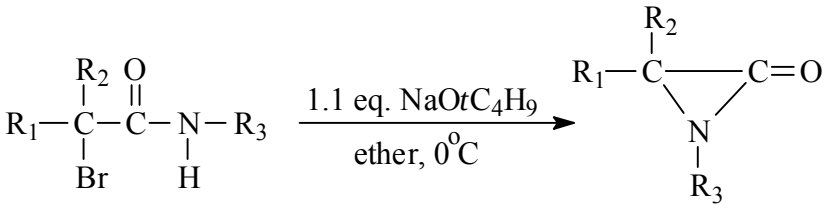


1g: R= *t*-C_4H_9
1k: R= Ad

2g: R= *t*-C_4H_9
2k: R= Ad

1-(1-Adamantyl)-3,3-dimethylaziridinone[16] **(2j)** was synthesized by 1,3-dehydrobromination of *N*-(1-adamantyl)-2-bromo-2-methylpropanamide **(1j)** with sodium *tert*-butoxide ($NaOtC_4H_9$), using the slight but important modification introduced by Simig *et al.*[17] (Scheme 8). The same procedure was used in the preparation of 3,3-dimethyl-1-triphenylmethylaziridinone **(2m)**[18] and 3-phenyl-1-triphenyl-methylaziridinone **(2o)**.

Scheme 8. Synthesis of α-lactams **(2j**, **2m**, **2o)**.



1j, **1m**, **1o** **2j**, **2m**, **2o**

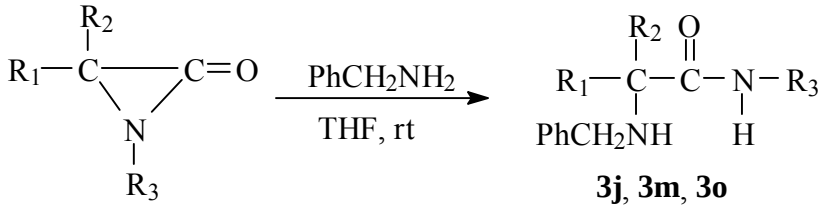
Amide	α-Lactam	R_1	R_2	R_3
1j	**2j**	CH_3	CH_3	Ad
1m	**2m**	CH_3	CH_3	Ph_3C
1o	**2o**	Ph	H	Ph_3C

The synthesis, physical and spectral properties, limits of thermal stability and some reactions of 1-*tert*-butyl-3-triphenylmethylaziridinone **(2i)**, 1-(1-adamantyl)-3-triphenylmethylaziridinone **(2*l*)**, and 3-(1-adamantyl)-1-triphenylmethylaziridinone **(2n)** are reported[18] elsewhere.

2. Reaction of the α-lactams with benzylamine.

Benzylamine, with a pK_b of 4.64, is a good nucleophile, which reacts even with the most stable α-lactams at room temperature. The reaction time varies from a few min to 120 h. Three of the α-lactams, 1-(1-adamantyl)-3,3-dimethylaziridinone **(2j)**, 3,3-dimethyl-1-triphenylmethylaziridinone **(2m)**, and 3-phenyl-1-triphenylmethylaziridinone **(2o)** gave α-benzylaminoamides **(3j**, **3m**, **3o)** resulting from cleavage of the C_3-N bond (Scheme 9).

Scheme 9. The reaction of α-lactams **(2j**, **2m**, and **2o)** with benzylamine.



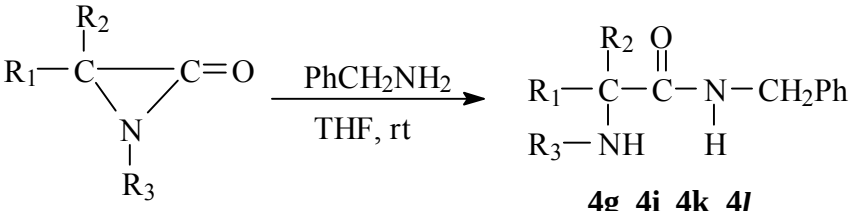
2j, **2m**, **2o** **3j**, **3m**, **3o**

α-Lactam	Product	R_1	R_2	R_3
2j	**3j**	CH_3	CH_3	Ad
2m	**3m**	CH_3	CH_3	Ph_3C
2o	**3o**	Ph	H	Ph_3C

Four α-lactams, namely 1,3-di-*tert*-butylaziridinone **(2g)**, 1-*tert*-butyl-3-triphenylmethylaziridinone **(2i)**, 1-(1-adamantyl)-3-*tert*-butylaziridinone **(2k)**, and 1-(1-adamantyl)-3-triphenylmethylaziridinone **(2*l*)** yielded α-alkylamino-*N*-benzylamides **(4g**, **i**, **k**, ***l*)** resulting from cleavage of the C_2-N bond

(Scheme 10) and one, 3-(1-adamantyl)-1-triphenylmethylaziridinone **(2n)** gave a mixture of both type of products **(3n** and **4n)**, with the predicted α-benzylaminoamide predominating (Scheme 11).

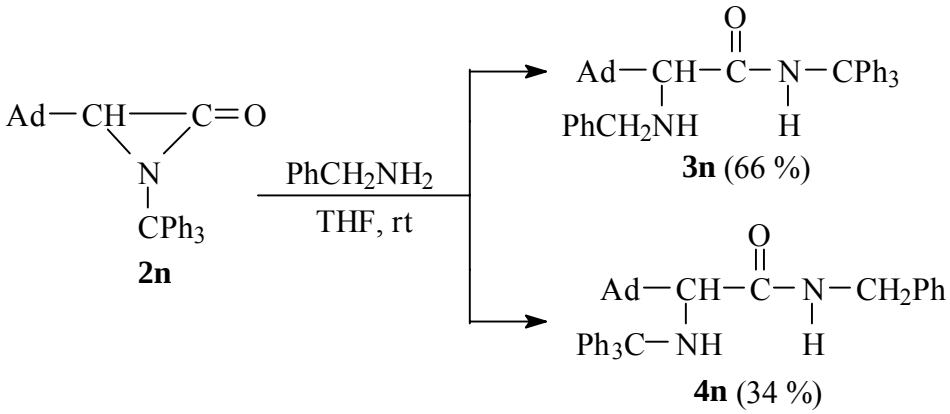
Scheme 10. The reaction of α-lactams **(2g**, **2i**, **2k**, and **2*l*)** with benzylamine.



2g, **2i**, **2k**, **2*l*** **4g**, **4i**, **4k**, **4*l***

α-Lactam	Product	R_1	R_2	R_3
2g	**4g**	*t*-C_4H_9	H	*t*-C_4H_9
2i	**4i**	Ph_3C	H	*t*-C_4H_9
2k	**4k**	*t*-C_4H_9	H	Ad
2*l*	**4*l***	Ph_3C	H	Ad

Scheme 11. The reaction of α-lactam (**2n**) with benzylamine.



The reactions with benzylamine were carried out on pure samples of α-lactams, except **2j**, **2m** and **2o**. The latter three proved difficult to purify and, therefore, were generated in ether solution at 0°C *in situ*, and reacted directly with benzylamine without isolation. These three α-lactams had an extent of purity of about 80 %, as calculated from the ratio of the α-lactam (1840 cm^{-1}) *versus* α-bromo amide (1685 cm^{-1}) carbonyl bands in the IR spectra.

All benzylamine reaction products obtained in this study have been fully characterized by mp, TLC R_f value, IR, ^{1}H-NMR, ^{13}C-NMR, MS, and elemental analysis. The reaction times, isolated yields (of pure products), mps, and IR carbonyl bands are listed in Tables 2 and 3.

Table 2. Reaction times, isolated yields of pure products, mps and IR carbonyl bands of the α-benzylaminoamide type products.

Product	Time (h)	% Yield	mp (°C)	IR (CCl_4; cm^{-1})
3j	2	84.5*	100-102	1678
3m	12	74.3*	156-157	1690
3o	2	52.6*	119-121	1696

*based on α-bromo amide as starting material

Table 3. Reaction times, isolated yields of pure products, mps, and IR carbonyl bands of the *N*-benzylamide type products.

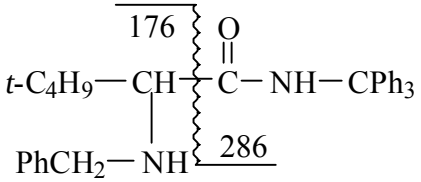
Product	Time (h)	% Yield	mp (°C)	IR (CCl_4; cm^{-1})
4g	120	98	90-91	1674
4i	120	84.6	151-154	1671
4k	72	83.8	132-134	1673
4*l*	22	99	164-165	1669

3. Structure Assignment.

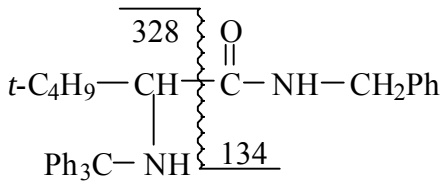
The two types of adducts **(3** and **4)** that can result from the reaction of α-lactams with benzylamine are not only structural isomers, they are also very closely related, both being α-*N*-alkylaminoamides. As a result, they are difficult to distinguish without having both structural isomers at hand. Therefore, it is essential to obtain and study all spectral data, and compare it with those available in the literature, before making a structure assignment.

An unequivocal distinction between the two structural alternatives can be made by a combination of MS, NMR, and to a lesser extent, IR. For example, Figure 1 and Table 4 demonstrate how one can distinguish between two structural isomers on the basis of their MS, NMR, and IR.

Figure 1. Distinguishing between the two structural isomers on the basis of MS.



3f



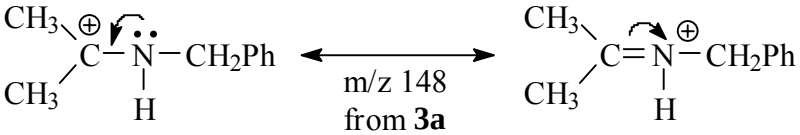
7 (unknown)

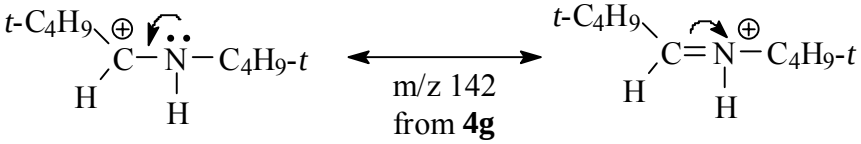
Table 4. Distinguishing between the two structural isomers on the basis of spectral data.

3f (from ref. 9)		**7** (predicted from results of this study)
176	MS of immonium ion	328
3.57, dd, 1H and 3.71, dd, 1H	^{1}H-NMR of benzylic protons	~ 4.15, dd, 1H and 4.35, dd, 1H
53.73	^{13}C-NMR of benzylic carbon	~ 43
1690 cm^{-1}	IR of amide carbonyl	1674 cm^{-1}

The most powerful and reliable corroboration (evidence) for a structure is derivable from the mass spectrum. It is known[7,9,13] that α-*N*-alkylaminoamides fragment upon electron impact into abundant resonance-stabilized immonium type ions which are *unique for each structure*. For example, it was such an ion at m/z 148 which served decisively in assigning the α-benzylamino-*N*-*tert*-butylamide structure **(3a)** to the product of 1-*tert*-butyl-3,3-dimethylaziridinone with benzylamine.[7] An ion of this MS cannot be derived from the alternative structure. The same type of immonium ions are also present in high abundance in the MS spectra of the products of other α-lactams with benzylamine, e.g., for **4g** at m/z 142 (Scheme 12).

Scheme 12. Resonance-stabilized structure-proving fragment ions in the mass spectra of **3a** and **4g**.





NMR spectra also contain valuable information, on the basis of which a distinction between the two isomeric structures can be made. Thus, the benzylic methylene protons of α-benzylaminoamides **(3)** have their signal in the ^{1}H-NMR at δ 3.5-3.9, while that of *N*-benzylamides **(4)** usually appears in the range δ 4.0-4.5. Our ^{1}H-NMR spectral assignment of the benzylic protons of isomers **(3)** and **(4)** is in agreement with D'Angeli *et al*.,[19] who reported that the benzylic protons of 2-benzylamino-*N*-*t*-butylpropanamide (type **3** product) appear at δ 3.72, while those of its isomer, *N*-benzyl-2-*t*-butylaminopropanamide (type **4** product), are at δ 4.43. In the ^{13}C-NMR spectrum, the benzylic carbon signal of α-benzylaminoamides **(3)** is exhibited in the δ 47-54 range, while that of *N*-benzylamides **(4)** appears at approximately δ 43.

Finally, the wavenumber of the amide carbonyl band in the infrared spectrum is also a useful indicator of which product is present. The α-benzylaminoamide **(3)** type products have an IR carbonyl band (in

CCl_4 solution) between 1680 and 1696 cm^{-1}, while the carbonyl band (CCl_4) of N-benzylamides **(4)** appears at 1669-1674 cm^{-1}.

4. Trends in the Structural Parameters Determining the Preferred Product.

Including the present study, there are now sixteen α-lactams, which have been reacted with benzylamine. The relevant spectroscopic data of the products of these reactions are assembled in Tables 5 and 6. Contemplating and comparing the structure of these sixteen α-lactams and reflecting upon the data in Tables 5 and 6, some general trends emerge:

(a) α-lactams of low thermal stability and high chemical reactivity give α-benzylaminoamides **(3)**,

(b) the thermally and chemically more stable α-lactams give *N*-benzylamides **(4)**,

(c) *N*-tritylsubstituted α-lactams give α-benzylaminoamides **(3)**, *irrespective of relative stability or reactivity*,

(d) there is a correlation between the *rate of the reaction at rt and the product*: the fastest reactions lead to α-benzylaminoamides **(3)**.

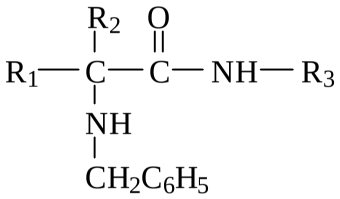
Even though the above correlations have been deduced entirely empirically, they enable us to predict with a high degree of confidence for any α-lactam, reported or unreported, with any substitution pattern, which product it will give with benzylamine. Thus, the stability and substitutents are critical factors in influencing the regioselectivity in nucleophilic ring-opening reactions of α-lactams.

EXPERIMENTAL

Melting points are uncorrected and were measured on a Thomas-Hoover® capillary melting point apparatus. TLC was performed with Analtech® silica gel glass backed plates (250 microns) and recorded as a function of R_f values. Flash chromatographic separations were performed using silica gel (JT Baker®, 40 μm) as the stationary phase. IR spectra were recorded on a Perkin Elmer® Fourier Transform (FT-IR) Spectrum 1000 Spectrophotometer. NMR spectra (^1H and ^{13}C) were obtained on a 400 MHz Bruker Spectrometer with tetramethylsilane as the internal standard. Microanalyses were performed by Atlantic Microlab, Inc. (Norcross, Georgia). MS spectra were recorded on a Hewlett Packard® G1800A GCD System or a Finnigan LCQ quadrupole ion trap spectrometer at Scripps Research Institute (La Jolla, California).

The reagents *tert*-butylamine, tritylamine, benzylamine (freshly distilled before use), sodium methoxide, sodium *tert*-butoxide, thionyl chloride, 1-adamantanamine, α-bromophenylacetic acid, and the solvents, ether and tetrahydrofuran (THF) were obtained from Aldrich® (Milwaukee, WI). Ethyl acetate, methylene chloride, *n*-heptane, benzene and *p*-xylene were obtained from J.T. Baker® (Phillipsburg, NJ). *n*-Hexane was obtained from EM Science (Gibbstown, NJ).

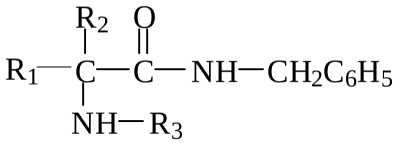
Table 5. Spectroscopic data of the α-benzylaminoamide (3) type adducts.



Product number	R_1	R_2	R_3	IR (CCl_4) (C=O) (cm^{-1})	^{1}H-NMR (benzylic CH_2 protons, δ)	^{13}C-NMR (benzylic CH_2 carbon, δ)	MS (structure proving ion, m/z)	Reference
3a	CH_3	CH_3	*tert*-butyl	1680	3.71, d, 2H	N.R.**	148	7
3f	*tert*-butyl	H	trityl	1690	3.57, dd, 1H 3.71, dd, 1H	53.73	176	9
3j	CH_3	CH_3	1-adamantyl	1678	3.50, s, 2H	47.47	148	*
3m	CH_3	CH_3	trityl	1690	3.62, d, 2H	47.43	148	*
3n	1-adamantyl	H	trityl	1686	3.36, d, 1H 3.53, d, 1H	52.61	254	*
3o	C_6H_5	H	trityl	1696	3.61, dd, 1H 3.68, dd, 1H	51.86	196	*

*: Reported in this paper, **N.R.: not reported.

Table 6. Spectroscopic data of the α-alkylaminobenzylamide (4) type adducts.



Product number	R_1	R_2	R_3	IR (C=O) (cm^{-1})	^{1}H-NMR (benzylic CH_2 protons, δ)	^{13}C-NMR (benzylic CH_2 carbon, δ)	MS (structure proving ion, m/z)	Reference
4b	$(C_6H_5)C(CH_3)_2$	H	*tert*-butyl	1655 (KBr)	4.30, q, 1H 4.45, q, 1H	N.R.**	N.R.**	8
4c	$(C_6H_5)C(CH_3)_2$	H	1-adamantyl	1650 (KBr)	4.32, dd, 1H 4.47, dd, 1H	N.R.**	N.R.**	8
4d	*tert*-butyl	H	$(C_6H_5)C(CH_3)_2$	1640 (KBr)	4.32-4.35, m, 2H	N.R.**	N.R.**	8
4e	$(C_6H_5)C(CH_3)_2$	H	$(C_6H_5)C(CH_3)_2$	1640 (KBr)	4.12, dd, 1H 4.31, dd, 1H	N.R.**	N.R.**	8
4g	*tert*-butyl	H	*tert*-butyl	1674 (CCl_4)	4.25, dd, 1H 4.29, dd, 1H	42.34	142	*
4i	trityl	H	*tert*-butyl	1671 (CCl_4)	3.33, d, 1H 4.12, m, 1H	43.79	328	*
4k	*tert*-butyl	H	1-adamantyl	1673 (CCl_4)	4.22, dd, 1H 4.32, dd, 1H	42.26	220	*
4*l*	trityl	H	1-adamantyl	1669 (CCl_4)	3.32, dd, 1H 4.21, dd, 1H	43.51	406	*
4n	1-adamantyl	H	trityl	1674 (CCl_4)	3.93, dd, 1H 4.00, dd, 1H	43.46	406	*
6	*tert*-butyl	H	1,8-*p*-menthylene	1674 (CCl_4)	4.36-4.56, m 2H	43.53 43.65	442 (357)	13

*: Reported in this paper, **N.R.: not reported.

I. The synthesis of α-lactams used in this study.

The general procedure of Scrimin *et al.*[15] was used to synthesize 1,3-di-*tert*-butylaziridinone[11] **(2g)** and 1-(1-adamantyl)-3-*tert*-butylaziridinone[12] **(2k)**. The synthesis of 1-*tert*-butyl-3-triphenylmethyl-aziridinone **(2i),** 1-(1-adamantyl)-3-triphenylmethylaziridinone **(2*l*),** 1-triphenylmethyl-3,3-dimethyl-aziridinone **(2m),** 3-(1-adamantyl)-1-triphenylmethylaziridinone **(2n)** are reported[18] elsewhere. All physical properties and spectral data for these α-lactams are in agreement with the previously reported literature values. The general procedure of Sheehan and Lengyel[7] was used to synthesize 1-(1-adamantyl)-3,3-dimethylaziridinone[16] **(2j)** and it was used without purification.

***N*-Triphenylmethyl-2-bromo-2-phenylacetamide (1o).**

The general procedure of Lengyel and Aaronson[20] was followed. To a solution of carbon tetrachloride (5 mL) and thionyl chloride (6.19 g, 0.052 mol), 2-bromophenylacetic acid (2.80 g, 0.013 mol) was added. The reaction mixture was heated to 65°C for 30 min. Carbon tetrachloride and thionyl chloride were removed under reduced pressure to yield crude 2-bromo-2-phenylacetyl chloride (3.04 g (100 %), a light yellow residue, which was used without further purification.

To a solution of triphenylmethylamine (3.71 g, 0.014 mol) and triethylamine (1.45 g, 0.014 mol) dissolved in methylene chloride (100 mL), crude 2-bromophenylacetyl chloride (3.04 g, 0.013 mol) in methylene chloride (20 mL) was added dropwise. The reaction mixture stirred for 70 h, and was then washed twice with 100 mL of distilled water, and once with 50 mL of 2N HCl. The solvent was removed under reduced pressure to obtain crude *N*-triphenylmethyl-2-bromo-2-phenylacetamide **(1o)**, a light-yellow solid. The crude product was recrystallized from a mixture of 40 mL of *n*-heptane and 15 mL of ethyl acetate to yield pure **1o**, (3.93 g, 66.3 %), mp 160 -162°C. TLC (80 % *n*-hexane: 20 % ethyl acetate) $R_f = 0.52$. IR (CCl_4): 3390 (N-H); 3058 and 3020 (aromatic C-H), 2927 and 2850 (aliphatic C-H), 1685 (amide C=O) cm^{-1}. ^1H NMR ($CDCl_3$): $\delta = 5.41$ (s, proton on the brominated carbon adjacent to the amide carbonyl, 1H); 7.31 (m, aromatic protons, 20H), 7.83 (br s, N-H proton, 1H). ^{13}C-NMR ($CDCl_3$): $\delta = 52.53$ (brominated carbon adjacent to amide carbonyl), 71.00 (tertiary carbon of the trityl moiety), 127.40 (carbons in *para* position of trityl moiety), 128.25 (carbons in *meta* position of trityl moiety), 128.54 (carbon in *para* position on phenyl group adjacent to the brominated carbon), 128.66 (carbons in *ortho* position of trityl moiety), 129.08 (carbons in *meta* position on phenyl group adjacent to the brominated carbon), 129.20 (carbons in *ortho* position on phenyl group adjacent to the brominated carbon), 137.24 (C1 carbon on phenyl group adjacent to the brominated carbon), 144.19 (C1 carbons of the trityl moiety), 165.90 (amide carbonyl). Anal. Calcd for $C_{27}H_{22}NOBr$: C 71.06, H 4.86, N 3.07, Br 17.51. Found: C 71.20, H 4.92, N 3.11, Br 17.50.

3-Phenyl-1-triphenylmethylaziridinone (2o).

The general synthesis procedure of Sheehan and Lengyel[7] was followed. To a suspension of *N*-

triphenylmethyl-2-bromo-2-phenylacetamide **(1o)** (1.00 g, 2.2 mmol) in ether (80 mL), sodium *tert*-butoxide (0.23 g, 2.42 mmol) in ether (10 mL) was added at 0°C. The reaction mixture stirred for 30 min and was then centrifuged at 2000 rpm for 3 min. The supernatant was decanted, and the workup procedure of Simig *et al*.[17] was followed. *n*-Hexane (80 mL) was added to the supernatant. The ether was removed under reduced pressure and the remaining *n*-hexane solution was chilled to -70°C using a dry ice/acetone bath. The mixture was filtered to obtain a white solid of nearly pure **2o** (0.52 g, 63%), which slowly decomposes at rt. IR (CCl_4): 3064 and 3034 (aromatic C-H); 2927 and 2956 (aliphatic C-H); 1843 (lactam C=O) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 3.47 (s, methine proton at C3 of aziridinone ring, 1H), 7.22-7.33 (m, aromatic protons, 20H). ^{13}C-NMR ($CDCl_3$): δ = 48.37 (C3 carbon of the aziridinone ring), 78.69 (tertiary carbon of trityl moiety), 127.20 (carbon in *para* position of the phenyl ring), 127.81 (carbons in *para* position of the trityl moiety), 127.95 (carbons in *meta* position of the phenyl ring), 128.18 (carbons in *meta* position of the trityl moiety), 128.63 (carbons in *ortho* position of the phenyl ring), 128.80 (carbons in *ortho* position of the trityl moiety), 134.81 (C1 carbon of the phenyl ring), 142.19 (C1 carbon of the trityl moiety), 154.83 (lactam carbonyl).

II. Reaction of the α-lactams with benzylamine.

The general procedure of Shimazu *et al*.[8] was followed for the reaction of α-lactams **(2g**, **i-*l***, **n**, **o)** with benzylamine.

Reaction of 1,3-di-*tert*-butylaziridinone (2g) with benzylamine.

1,3-Di-*tert*-butylaziridinone **(2g)** (0.20 g, 1.18 mmol) was dissolved in THF (6 mL) and benzylamine (0.506 g, 4.72 mmol) was added. The reaction mixture stirred at rt for 5 days. Excess solvent was removed under reduced pressure to afford a solid of crude *N*-benzyl-2-*tert*-butylamino-3,3-dimethylbutanamide **(4g)**. Flash chromatography using 80 % *n*-hexane: 20 % ethyl acetate as the eluent yielded 0.32 g (98.0 %) of pure **4g**, mp 90-91°C. TLC (80 % *n*-hexane: 20 % ethyl acetate): R_f = 0.34. IR (CCl_4): 3450 (amide N-H); 3367 (amine N-H); 3087, 3067 and 3031 (aromatic C-H); 2963 and 2870 (aliphatic C-H); 1674 (amide C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6): δ = 0.85, (s, methyl protons of *tert*-butyl moiety, 9H), 0.95 (s, methyl protons of *tert*-butylamino moiety, 9H), 1.70 (br s, N-H proton of *tert*-butylamino moiety, exchanges for deuterium in DMSO-d_6/D_2O, 1H), 2.82 (s, methine proton adjacent to carbonyl carbon, 1H), 4.25 (dd, J = 14.77, 5.72 Hz, benzylic proton of the benzylamide group, 1H), 4.29 (dd, J = 14.77, 5.96 Hz, the other benzylic proton of the benzylamide group, 1H), 7.21-7.50 (m, aromatic protons, 5H), 8.33 (t, J = 5.72 Hz, N-H proton of amide, exchanges for deuterium when heated in DMSO-d_6/D_2O, 1H). ^{13}C-NMR (DMSO-d_6): δ = 26.81 (methyl carbons of *tert*-butyl moiety), 29.42 (methyl carbons of *tert*-butylamino moiety), 33.92 (quaternary carbon of

tert-butyl moiety), 42.34 (benzylic carbon of N-benzylamide), 50.07 (quaternary carbon of *tert*-butylamino moiety), 62.94 (methine carbon adjacent to carbonyl carbon), 126.79 (carbon in *para* position of phenyl ring), 127.73 (carbons in *meta* position of phenyl ring), 128.19 (carbons in *ortho* position of phenyl ring), 139.44 (C1 carbon of phenyl ring), 175.05 (amide carbonyl). GC/MS: m/z 276 (M^+, $C_{17}H_{28}N_2O$); 261 $(M - CH_3)^+$; 219 $(M - C_4H_9)^+$; 142 $((CH_3)_3C\text{-}CH\text{-}NH\text{-}C(CH_3)_3 \leftrightarrow (CH_3)_3C\text{-}CH{=}NH\text{-}C(CH_3)_3)^+$; 86 (*base peak*, $(CH_3)_3C\text{-}CH{=}NH_2^+$). Anal. Calcd for $C_{17}H_{28}N_2O$: C 73.83, H 10.21, N 10.13. Found: C 73.89, H 10.25, N 10.15

Reaction of 1-*tert*-butyl-3-triphenylmethylaziridinone (2i) with benzylamine.

α-Lactam **(2i)** (0.10 g, 0.28 mmol) was dissolved in THF (3 mL) and a solution of benzylamine (0.12 g, 1.13 mmol, 4 equivalents) in THF (1 mL) was added. The reaction mixture stirred at rt for 5 days. Excess solvent and benzylamine were removed under reduced pressure to afford 0.13 g of crude *N*-benzyl-2-*tert*-butylamino-3,3,3-triphenylpropanamide **(4i)**. Flash chromatography, using 80 % *n*-hexane: 10 % ethyl acetate as eluent, afforded 0.11 g (84.6 %) of pure **4i**, mp 151–154°C. TLC (80 % *n*-hexane: 20 % ethyl acetate) $R_f = 0.48$. IR (CCl_4): 3389 (amide N-H); 3335 (amine N-H); 3062 and 3034 (aromatic C-H); 2969 and 2870 (aliphatic C-H); 1671 (amide carbonyl) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 0.97 (s, *tert*-butyl protons, 9H), 1.59 (s, proton on nitrogen of the *tert*-butylamino moiety, exchanges for deuterium in $CDCl_3/D_2O$, 1H), 3.33 (d, J = 14.34 Hz, benzylic proton of the benzylamide group, 1H), 4.12 (m, the other benzylic proton of the benzylamide moiety, 1H), 5.06 (s, proton on the carbon adjacent to the carbonyl carbon, 1H), 6.48 (br s, proton attached to the amide nitrogen, exchanges for deuterium in $CDCl_3/CF_3COOD$, 1H), 6.91–7.50 (m, aromatic protons of the trityl moiety and the phenyl ring of the benzylamide group, 20H). ^{13}C-NMR ($CDCl_3$): δ = 29.83 (methyl carbons of the *tert*-butyl group), 43.79 (benzylic methylene carbon), 52.30 (quaternary carbon of the *tert*-butyl group), 62.01(methine carbon adjacent to the carbonyl carbon), 63.48 (quaternary carbon of the trityl moiety), 126.51 (carbon in *para* position of the benzylic phenyl ring), 127.35 (phenyl carbons in *para* position of the trityl moiety), 127.65 (carbons in *meta* position of the benzylic phenyl ring), 128.52 (phenyl carbons in *meta* position of the trityl moiety), 128.55 (carbons in *ortho* position of the benzylic phenyl ring), 130.94 (phenyl carbons in *ortho* position of the trityl moiety), 137.87 (C1 carbon in the benzylic phenyl ring), 144.43 (three C1 carbons in the trityl moiety) and 173.63 (carbonyl carbon of the amide). MS: m/z 463, $(M + H)^+$, $C_{32}H_{35}N_2O$; 407, $(M - 55)^+$; 328, $[(C_6H_5)_3C\text{-}CH{=}NH\text{-}C(CH_3)_3]^+$; 272, $(328 - C_4H_8)^+$; 257, $(272 - NH)^+$; 243, $[(C_6H_5)_3C]^+$, *base peak*. Anal. Calcd for $C_{32}H_{34}N_2O$: C 83.08; H 7.41; N 6.06. Found: C 82.79; H 7.50; N 6.02.

Reaction of 1-(1-adamantyl)-3,3-dimethylaziridinone (2j) with benzylamine.

Crude 1-(1-adamantyl)-3,3-dimethylaziridinone **(2j)** (0.34 g, 1.55 mmol) (estimated to be 80% pure by IR) was dissolved in THF (10 mL) and benzylamine (0.527 g, 4.92 mmol, 4 equivalents) was added.

The reaction mixture stirred at rt for 2 h. Excess solvent was removed under reduced pressure to afford crude *N*-(1-adamantyl)-2-benzylamino-2-methylpropanamide **(3j)**. Flash chromatography using 70 % *n*-hexane: 30 % ethyl acetate as the eluent afforded 0.24 g of *N*-(1-adamantyl) 2-bromo-2-methylpropanamide **(1j)**, mp 105-107°C which is the precursor to 1-(1-adamantyl)-3,3-dimethylaziridinone **(2j)**, and 0.34 g (84.5 %) of pure *N*-(1-adamantyl)-2-benzylamino-2-methylpropanamide **(3j),** mp 100-102°C. TLC (70 % *n*-hexane: 30 % ethyl acetate): $R_f = 0.40$. IR (CCl_4): 3356 (N-H); 3080 and 3030 (aromatic C-H); 2909 and 2851 (aliphatic C-H); 1678 (amide C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6): $\delta = 1.19$ (s, methyl protons, 6H), 1.61 (s, methylene protons of adamantane moiety farther away from N, 6H), 1.90 (s, methylene protons of adamantane moiety closer to N, 6H), 2.00 (s, methine protons of adamantane moiety, 3H), 2.60 (br s, N-H proton of benzylamino moiety, exchanges for deuterium in DMSO-d_6/D_2O, 1H), 3.50 (br s, benzylic protons, 2H), 7.21-7.39 (m, aromatic protons of benzylamino moiety, 5H and N-H proton of the amide, exchanges for deuterium in hot DMSO-d_6/D_2O, 1H). ^{13}C-NMR (DMSO-d_6): $\delta = 25.22$, (methyl carbons), 28.80 (methine carbons of the adamantane moiety), 36.00 (methylene carbons of the adamantane moiety farther away from N), 41.00 (methylene carbons of the adamantane moiety closer to N), 47.47 (benzylic carbon), 49.91 (C1 carbon of the adamantane moiety), 58.81 (carbon adjacent to carbonyl carbon of amide), 126.61 (carbon in *para* position of phenyl ring), 127.92 (carbons in *meta* position of phenyl ring), 128.20 (carbons in *ortho* position of phenyl ring), 141.04 (C1 carbon of phenyl ring), 175.07 (amide carbonyl). MS: m/z 326 (M^+, $C_{21}H_{30}N_2O$); 311 $(M - CH_3)^+$; 148 (*base peak*, $(CH_3)_2C{=}NH\text{-}CH_2\text{-}C_6H_5)^+$; 135 $(C_{10}H_{15})^+$; 91 $(C_7H_7)^+$. Anal. Calcd for $C_{21}H_{30}N_2O$: C 77.26, H 9.26, N 8.58. Found: C 76.96, H 9.36, N 8.40.

Reaction of 1-(1-adamantyl)-3-*tert*-butylaziridinone (2k) with benzylamine.

1-(1-Adamantyl)-3-*tert*-butylaziridinone **(2k)** (0.25 g, 1.01 mmol) was dissolved in THF (6 mL) and benzylamine (0.433 g, 4.04 mmol, 4 equivalents) was added. The reaction mixture stirred at rt for 72 h. The solvent was removed under reduced pressure to afford a solid, crude *N*-benzyl-2-(1-adamantylamino)-3,3-dimethylbutanamide **(4k)**. Flash chromatography, using 80 % *n*-hexane: 20 % ethyl acetate as the eluent, afforded 0.30 g (83.8 %) of pure **4k**, mp 132-134°C. TLC (70 % *n*-hexane: 30 % ethyl acetate): $R_f = 0.40$. IR (CCl_4): 3446 (amide N-H); 3358 (amine N-H); 3031 (aromatic C-H); 2908 and 2851 (aliphatic C-H); 1673 (amide C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6): $\delta = 0.84$ (s, methyl protons, 9H), 1.42-1.46 (m, methylene protons of the adamantane moiety 12H, and N-H proton of 1-adamantylamino moiety, exchanges for deuterium in DMSO-d_6/D_2O, 1H), 1.87-2.09 (m, methine protons of the adamantane moiety, 3H), 2.90 (s, methine proton adjacent to amide carbonyl, 1H), 4.22 (dd, J = 14.77, 5.63 Hz, benzylic proton on the benzylamide group, 1H), 4.32 (dd, J = 14.77, 6.11 Hz, the other benzylic proton, 1H), 7.14-7.48 (m, aromatic protons, 5H), 8.31(t, J = 6.11, 5.63 Hz, N-H

proton of amide, exchanges for deuterium in warm DMSO-d_6/D_2O, 1H). ^{13}C-NMR (DMSO-d_6): δ = 26.85 (methyl carbons of *tert*-butyl moiety), 28.99 (methine carbons of the adamantane moiety), 33.80 (quaternary carbon of *tert*-butyl moiety), 36.25 (methylene carbons of the adamantane moiety farther away from N), 42.26 (benzylic carbon of benzylamino moiety), 42.83 (methylene carbons of the adamantane moiety closer to N), 49.88 (C1 carbon of the adamantane moiety), 60.97 (methine carbon adjacent to carbonyl carbon of amide), 126.76 (carbon in *para* position of the phenyl ring), 127.72 (carbons in *meta* position of the phenyl ring), 128.14 (carbons in *ortho* position of the phenyl ring), 139.50 (C1 carbon of the phenyl ring), 174.99 (amide carbonyl). MS: m/z 354 (M^+, $C_{23}H_{34}N_2O$); 297 $(M - C_4H_9)^+$; 220 (*base peak*, $(CH_3)_3$-CH=NH-$C_{10}H_{15})^+$; 135 $(C_{10}H_{15})^+$; 107 $(C_6H_5$-CH_2-$NH_2)^+$; 91 $(C_7H_7)^+$. Anal. Calcd for $C_{23}H_{34}N_2O$: C 77.92, H 9.67, N 7.90. Found: C 77.94, H 9.78, N 7.85

Reaction of 1-(1-adamantyl)-3-triphenylmethylaziridinone (2*l*) with benzylamine.

1-(1-Adamantyl)-3-triphenylmethylaziridinone **(2*l*)** (0.10 g, 0.23 mmol) was dissolved in THF (3 mL) and benzylamine (0.099 g, 0.924 mmol, 4 equivalents) was added. The reaction mixture stirred at rt for 22 h. Excess solvent and benzylamine were removed under reduced pressure to afford a near quantitative yield of pure *N*-benzyl-2-(1-adamantylamino)-3,3,3-triphenylpropanamide **(4*l*)**, mp 164–165°C. TLC (80 % *n*-hexane: 20 % ethyl acetate): R_f = 0.56. IR (CCl_4): 3386 (amide N-H); 3327 (amine N-H); 3062 and 3033 (aromatic C-H); 2908 and 2850 (aliphatic C-H); 1669 (amide C=O) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 1.29 (br s, N-H of 1-adamantylamino moiety, exchanges for deuterium in D_2O, 1H), 1.47 (t, J = 11.64 Hz, methylene protons of the adamantane moiety farther away from N, 6H), 1.58 (d, J = 11.98 Hz, methylene protons of the adamantane moiety closer to N, 6H), 1.95 (s, methine protons of the adamantane moiety, 3H), 3.32 (dd, J = 14.29, 3.82 Hz, benzylic proton of the benzylamido moiety, 1H), 4.21 (dd, J = 14.29, 7.15 Hz, the other benzylic proton of the benzylamido moiety, 1H), 5.26 (s, methine proton adjacent to the carbonyl carbon, 1H), 6.57 (t, J = 5.28 Hz, N-H proton of amide, exchanges for deuterium with TFD, 1H), 6.93–7.43 (m, aromatic protons of the trityl and benzyl moiety, 20H). ^{13}C-NMR (DMSO-d_6): δ = 29.96 (methine carbons of the adamantane moiety), 35.95 and 43.70 (methylene carbons of the adamantane moiety), 43.51 (benzylic methylene carbon), 52.01 (C1 carbon of the adamantane moiety), 60.77 (methine carbon adjacent to the carbonyl carbon), 62.63 (quaternary carbon of the trityl moiety), 126.72 (carbon in *para* position of the benzylamido moiety), 127.43 (carbons in *para* position of the trityl moiety), 127.68 (carbons in *meta* position of the benzylamido moiety), 128.49 (carbons in *meta* position of the trityl moiety), 128.81 (carbons in *ortho* position of the benzylamido moiety), 131.57 (carbons in *ortho* position of the trityl moiety), 139.25 (C1 carbon of the benzylamido moiety), 146.18 (C1 carbons of the trityl moiety), 174.46 (amide C=O). MS: m/z 541.2 (MH^+, $(C_{38}H_{41}N_2O)^+$); 406.2, (*base peak*, M – $C_6H_5CH_2$-NHC=O)$^+$. Anal. Calcd for $C_{38}H_{40}N_2O$: C 84.40, H 7.46, N 5.18. Found: C 84.19, H 7.45, N 5.20

Reaction of 3,3-dimethyl-1-triphenylmethylaziridinone (2m) with benzylamine.

3,3-Dimethyl-1-triphenylmethylaziridinone **(2m)** was generated *in situ* and reacted with benzylamine by the following procedure: to a solution of *N*-trityl-2-bromo-2-methylpropanamide **(1m)** (1.02 g, 0.0025 mol) in 50 mL of anhydrous ether cooled to 0°C, sodium *tert*-butoxide (0.264 g, 0.00275 mol) was added with stirring. After 30 min, freshly distilled benzylamine (0.80 g, 0.0075 mol, 3 equivalents) was added in one portion, and the reaction mixture stirred at 0°C for 90 min. It then stirred overnight at rt. The ether and excess benzylamine were removed under reduced pressure to yield a crude solid residue. The residue was taken up in 50 mL of ethyl acetate and washed with distilled water (3 x 20 mL). The organic layer was dried with Na_2SO_4 and solvent evaporated under reduced pressure to afford crude *N*-trityl-2-benzylamino-2-methylpropanamide **(3m)**, which was flash chromatographed using 85 % *n*-hexane: 15 % ethyl acetate as eluent, to give 0.81 g (74.3%) of pure **3m**, mp 156-157°C. TLC (80% *n*-hexane: 20% ethyl acetate) $R_f = 0.30$. IR (CCl_4) 3335 (amine N-H); 3092, 3062, 3025 (aromatic C-H); 2981, 2929 (aliphatic C-H); 1690 (amide C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6): δ = 1.25 (s, methyl protons, 6H), 2.95 (t, J = 8.04, 7.91 Hz, amine NH-proton, exchanges for deuterium in D_2O, 1H), 3.62 (d, J = 7.75 Hz, benzylic methylene protons, 2H), 7.05-7.30 (m, phenyl protons, 20H), 9.09 (s, amide NH-proton, exchanges for deuterium in D_2O, 1H). ^{13}C-NMR (DMSO): δ = 24.78 (methyl carbons), 47.43 (methylene carbon), 59.02 (tertiary carbon attached to carbonyl carbon), 68.50 (tertiary carbon of the trityl moiety), 126.51 (*para* carbons of benzyl moiety), 126.57 *(para* carbons of trityl moiety), 127.62 (*meta* carbons of all the phenyl moieties), 127.98 (*ortho* carbons of benzyl moiety), 128.13 (*ortho* carbons of trityl moiety), 140.51 (C1 of the benzyl moiety), 144.74 (C_1 of the trityl moiety), and 174.80 (carbonyl carbon). MS: chemical ionization m/z 435 (MH^+, $(C_{30}H_{31}N_2O)^+$); 243, ($(C_6H_5)_3C^+$), 165 (*base peak*, $C_{13}H_9^+$); electron impact m/z 419 $(M\text{-}CH_3)^+$, 148 *base peak* $[(CH_3)_2C{=}NHCH_2C_6H_5]^+$. Anal. Calcd for $C_{30}H_{30}N_2O$: C 82.91; H 6.96; N 6.45. Found: C 82.76; H 7.01; N 6.46.

Reaction of 3-(1-adamantyl)-1-triphenylmethylaziridinone (2n) with benzylamine.

3-(1-adamantyl)-1-triphenylmethylaziridinone **(2n)** (0.20 g, 0.46 mmol) was dissolved in THF (6 mL) and benzylamine (0.193 g, 1.80 mmol, 4 equivalents) was added. The reaction mixture stirred at rt for 48 h. Excess solvent and benzylamine were removed under reduced pressure to yield a crude mixture of *N*-triphenylmethyl-2-benzylamino-2-(1-adamantyl)acetamide **(3n)** and *N*-benzyl-2-(1-adamantyl)-2-triphenylmethylaminoacetamide **(4n)**. The mixture was flash chromatographed using 90 % *n*-hexane:10 % ethyl acetate as the eluent to afford 0.18 g (combined yield of 72%) of **3n** and **4n**, which was recrystallized from a mixture of 10 mL of *n*-heptane and 3 mL of ethyl acetate to give a mixture of *N*-triphenylmethyl-2-benzylamino-2-(1-adamantyl)acetamide **(3n)** and *N*-benzyl-2-(1-adamantyl)-2-triphenylmethylaminoacetamide **(4n)** (0.12 g, 50 %). The ratio of **3n** to **4n** was 66 % to 34 % based

on ^{1}H-NMR data. TLC (90 % *n*-hexane: 10 % ethyl acetate): R_f = 0.68. IR (CCl_4): 3448 (amide N-H); 3340 (amine N-H); 3063 and 3031 (aromatic C-H); 2906 and 2850 (aliphatic C-H); 1686 (amide C=O) of **3n**; 1674 (amide C=O) of **4n**, cm^{-1}. ^{1}H-NMR (DMSO-d_6): **3n**: δ = 3.36 (d, J = 13.88 Hz, benzylic protons of the benzylamino group, 1H), 3.53 (d, J = 13.84 Hz, benzylic proton of the benzylamino group, 1H). **4n**: δ = 3.93 (dd, J = 14.90, 6.14 Hz, benzylic proton of the benzylamido group, 1H), 4.00 (dd, J = 14.24, 7.11 Hz, benzylic proton of the benzylamido group, 1H). ^{13}C-NMR (DMSO-d_6): **3n**: δ = 52.61 (benzylic methylene carbon). **4n**: δ = 43.46 (benzylic methylene carbon). MS: **3n**: m/z 254 $(C_{10}H_{15}CH{=}NHCH_2C_6H_5)^+$; 243, (*base peak*, $(C_6H_5)_3C)^+$; 165 $(C_{13}H_9)^+$; 135 $(C_{10}H_{15})^+$; 91 $(C_6H_5CH_2)^+$. MS: **4n**: m/z 406 $(C_{10}H_{15}CH{=}NHC(C_6H_5)_3)^+$; 243, (*base peak*, $(C_6H_5)_3C)^+$; 165 $(C_{13}H_9)^+$; 135 $(C_{10}H_{15})^+$; 91 $(C_6H_5CH_2)^+$. Anal. Calcd for $C_{38}H_{40}N_2O$: C 84.40, H 7.46, N 5.18. Found: C 84.14, H 7.54, N 5.13

Reaction of 1-triphenylmethyl-3-phenylaziridinone (2o) with benzylamine.

Crude 1-triphenylmethyl-3-phenylaziridinone **(2o)** (0.15 g, 0.399 mmol) was dissolved in THF (6 mL) and a solution of benzylamine (0.171 g, 1.60 mmol, 4 equivalents) in THF (2 mL) was added. The reaction mixture stirred at rt for 2 h. Excess solvent was removed under reduced pressure to afford a white solid, which was flash chromatographed using 80 % *n*-hexane: 20 % ethyl acetate as the eluent, to afford 0.05 g of *N*-triphenylmethyl-2-bromophenylacetamide **(1o)**, the precursor to 1-triphenylmethyl-3-phenylaziridinone **(2o)** and 0.10 g (52.6 %) of pure *N*-triphenylmethyl-2-benzylamino-2-phenylacetamide **(3o)**, mp 119-121°C. TLC (80 % *n*-hexane: 20 % ethyl acetate): R_f = 0.40. IR (CCl_4): 3419 (amide N-H); 3330 (amine N-H); 3088, 3064 and 3031 (aromatic C-H); 2927, 2957 and 2858 (aliphatic C-H); 1696 (amide C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6): δ = 3.16 (br m, N-H of the benzylamino group, exchanges for deuterium in DMSO-d_6 /D_2O, 1H), 3.61 (dd, J = 13.75, 5.67 Hz, benzylic proton of the benzylamino group, 1H), 3.68 (dd, J = 13.75, 5.69 Hz, benzylic proton on the benzylamino group, 1H,), 4.32 (d, J = 7.04 Hz, methine proton adjacent to amide carbonyl, 1H), 7.07-7.37 (m, aromatic protons, 25H), 9.04 (br s, N-H of amide, exchanges for deuterium in DMSO-d_6/D_2O, 1H). ^{13}C-NMR (DMSO-d_6): δ = 50.97 (benzylic methylene carbon), 65.40 (methine carbon adjacent to carbonyl carbon), 69.06 (tertiary carbon of trityl moiety adjacent to nitrogen), 126.57 (carbon in *para* position of the benzylamino moiety), 126.73 (carbon in *para* position of the phenyl ring), 127.26 (carbons in *para* position of the trityl moiety), 127.35 (carbons in *meta* position of the benzylamino moiety), 127.57 (carbons in *meta* position of the phenyl ring), 127.93 (carbons in *meta* position of the trityl moiety), 128.13 (carbons in *ortho* position of the benzylamino moiety), 128.16 (carbons in *ortho* position of the phenyl ring), 128.27 (carbons in *ortho* position of the trityl moiety), 139.74 (C1 carbon in the benzylamino moiety), 140.05 (C1 carbon of the phenyl ring), 144.62 (C1 carbons of the trityl moiety), 171.05 (amide carbonyl). MS: m/z 243, $(C_6H_5)_3C)^+$; 196 (*base peak*,

($C_6H_5CH=NHCH_2C_6H_5)^+$; 165 $(C_{13}H_9)^+$; 91 $(C_6H_5CH_2)^+$. Anal. Calcd for $C_{34}H_{30}N_2O$: C 84.62, H 6.27, N 5.80. Found: C 84.35, H 6.54, N 5.85.

REFERENCES

1. I. Lengyel and J.C. Sheehan, *Angew. Chem.*, 1968, **80**, 27; see also *Angew. Chem., Int. Ed. Engl.*, 1968, ***7***, 25.
2. J. Backes, Houben-Weyl, *Methoden der Organischen Chemie*, Vierte Auflage, Band E16b, Georg Thieme Verlag, Stuttgart, New York, 1991, pp. 1-30.
3. E. R. Talaty, A. E. Dupuy, Jr., and C. M. Utermohlen, *Chem. Commun.*, 1971, 16.
4. M. M. Yusoff and E. R. Talaty, *Tetrahedron Lett.*, 1996, **48**, 8695.
5. D. J. Tantillo, K. N. Houk, R. V. Hoffman, and J. Tao, *J. Org. Chem.*, 1999, **64**, 3830.
6. R. V. Hoffman, The Amide Linkage: Selected Structural Aspects in Chemistry, Biochemistry, and Material Science, ed. by A. Greenberg, C. M. Breneman, and J. F. Liebman, John Wiley & Sons, Inc., New York, 2000, pp. 137-155.
7. J. C. Sheehan and I. Lengyel, *J. Amer. Chem. Soc*., 1964, **86**, 1356.
8. M. Shimazu, Y. Endo, and K. Shudo, *Heterocycles*, 1997, **45**, 735.
9. I. Lengyel, V. Cesare, H. Karram, and T. Taldone, *J. Heterocycl. Chem.*, 2001, **38**, 997.
10. E. R. Talaty and M. M. Yusoff, *Chem. Commun.*, 1998, 985.
11. J. C. Sheehan and J. H. Beeson, *J. Amer. Chem. Soc.*, 1967, **89**, 362.
12. K. Bott, *Angew. Chem., Int. Ed. Engl*., 1967, **6**, 946.
13. I. Lengyel, V. Cesare, T. Taldone, and D. Uliss, *Synth. Commun.*, 2001, **31**, 3671.
14. I. Lengyel and D. B. Uliss, *Chem. Commun.*, 1968, 1621.
15. P. Scrimin, F. D'Angeli, and A. C. Veronese, *Synthesis*, 1982, 586.
16. H. Stetter, P. Mayska, and U. Wießner, Forschungsbericht des Landes Nordrhein-Westfalen Nr. 3095, Westdeutscher Verlag, Opladen, Germany, 1982.
17. Gy. Simig, K. Lempert, J. Tamas, and G. Czira, *Tetrahedron*, 1975, **31**, 1195.
18. I. Lengyel, V. Cesare, I. Adam, and T. Taldone, *Heterocycles*, 2002, **57**, 73.
19. F. D'Angeli, P. Marchetti, G. Cavicchioni, V. Bertolasi, and F. Maran, *Tetrahedron Assymm.*, 1991, **2**, 1111.
20. I. Lengyel and M. J. Aaronson, *Can. J. Spectroscopy*, 1974, **19**, 95.