Mendeleev Commun., 2003, 13(2), 74–76

Reactions of *N*,*N*-bis(silyloxy)enamines with sulfur-centered and sulfur-stabilised electrophiles[†]

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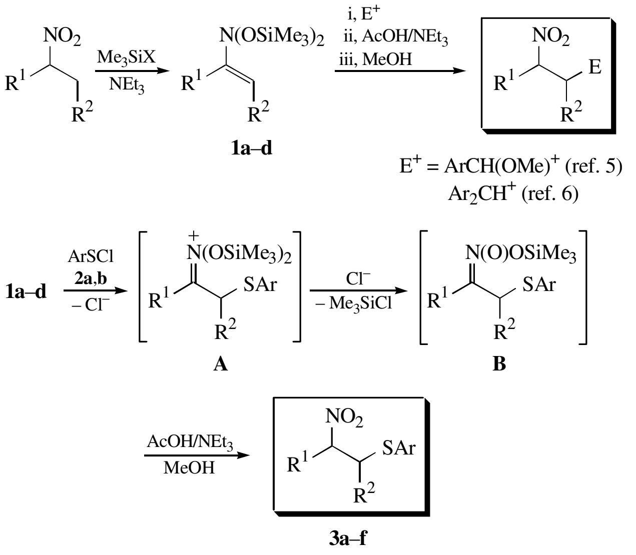
10.1070/MC2003v013n02ABEH001720

The coupling of *N*,*N*-bis(silyloxy)enamines with arylsulfenyl chlorides and episulfonium cations proceeds as an electrophilic attack at the β-carbon atoms of *N*,*N*-bis(silyloxy)enamines and affords functionalised aliphatic compounds.

Doubly silylated derivatives of aliphatic nitro compounds – *N*,*N*-bis(silyloxy)enamines (BENA) **1** – represent a hybrid of conventional enamines with dialkoxyamines.[2] As a consequence, these species are endowed with reactivity toward coupling with both nucleophiles[3,4] and electrophiles.[5]

Recently, we demonstrated that BENA are much less nucleophilic than *N*,*N*-dialkylenamines, and are similar in reactivity to silyl enol ethers.[6] Thus, the interaction of BENA with acetals[5] and benzhydrilium derivatives[6] affords nitro compounds, thereby resulting in the functionalization of the β-carbon atom of starting nitro substrates (Scheme 1). Here, we report on an extension of this methodology by employing sulfur-centered and sulfur-stabilised electrophiles.



BENA	R^1	R^2	ArSCl	Ar	Product	Yield (%)
1a	Me	H	**2a**	*p*-Tol	**3a**	82
1b	H	H	**2a**	*p*-Tol	**3b**	25
1a	Me	H	**2b**	*p*-ClC_6H_4	**3c**	70
1b	H	H	**2b**	*p*-ClC_6H_4	**3d**	77
1c	H	Me	**2b**	*p*-ClC_6H_4	**3e**	33
1d	$(CH_2)_2CO_2Me$	H	**2b**	*p*-ClC_6H_4	**3f**	82

Scheme 1

Arenesulfenyl chlorides rapidly react with BENA at –78 °C leading to β-arenethio-substituted nitro compounds (Scheme 1).[‡] For reproducibility, it is important to use freshly distilled sulfenyl chlorides and to add 2 mol% Pr^i_2NEt, which scavenges acidic impurities into reaction mixture. The reaction proceeds as an electrophilic attack of sulfenyl chloride on the double bond followed by the formation of silylnitronate **B** and Me_3SiCl.

The structures of the obtained products were confirmed by ^1H, ^{13}C and ^{14}N NMR spectroscopy,[§] and the elemental composition was confirmed by microanalysis.

Note that BENA, possessing hydrogen at the α-position (R^1 = H), may be successfully involved in this reaction. Indeed, previously we could not isolate any products from interaction of α-unsubstituted BENA with strong carbocationic electrophiles paired with non-nucleophilic counterion presumably owing to the extreme reactivity of the unsubstituted iminium ion. In the present system, the chloride ion suppresses possible side reactions by the rapid desilylation of the initially formed iminium ion **A**.

As sulfur stabilised cations, we tested episulfonium ions, which can be easily generated from alkyl enol ethers, arenesulfenyl chloride, and Me_3SiOTf (Scheme 2). The reaction of cations **C** with BENA occurs at –78 °C.[¶] Apparently, the equilibrium mixture of species **A′** and **B′** was first formed. The subsequent addition of the external chloride ion ($BnNEt_3^+$ Cl^-) shifts the equilibrium to the right. Finally, the hydrolysis of silylnitronate with an NEt_3/AcOH mixture affords nitro compounds **4** as a 1:1 mixture of diastereomers.[††] The reaction is applicable only to BENA containing a substituent at the α-position ($R^1 \neq$ H). Attempts to involve α-unsubstituted enol

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[†] Chemistry of *N*,*N*-bis(silyloxy)enamines. Part 6, previous see ref. 1.

[‡] All reactions were carried out in an atmosphere of dry argon using dry solvents. Starting BENA were obtained by a previously described procedure.[7]

General procedure for the preparation of **3a–f**. To ArSCl (0.6 mmol) in toluene (1.5 ml) and CH_2Cl_2 (0.5 ml) neat Pr^i_2NEt (0.03 mmol, 5.2 μl) was added at –78 °C; then, a solution of BENA **1** (0.6 mmol) in toluene (1 ml) was added dropwise. After stirring for 30 min at –78 °C, the solutions of Et_3N (0.72 mmol) in toluene (0.5 ml) and AcOH (1.3 mmol) in MeOH (0.5 ml) were added successively. The resulting mixture was warmed to room temperature and stirred for 30 min; then, it was poured into a two-phase mixture of 20 ml Et_2O and 20 ml of H_2O; the aqueous layer was extracted three times with Et_2O; the organic phase was washed with brine, dried over Na_2SO_4 and concentrated *in vacuo*. Purification of the residue using flash chromatography (SiO_2; light petroleum–EtOAc, 10:1) gave *1-(4-tolyl)thio-2-nitropropane* **3a**: R_f 0.16 (light petroleum–EtOAc, 10:1), oil. Found (%): C, 56.78; H, 6.11; N, 6.48; S 14.99. Calc. for $C_{10}H_{13}NO_2S$ (%): C, 56.85; H, 6.20; N, 6.63; S 15.18.

1-(4-Tolyl)thio-2-nitroethane **3b**: R_f 0.45 (light petroleum–EtOAc, 3:1), oil. Found (%): C, 54.77; H, 5.60; N, 7.04; S, 16.01. Calc. for $C_9H_{11}NO_2S$ (%): C, 54.80; H, 5.62; N, 7.10; S, 16.26.

1-(4-Chlorophenyl)thio-2-nitropropane **3c**. R_f 0.42 (light petroleum–EtOAc, 3:1), oil. Found (%): C, 46.12; H, 4.39; N, 5.12; S, 13.62; Cl, 15.06. Calc. for $C_9H_{10}ClNO_2S$ (%): C, 46.65; H, 4.35; N, 6.05; S, 13.84; Cl, 15.30.

1-(4-Chlorophenyl)thio-2-nitroethane **3d**. R_f 0.40 (light petroleum–EtOAc, 10:1), oil. Found (%): C, 44.16; H, 3.64; N, 6.18; S, 14.72; Cl, 16.27. Calc. for $C_8H_8ClNO_2S$ (%): C, 44.14; H, 3.70; N, 6.43; S, 14.73; Cl, 16.29.

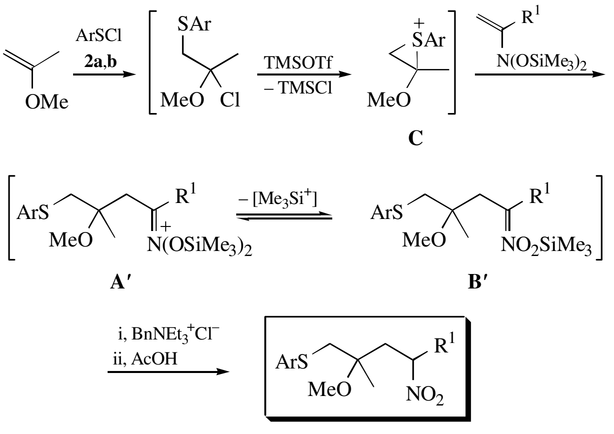
1-Nitro-2-(4-chlorophenyl)thiopropane **3e**. R_f 0.58 (light petroleum–EtOAc, 3:1), oil. Found (%): C, 46.77; H, 4.22; N, 6.31; S, 13.71; Cl, 15.22. Calc. for $C_9H_{10}ClNO_2S$ (%): C, 46.65; H, 4.35; N, 6.05; S, 13.84; Cl, 15.30.

Methyl 4-nitro-5-(4-chlorophenyl)thiopentanoate **3f**. R_f 0.34 (light petroleum–EtOAc, 3:1), oil. Found (%): C, 47.86; H, 4.72; N, 4.86; S, 9.84; Cl, 10.88. Calc. for $C_{12}H_{14}ClNO_4S$ (%): C, 47.75; H, 4.65; N, 4.61; S, 10.56; Cl, 11.67.

ethers (ethyl vinyl ether, dihydropyrane) in this coupling also failed: under standard conditions, no reaction takes place. Probably, less stable unsubstituted episulfonium cations are not formed at low temperature, while an increase in the temperature leads to the decomposition of BENA by Me$_3$SiOTf.

In summary, we found that sulfur-containing electrophiles can be used in the functionalization of nitro compounds through the silylation of the latter.

This work was performed at the Scientific Educational Centre for Young Scientists and supported by the Russian Foundation for Basic Research (grant no. 99-03-32015), the Special Programme ‘Integration’ (project nos. A0082 and B0062) and the US CRDF (grant no. RC-2 2207).



4a–c

BENA	R^1	ArSCl	Ar	Product	Yield (%)
1a	Me	**2a**	*p*-Tol	**4a**	53
1d	(CH$_2$)$_2$CO$_2$Me	**2a**	*p*-Tol	**4b**	37
1a	Me	**2b**	*p*-ClC$_6$H$_4$	**4c**	57

Scheme 2

References

1 A. V. Lesiv, S. L. Ioffe, Yu. A. Strelenko and V. A. Tartakovskii, *Helv. Chim. Acta*, 2002, **85**, 3489.

2 A. A. Tishkov, A. D. Dilman, V. I. Faustov, A. A. Birukov, K. A. Lysenko, P. A. Belyakov, S. L. Ioffe, Yu. A. Strelenko and M. Yu. Antipin, *J. Am. Chem. Soc.*, 2002, **124**, 11358.

3 A. D. Dilman, I. M. Lyapkalo, S. L. Ioffe, Yu. A. Strelenko and V. A. Tartakovsky, *Synthesis*, 1999, 1767.

[§] NMR spectra were recorded on a Bruker AM-300 instrument in CDCl$_3$ as a solvent. Chemical shifts were measured relative to internal Me$_4$Si (δ = 0; ^{1}H; ^{13}C) or external MeNO$_2$ (δ = 0; ^{14}N) standards.

3a: ^{1}H NMR, δ: 1.59 (d, 3H, CH*Me*, J 6.6 Hz), 2.33 (s, 3H, *Me*Ar), 3.07 (dd, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$, J 6.6, 14.0 Hz), 3.42 (dd, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$, J 7.4, 14.0 Hz), 4.49–4.61 (m, 1H, CHNO$_2$), 7.13 (d, 2H, J 8.1 Hz) and 7.32 (d, 2H, J 8.1 Hz). ^{13}C NMR, δ: 18.7 (*Me*CH), 21.1 (*Me*Ar), 39.2 (CH$_2$), 82.1 (CHNO$_2$), 129.8 (C$_i$), 130.1 (2CH$_\mathrm{Ar}$), 132.1 (2CH$_\mathrm{Ar}$), 138.1 (C$_i$). ^{14}N NMR, δ: 11.5 ($\Delta\nu_{1/2}$ 290 Hz).

3b: ^{1}H NMR, δ: 2.34 (s, 3H, *Me*Ar), 3.37 (t, 2H, CH$_2$S, J 7.2 Hz), 4.47 (t, 2H, CH$_2$NO$_2$, J 7.2 Hz), 7.15 (d, 2H, J 8.2 Hz) and 7.34 (d, 2H, J 8.2 Hz). ^{13}C NMR, δ: 21.1 (Me), 31.8 (CH$_2$S), 74.0 (CH$_2$NO$_2$), 129.2 (C$_i$), 130.2 (2CH$_\mathrm{Ar}$), 132.3 (2CH$_\mathrm{Ar}$), 138.3 (C$_i$). ^{14}N NMR, δ: 0.2 ($\Delta\nu_{1/2}$ 100 Hz).

3c: ^{1}H NMR, δ: 1.60 (d, 3H, CH*Me*, J 6.6 Hz), 3.13 (dd, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$, J 6.6, 14.7 Hz), 3.44 (dd, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$, J 7.7, 14.7 Hz), 4.51–4.63 (m, 1H, CHNO$_2$), 7.25–7.37 (m, 4H, CH$_\mathrm{Ar}$). ^{13}C NMR, δ: 18.7 (*Me*CH), 38.7 (CH$_2$), 82.0 (CHNO$_2$), 129.5 (C$_i$), 132.0 (2CH$_\mathrm{Ar}$), 132.7 (2CH$_\mathrm{Ar}$), 133.9 (C$_i$). ^{14}N NMR, δ: 10.7 ($\Delta\nu_{1/2}$ 154 Hz).

3d: ^{1}H NMR, δ: 3.40 (t, 2H, CH$_2$S, J 7.0 Hz), 4.48 (t, 2H, CH$_2$NO$_2$, J 7.0 Hz), 7.26–7.38 (m, 4H, CH$_\mathrm{Ar}$). ^{13}C NMR, δ: 31.3 (CH$_2$S), 73.8 (CH$_2$NO$_2$), 129.6 (2CH$_\mathrm{Ar}$), 131.5 (C$_i$), 132.9 (2CH$_\mathrm{Ar}$), 134.1 (C$_i$). ^{14}N NMR, δ: –0.5 ($\Delta\nu_{1/2}$ 133 Hz).

3e: ^{1}H NMR, δ: 1.37 (d, 3H, Me, J 6.6 Hz), 3.70–3.86 (m, 1H, CH), 4.26–4.50 (m, 2H, CH$_\mathrm{A}$H$_\mathrm{B}$NO$_2$), 7.31 (d, 2H, CH$_\mathrm{Ar}$, J 8.1 Hz), 7.40 (d, 2H, CH$_\mathrm{Ar}$, J 8.1 Hz). ^{13}C NMR, δ: 18.3 (Me), 40.7 (CHS), 79.8 (CH$_2$NO$_2$), 129.5 (2CH$_\mathrm{Ar}$), 130.0 (C$_i$), 135.0 (2CH$_\mathrm{Ar}$), 136.7 (C$_i$). ^{14}N NMR, δ: 7.0 ($\Delta\nu_{1/2}$ 152 Hz).

3f: ^{1}H NMR, δ: 2.15–2.45 (m, 4H, CH$_2$CH$_2$), 3.17 (dd, 1H, CH$_\mathrm{A}$S, J 14.2, 5.5 Hz), 3.39 (dd, 1H, CH$_\mathrm{B}$S, J 14.4, 8.2 Hz), 3.65 (s, 3H, OMe), 4.47–4.69 (m, 2H, CHNO$_2$), 7.27 (d, 2H, CH$_\mathrm{Ar}$, J 8.7 Hz), 7.39 (d, 2H, CH$_\mathrm{Ar}$, J 8.7 Hz). ^{13}C NMR, δ: 28.0, 29.8 (CH$_2$), 37.6 (CH$_2$S), 52.0 (OMe) 86.1 (CH$_2$NO$_2$), 129.5 (2CH$_\mathrm{Ar}$), 131.8 (C$_i$), 133.0 (2CH$_\mathrm{Ar}$), 134.1 (C$_i$), 171.9 (C=O). ^{14}N NMR, δ: 7.0 ($\Delta\nu_{1/2}$ 343 Hz).

[¶] *General preparation procedure for* **4a–c**. 2-Methoxypropene (1 mmol), Pr$^\mathrm{i}_2$NEt (3.5 µl) and a BENA **1** (1 mmol) solution in CH$_2$Cl$_2$ (0.5 ml) were added successively to a solution of ArSCl (1 mmol) in CH$_2$Cl$_2$ (2.3 ml) at –78 °C. Then, a TMSOTf (1.1 mmol, 0.21 ml) solution in CH$_2$Cl$_2$ (1 ml) was added dropwise. After stirring for 30 min at –78 °C, BnNEt$_3^+$Cl$^-$ (1.4 mmol) in CH$_2$Cl$_2$ (2 ml), Et$_3$N (2.2 mmol) + AcOH (2 mmol) in CH$_2$Cl$_2$ (2 ml), and AcOH (2 mmol) in MeOH (2 ml) solutions were added successively. The resulting mixture was warmed to room temperature and stirred for 30 min; then, it was poured into a two-phase mixture of 20 ml of Et$_2$O and 20 ml of H$_2$O; the aqueous layer was extracted three times with Et$_2$O; the organic phase was washed with brine, dried over Na$_2$SO$_4$ and concentrated *in vacuo*. Purification of the residue using flash chromatography (SiO$_2$, light petroleum–EtOAc, 10:1 to 3:1) gave *1-(4-tolyl)thio-2-methoxy-4-nitro-2-methylpentane* **4a**: Found (%): C, 59.57; H, 7.39; N, 4.70; S, 11.29. Calc. for C$_{14}$H$_{21}$NO$_3$S (%): C, 59.34; H, 7.47; N, 4.94; S, 11.32. Isomer ratio *ca.* 1:1. *Upper isomer,* R_f 0.42 (light petroleum–EtOAc, 3:1), oil. *Lower isomer,* R_f 0.38 (light petroleum–EtOAc, 3:1), oil.

Methyl 4-nitro-6-methoxy-7-(4-tolyl)thio-6-methylheptanoate **4b**: Found (%): C, 57.58; H, 6.92; N, 3.78; S, 8.77. Calc. for C$_{17}$H$_{25}$NO$_5$S (%): C, 57.44; H, 7.09; N, 3.94; S, 9.02. Isomer ratio *ca.* 1:1. *Upper isomer,* R_f 0.31 (light petroleum–EtOAc, 3:1), oil. *Lower isomer,* R_f 0.26 (light petroleum–EtOAc, 3:1), oil.

1-(4-Chlorophenyl)thio-2-methoxy-4-nitro-2-methylpentane **4c**: Found (%): C, 51.27; H, 5.94; N, 5.12; S, 10.77; Cl, 11.79. Calc. for C$_{13}$H$_{18}$ClNO$_3$S (%): C, 51.39; H, 5.97; N, 4.61; S, 10.55; Cl, 11.67. Isomer ratio *ca.* 1:1. *Upper isomer,* R_f 0.37 (light petroleum–EtOAc, 3:1), oil. *Lower isomer,* R_f 0.33 (light petroleum–EtOAc, 3:1), oil.

[††] **4a**. *Upper isomer:* ^{1}H NMR, δ: 1.23 [s, 3H, *Me*C(OMe)], 1.47 (d, 3H, CH*Me*, J 6.6 Hz), 2.02 (dd, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$CH, J 2.6, 15.1 Hz), 2.33 (s, 3H, *Me*Ar), 2.48 (dd, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$CH, J 9.8, 15.1 Hz), 3.05 (d, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$S, J 13.1 Hz), 3.14 (s, 3H, OMe), 3.16 (d, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$S, J 13.1 Hz), 4.68–4.85 (m, 1H, CHNO$_2$), 7.12 (d, 2H, J 8.2 Hz) and 7.31 (d, 2H, J 8.2 Hz). ^{13}C NMR, δ: 21.0, 21.5, 21.6 (all C*Me*), 41.9, 43.1 (2CH$_2$), 49.6 (OMe), 76.1 (*C*OMe), 79.7 (CHNO$_2$), 129.8 (2CH$_\mathrm{Ar}$), 130.43 (2CH$_\mathrm{Ar}$), 132.7 (C$_i$), 136.6 (C$_i$). *Lower isomer*: ^{1}H NMR, δ: 1.27 [s, 3H, *Me*C(OMe)], 1.53 (d, 3H, CH*Me*, J 6.6 Hz), 1.84 (dd, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$CH, J 3.6, 15.1 Hz), 2.64 (dd, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$CH, J 8.5, 15.1 Hz), 2.33 (s, 3H, *Me*Ar), 3.00–3.21 (m, 5H, OMe + CH$_2$S), 4.68–4.85 (m, 1H, CHNO$_2$), 7.10 (d, 2H, J 8.0 Hz) and 7.29 (d, 2H, J 8.0 Hz). ^{13}C NMR, δ: 21.0, 21.4, 21.8 (all C*Me*), 42.5, 43.2 (2CH$_2$), 49.5 (OMe), 76.0 (*C*OMe), 79.6 (CHNO$_2$), 129.7 (2CH$_\mathrm{Ar}$), 130.40 (2CH$_\mathrm{Ar}$), 132.9 (C$_i$), 136.5 (C$_i$). ^{14}N NMR, δ: 13.9 ($\Delta\nu_{1/2}$ 310 Hz).

4b: *Upper isomer:* ^{1}H NMR, δ: 1.24 [s, 3H, *Me*C(OMe)], 1.89–2.51 (m, 4H, CH$_2$CH$_2$), 2.33 (s, 3H, *Me*Ar), 2.98–3.27 (m, 2H, C*H*$_\mathrm{A}$H$_\mathrm{B}$S), 3.13 (s, 3H, OMe), 3.69 (s, 3H, COOMe), 4.61–4.83 (m, 1H, CHNO$_2$), 7.13 (d, 2H, CH$_\mathrm{Ar}$, J 7.8 Hz), 7.32 (d, 2H, CH$_\mathrm{Ar}$, J 7.8 Hz). ^{13}C NMR, δ: 20.9, 21.6 (all C*Me*), 29.8, 30.1, 40.6, 42.9 (all CH$_2$), 49.6 [MeC(O*Me*)], 51.8 (CO$_2$*Me*), 76.2 [*C*(OMe)], 83.5 (CHNO$_2$), 129.8 (2CH$_\mathrm{Ar}$), 130.6 (2CH$_\mathrm{Ar}$), 132.6 (C$_i$), 136.8 (C$_i$), 172.1 (C=O). *Lower isomer:* ^{1}H NMR, δ: 1.28 [s, 3H, *Me*C(OMe)], 1.79–2.41 (m, 4H, CH$_2$CH$_2$), 2.32 (s, 3H, *Me*Ar), 2.53–2.82 and 3.00–3.29 (m, 2H, C*H*$_\mathrm{A}$H$_\mathrm{B}$S), 3.11 (s, 3H, OMe), 3.68 (s, 3H, COOMe), 4.57–4.86 (m, 1H, CHNO$_2$), 7.10 (d, 2H, CH$_\mathrm{Ar}$, J 7.9 Hz), 7.29 (d, 2H, CH$_\mathrm{Ar}$, J 7.9 Hz). ^{13}C NMR, δ: 21.0, 21.6 (all C*Me*), 29.8, 30.3, 41.5, 43.2 (all CH$_2$), 49.6 [MeC(O*Me*)], 51.8 (CO$_2$*Me*), 76.1 [*C*(OMe)], 83.6 (CHNO$_2$), 129.8 (2CH$_\mathrm{Ar}$), 130.5 (2CH$_\mathrm{Ar}$), 132.8 (C$_i$), 136.6 (C$_i$), 172.2 (C=O). ^{14}N NMR, δ: 14.7 ($\Delta\nu_{1/2}$ 387 Hz).

4c: *Upper isomer:* ^{1}H NMR, δ: 1.22 [s, 3H, *Me*C(OMe)], 1.50 (d, 3H, CH*Me*, J 7.2 Hz), 1.99 (dd, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$CH, J 2.3, 15.4 Hz), 2.47 (dd, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$CH, J 8.9, 15.4 Hz), 2.97–3.18 (m, 2H, C*H*$_\mathrm{A}$H$_\mathrm{B}$S), 3.13 (s, 3H, OMe), 4.69–4.87 (m, 1H, CHNO$_2$), 7.18–7.38 (m, 4H, CH$_\mathrm{Ar}$). ^{13}C NMR, δ: 21.5 (2Me), 42.2, 42.8 (2CH$_2$), 49.7 (OMe), 75.8 (*C*OMe), 79.7 (CHNO$_2$), 129.1 (2CH$_\mathrm{Ar}$), 131.0 (2CH$_\mathrm{Ar}$), 132.4 (C$_i$), 135.1 (C$_i$). ^{14}N NMR, δ: 11.4 ($\Delta\nu_{1/2}$ 246.2 Hz). *Lower isomer:* ^{1}H NMR, δ: 1.28 [s, 3H, *Me*C(OMe)], 1.54 (d, 3H, CH*Me*, J 7.2 Hz), 1.82 (dd, 1H, C*H*$_\mathrm{A}$H$_\mathrm{B}$CH, J 3.6, 15.4 Hz), 2.65 (dd, 1H, CH$_\mathrm{A}$*H*$_\mathrm{B}$CH, J 8.5, 15.7 Hz), 3.04 (s, 2H, C*H*$_\mathrm{A}$H$_\mathrm{B}$S), 3.12 (s, 3H, OMe), 4.69–4.84 (m, 1H, CHNO$_2$), 7.20–7.33 (m, 4H, CH$_\mathrm{Ar}$). ^{13}C NMR, δ: 21.5, 21.9 (2Me), 42.6, 42.9 (2CH$_2$), 49.7 (OMe), 75.9 (*C*OMe), 79.6 (CHNO$_2$), 129.1 (2CH$_\mathrm{Ar}$), 131.0 (2CH$_\mathrm{Ar}$), 132.4 (C$_i$), 135.2 (C$_i$). ^{14}N NMR, δ: 11.7 ($\Delta\nu_{1/2}$ 254.3 Hz).

4 A. V. Ustinov, A. D. Dilman, S. L. Ioffe, P. A. Belyakov and Yu. A. Strelenko, *Izv. Akad. Nauk, Ser. Khim.*, 2002, 1343 (*Russ. Chem. Bull., Int. Ed.*, 2002, **51**, 1455).
5 A. D. Dilman, I. M. Lyapkalo, S. L. Ioffe, Yu. A. Strelenko and V. A. Tartakovsky, *J. Org. Chem.*, 2000, **65**, 8826.
6 A. D. Dilman, S. L. Ioffe and H. Mayr, *J. Org. Chem.*, 2001, **66**, 3196.
7 A. D. Dilman, A. A. Tishkov, I. M. Lyapkalo, S. L. Ioffe, Yu. A. Strelenko and V. A. Tartakovsky, *Synthesis*, 1998, 181.

Received: 30th January 2003; Com. 03/2046