A Novel Catalyst-Free One-Pot Synthesis of Some New *N*-(α-Hydroxybenzyl)formamides by Treatment of 2,2-Dichloroaziridines with Dimethyl Sulfoxide and Water under Neutral Conditions

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A novel, mild, and efficient method is reported for the preparation of new *N*-(α-hydroxybenzyl)-formamides in excellent yields and appropriate reaction times through the reaction of 2,2-dichloroaziridines with aqueous DMSO as the O-donor under neutral conditions at 70–85°.

Introduction. – The formyl group is an important amino protecting group in peptide synthesis [1]. Formamides have wide applications as intermediates in the preparation of pharmaceuticals such as fluoroquinolines [2], substituted 1-aryl-1*H*-imidazoles [1], 1,2-dihydroquinolines, N-bridged heterocycles, oxazolidinones, cancer chemotherapeutic agents, and as important reagents for *Vilsmeier* formylation [2a][3–6]. They are also in use as *Lewis* base catalysts in organic transformations such as allylation [7] and hydrosilylation [8] of carbonyl compounds. In addition, the *N*-formyl derivatives are useful precursors in the preparation of *N*-methyl compounds [9], formamidines [10], and isocyanide [11–13]. Also, L-pipecolinic acid-derived 1-formyl derivatives have been developed as new *Lewis*-basic organocatalysts that promote the asymmetric reduction of *N*-arylketimines with trichlorosilane as the reducing agent [14]. For this reason, numerous methods have been reported for the preparation of formamide derivatives by *N*-formylation of amines [15–18].

Aziridines are highly reactive molecules, in part due to ring strain [19]. As a consequence of their high reactivity, these small heterocycles play an important role in organic chemistry and as intermediates in the synthesis of organic, pharmaceutical [20], and natural product intermediates. Due to the widespread applications of aziridines, a number of articles have been published about these compounds [21–25].

In this study, with attention to the importance and significant applications of formamide derivatives, we elaborated a novel method for the synthesis of new *N*-(α-hydroxybenzyl)formamides (= *N*-(arylhydroxymethyl)formamides) through the reaction of 2,2-dichloroaziridines with DMSO/H_2O at temperatures between 70–85°, *i.e.*, in the absence of an activating reagent and under neutral conditions.

Results and Discussion. – In the *Scheme*, the preparation of *N*-(α-hydroxybenzyl)-formamide derivatives **5a**–**5i** from 2,2-dichloroaziridines **4a**–**4i** is shown. The latter were obtained by treatment of *Schiff* bases **3a**–**3i** with dichlorocarbene [26–33]

generated from $CHCl_3$ and NaOH under phase-transfer-catalysis (PTC) conditions (CTAB = cetyltrimethylammonium bromide = *N*,*N*,*N*-trimethylhexadecanaminium bromide).

Hydrolysis of *geminal*-dihalo compounds to the corresponding carbonyl compounds often employs harsh reaction conditions, such as strong acids or strong bases [35]. This is not the case with 2,2-dichloroaziridines which are valuable precursors for the preparation of pharmacologically active compounds such as indolinones [28], isoquinolines [36], and N-containing building blocks such as amidines [37] and aziridinones [38]. In contrast to their carbocyclic analogs, 2,2-dichloroaziridines undergo facile hydrolysis and alcoholysis [32a]. Thus, we first performed this hydrolysis with 2,2-dichloro-1,3-bis(4-chlorophenyl)aziridine (**4d**) as a model in DMSO/H_2O at various temperatures (see *Table 1*). When the reaction was carried out at 70°, as the best condition, the desired product **5d** was obtained in excellent yield (*Entry 5*). No product was formed at room temperature even after 72 h (*Entry 7*).

Scheme. *Synthesis of* N-*(α-Hydroxybenzyl)formamide Derivatives* [32a][26][34]

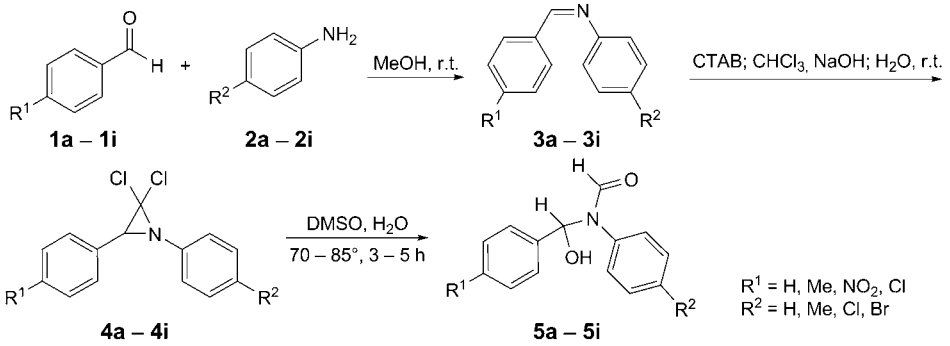
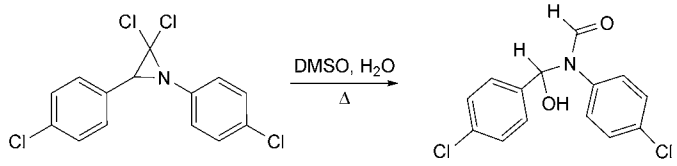


Table 1. *Temperature Effect on the Formation of* **5d**[a])

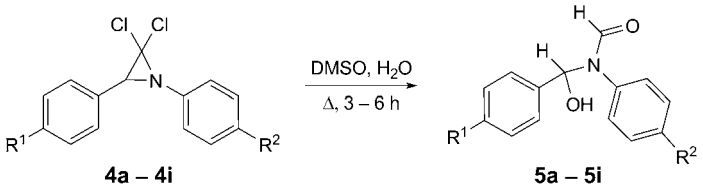


Entry	Temperature [°]	Time [h]	Yield [%][b])
1	50	14	15
2	55	11	30
3	60	7	60
4	65	5	85
5	70	3	98
6	85	3	98
7	r.t.	72	–

[a]) Reaction conditions: 4d (0.01 mol), DMSO (6 ml), and H_2O (1 ml). [b]) Yield of isolated **5d**.

In view of the above results, conversion of different 2,2-dichloroaziridines **4** to the corresponding *N*-(α-hydroxybenzyl)formamides **5** in DMSO/H_2O was investigated. As can be seen in *Table 2*, when 0.01 mol of **4** were treated with 6 ml of DMSO and 1 ml of H_2O at a temperature between 70° and 85°, the corresponding *N*-(α-hydroxybenzyl)-formamides **5** were obtained in excellent yields and appropriate reaction times.

Table 2. *Synthesis of* N-*(Hydroxybenzyl)formamides*[a])[b])



4a – 4i **5a – 5i**

Entry	R^1	R^2	Product	Temperature [°]	Time [h]	Yield [%][c])
1	H	H	**5a**	80	6	90
2	H	Br	**5b**	85	5	94
3	H	Cl	**5c**	85	5	92
4	Cl	Cl	**5d**	70	3	98
5	Cl	Br	**5e**	70	3	98
6	NO_2	Me	**5f**	85	4	92
7	Me	Br	**5g**	70	3	96
8	Cl	Me	**5h**	70	3	97
9	NO_2	Br	**5i**	85	4	90

[a]) The synthesis of the 2,2-dichloroaziridines **4** has been described in [26]. [b]) Reaction conditions: 2,2-dichloroaziridine (0.01 mol), DMSO (6 ml), and H_2O (1 ml). [c]) Yield of isolated product **5** based on starting material **4**.

The structure of the products **5** was confirmed by spectroscopic methods such as IR, ^{1}H- and ^{13}C-NMR, and mass spectroscopy. In the IR spectra, the stretching frequency of aromatic C=C bonds was observed between 1489 and 1598 cm^{-1}. The stretching vibration of the O–H group appeared at 3285–3340 cm^{-1}, and the absorption in the region between 1639–1659 cm^{-1} was assigned to the formyl group. In the ^{1}H-NMR spectra, the C*H*–OH group appeared at δ(H) 5.09–5.28. The signals at δ(H) 6.99–8.23 were assigned to the aromatic protons. The H–C=O proton was observed at δ(H) 9.97–10.11, and the OH signal appeared at δ(H) 6.40–6.80. In the ^{13}C-NMR spectra, δ(C) 73.0–74.1 was assigned to the O–C–N group and δ(C) 171.0–172.0 to the H–C=O group. The mass spectra (EI) of all products showed the corresponding molecular-ion peak.

Conclusion. – A simple and efficient method for the transformation of 2,2-dichloroaziridines into the corresponding *N*-(α-hydroxybenzyl)formamides was elaborated. The reaction could be carried out conveniently and proceeded with excellent yield and purity of products. To the best of our knowledge, this is the first conversion of a 2,2-dichloroaziridine into the corresponding formamide with DMSO/H_2O as the sole reagent.

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Experimental Part

General. All the materials were of commercial reagent grade. The *Schiff* bases and 2,2-dichloroaziridines were prepared and characterized according to our previously reported procedures [26][39–41]. M.p.: *Yanagimoto* micro-melting-point apparatus; uncorrected. TLC: SiO_2 *Polygram-SIL-G/UV 254* plates (*Merck*). IR Spectra: *Perkin–Elmer-781* and *Impact-400-Nicolet-FTIR* spectrophotometer; KBr pellets; $\tilde{\nu}$ in cm^{-1}. ^1H- and ^{13}C-NMR Spectra: *Bruker-DRX-400* spectrometer; in $(D_6)DMSO$; δ in ppm rel. to Me_4Si as internal standard, J in Hz. MS: *Finnigan-MAT-44S* instrument, electron impact (EI) ionization, ionization voltage 70 eV; in m/z (rel. %).

Schiff *Bases* **3a**–**3i**. In a typical reaction, 2 mmol of aldehyde **1** was added to the aniline **2** (2 mmol) in MeOH (3 ml) and mechanically stirred at r.t. (TLC monitoring). After completion of the reaction, the crude product was purified by recrystallization from petroleum ether: pure *Schiff* bases **3a**–**3i** with good to excellent yields. The *Schiff* bases were identified by spectroscopic data [39][40].

2,2-Dichloroaziridines **4a**–**4i**. Measured quantities of NaOH (0.08 mol, 3.2 g) and *N*-cetyl-*N,N,N*-trimethylammonium bromide (CTAB; 0.3 g) were dissolved in 30 ml of H_2O. The mixture was uniformly agitated at 70–85°. Then, *Schiff* base **3** (0.027 mol) in $CHCl_3$ (9.5 ml, 0.08 mol) was added dropwise to the soln. (TLC monitoring). After completion of the reaction, the aq. phase, was extracted twice by Et_2O and the combined extract dried ($MgSO_4$) and concentrated: 2,2-dichloro-1,3-diarylaziridines **4a**–**4i** in excellent yields. Pale yellow solids which were identified by physical and spectroscopic data [26][41].

N-*(4-Chlorophenyl)*-N-*[4-(chlorophenyl)hydroxymethyl]formamide* (**5d**): *Typical Procedure*. A soln. of 2,2-dichloro-1,3-bis(4-chlorophenyl)aziridine (**4d**; 3.3 g, 0.01 mol) in 6 ml (0.08 mol) of DMSO and 1 ml of H_2O was heated at 70° in an oil bath (TLC monitoring). After completion of the reaction, the mixture was poured into cold H_2O (15 ml), the resulting mixture extracted with CH_2Cl_2 (2 × 10 ml), and the org. layer dried (Na_2SO_4) and concentrated: crude **5d** as a white solid. The crude product was recrystallized from EtOH to give **5d** in 98% yield in high purity. For data, see below. Formarides **5a**–**5c** and **5e**–**5i** were prepared similarly (*cf. Table 2*).

N-*(Hydroxyphenylmethyl)*-N-*phenylformamide* (**5a**): Yield 2.045 g (90%). White solid. M.p. 120–122°. IR (KBr): 3300 (OH), 3090, 2930, 1649 (C=O), 1509, 1590 (C=C, Ar). ^1H-NMR ($(D_6)DMSO$): 5.09 (*s*, C*H*–OH); 6. 48 (*s*, OH); 6.99–7.56 (*m*, 10 arom. H); 10.01 (*s*, CHO). ^{13}C-NMR ($(D_6)DMSO$): 74.1; 121.0; 127.0; 128.0; 131.1; 132.0; 137.0; 138.1 141.1; 171.0. MS: 228 (3, $[M+1]^+$), 227 (13, M^+), 120 (45), 107 (74), 77 (100). Anal. calc. for $C_{14}H_{13}NO_2$ (227.26): C 73.99, H 5.77, N 6.16; found: C 74.08, H 5.80, N 6.18.

N-*(4-Bromophenyl)*-N-*(hydroxyphenylmethyl)formamide* (**5b**): Yield 2.878 g (94%). Pale yellow solid. M.p. 135–137°. IR (KBr): 3307 (OH), 3088, 2930, 1659 (C=O), 1514, 1588 (C=C, Ar). ^1H-NMR ($(D_6)DMSO$): 5.15 (*s*, C*H*–OH); 6.50 (*s*, OH); 7.34–7.69 (*m*, 9 arom. H); 10.00 (*s*, CHO). ^{13}C-NMR ($(D_6)DMSO$): 73.0; 119.4; 122.8; 128.9; 129.0; 131.9; 132.3; 136.0; 151.0; 171.0. MS: 307 (13, M^+ (^{81}Br)), 305 (13, M^+ (^{79}Br)), 200 (39), 198 (39), 171 (65), 169 (65), 157 (46), 155 (46), 107 (75), 77 (100). Anal. calc. for $C_{14}H_{12}BrNO_2$ (306.17): C 54.92, H 3.95, N 4.57; found: C 55.18, H 4.06, N 4.59.

N-*(4-Chlorophenyl)*-N-*(hydroxyphenylmethyl)formamide* (**5c**): Yield 2.408 g (92%). White solid. M.p. 127–129°. IR (KBr): 3267 (OH), 3090, 2900, 1653 (C=O), 1599, 1492 (C=C, Ar). ^1H-NMR ($(D_6)DMSO$): 5.08 (*s*, C*H*–OH); 6. 47 (*s*, OH); 7.33–7.72 (*m*, 9 arom. H); 10.08 (*s*, CHO). ^{13}C-NMR ($(D_6)DMSO$): 74.0; 121.8; 127.0; 128.4; 128.6; 129.0; 129.1; 137.9 141.0; 171.8. MS: 263 (6, M^+ (^{37}Cl)), 261 (18, M^+ (^{35}Cl)), 156 (13), 154 (35), 127 (18), 125 (55), 107 (60), 77 (100). Anal. calc. for $C_{14}H_{12}ClNO_2$ (261.71): C 64.25, H 4.62, N 5.35; found: C 64.38, H 4.72, N 5.36.

N-*(4-Chlorophenyl)*-N-*[(4-chlorophenyl)hydroxymethyl]formamide* (**5d**): Yield 2.902 g (98%). White solid. M.p. 138–140°. IR (KBr): 3285 (OH), 3090, 2900, 1639 (C=O), 1590, 1490 (C=C, Ar). ^1H-NMR ($(D_6)DMSO$): 5.07 (*s*, C*H*–OH); 6. 50 (*s*, OH); 7.33, 7.41, 7.52, 7.73 (2 *AA′BB′*, $J_{AB} = 7.4$, 2.9, 8 arom. H); 10.11 (*s*, CHO). ^{13}C-NMR ($(D_6)DMSO$): 73.7; 121.8; 127.7; 128.6; 128.8; 128.9; 132.7; 137.9; 140.1; 171.5. MS: 300 (3), 298 (6), 296 (10, M^+), 156.5 (28), 154.5 (39), 143.5 (55), 141.5 (75), 127.5 (25),

125.5 (40), 113.5 (50), 111.5 (38), 77 (100). Anal. calc. for $C_{14}H_{11}Cl_2NO_2$ (296.15): C 56.78, H 3.74, N 4.72; found: C 56.86, H 3.80, N 4.81.

N-*(4-Bromophenyl)*-N-*[(4-chlorophenyl)hydroxymethyl]formamide* (**5e**): Yield 3.337 g (98%). White solid. M.p. 124–126°. IR (KBr): 3288 (OH), 3100, 2900, 1639 (C=O), 1589, 1488 (C=C, Ar). ^{1}H-NMR ((D$_6$)DMSO): 5.11 (*s*, C*H*–OH); 6. 58 (*s*, OH); 7.45–7.67 (*m*, 8 arom. H); 10.10 (*s*, CHO). ^{13}C-NMR ((D$_6$)DMSO): 73.7; 115.8; 122.2; 128.6; 128.8; 132.3; 132.8; 138.3; 140.1; 171.5. MS: 343 (4), 341 (15), 339 (10, M^+), 200 (18), 198 (18), 143 (55), 141 (70), 171 (48), 169 (48), 113.5 (45), 111.5 (60), 77 (100). Anal. calc. for $C_{14}H_{11}BrClNO_2$ (340.61): C 49.37, H 3.25, N 4.11; found: C 49.51, H 3.30, N 4.18.

N-*[Hydroxy(4-nitrophenyl)methyl]*-N-*(4-methylphenyl)formamide* (**5f**): Yield 2.634 g (92%). Yellow liquid. IR (KBr): 3340 (OH), 3090, 2910, 1649 (C=O), 1520, 1595 (C=C, Ar), 1350, 1530. ^{1}H-NMR ((D$_6$)DMSO): 2.21 (*s*, Me); 5.28 (*s*, C*H*–OH); 6.80 (*s*, OH); 7.07, 7.54, 7.79, 8.22 (2 *AA′BB′*, $J_{AB} = 7.4$, 2.9, 8 arom. H); 9.97 (*s*, CHO). ^{13}C-NMR ((D$_6$)DMSO): 21.0; 73.4; 120.5; 123.6; 127.9; 129.5; 134.0; 135.5; 147.4; 148.2; 170.5. MS: 287 (<2, $[M+1]^+$), 286 (11, M^+),152 (85), 134 (55), 122 (60), 105 (76), 91 (100), 77 (95). Anal. calc. for $C_{15}H_{14}N_2O_2$ (286.29): C 62.93, H 4.93, N 9.78; found: C 71.20, H 5.59, N 11.06.

N-*(4-Bromophenyl)*-N-*[hydroxy(4-methylphenyl)methyl]formamide* (**5g**): Yield 3.074 g (96%). Pale yellow solid. M.p. 155–157°. IR (KBr): 3285 (OH), 3090, 2910, 1642 (C=O), 1490, 1580 (C=C, Ar), 1350, 1530. ^{1}H-NMR ((D$_6$)DMSO): 2.27 (*s*, Me); 5.01 (*s*, C*H*–OH); 6. 40 (*s*, OH); 7.13–7.69 (*m*, 8 arom. H); 10.05 (*s*, CHO). ^{13}C-NMR ((D$_6$)DMSO): 22.0; 73.0; 120.0; 127.0; 129.1; 130.0; 132.0; 133.0; 135.0; 141.0; 171.0. MS: 321 (12, M^+ (^{81}Br)), 319 (12, M^+ (^{79}Br)), 200 (40), 198 (40), 171 (60), 169 (60), 157 (46), 155 (46), 120 (70), 91 (100), 77 (85). Anal. calc. for $C_{15}H_{14}BrNO_2$ (320.19): C 56.26, H 4.41, N 4.37; found: C 56.59, H 4.55, N 4.40.

N-*[(4-Chlorophenyl)hydroxymethyl]*-N-*(4-methylphenyl)formamide* (**5h**): Yield 2.675 g (96%). White solid. M.p. 142–144°. IR (KBr): 3286 (OH), 3090, 2920, 1638 (C=O), 1489, 1598 (C=C, Ar). ^{1}H-NMR ((D$_6$)DMSO): 2.23 (*s*, Me); 5.09 (*s*, C*H*–OH); 6. 50 (*s*, OH); 7.07–7.56 (*m*, 8 arom. H); 9.86 (*s*, CHO). ^{13}C-NMR ((D$_6$)DMSO): 20.9; 73.7; 120.2; 128.5; 128.8; 129.5; 132.7; 133.1; 136.2; 140.4; 171.0. MS: 277 (6, M^+ (^{37}Cl)), 275 (14, M^+ (^{35}Cl)), 143 (25), 141 (60), 134 (60), 113.5 (20), 111.5 (55), 105 (45), 91 (96), 77 (100). Anal. calc. for $C_{15}H_{14}ClNO_2$ (275.74): C 65.34, H 5.12, N 5.08; found: C 65.45, H 5.16, N 5.10.

N-*(4-Bromophenyl)*-N-*[hydroxy(4-nitrophenyl)methyl]formamide* (**5i**): Yield 3.160 g (90%). Yellow liquid. IR (KBr): 3289 (OH), 3100, 2900, 1649 (C=O), 1589, 1488 (C=C, Ar). ^{1}H-NMR ((D$_6$)DMSO): 5.10 (*s*, C*H*–OH); 6. 54 (s, OH); 6.95, 7.50, 7.71, 8.31 (2 *AA′BB′*, $J_{AB} = 7.4$, 2.9, 8 arom. H); 10.10 (*s*, CHO). ^{13}C-NMR ((D$_6$)DMSO): 75.9; 122.3; 128.9; 129.2; 129.7; 130.1; 132.1; 134.1; 143.6; 171.5. MS: 341 (10, M^+ (^{81}Br)), 339 (10, M^+ (^{79}Br)), 200 (18), 198 (18), 143.5 (55), 141.5 (70), 171 (48), 169 (48), 113.5 (45), 111.5 (60), 77 (100); Anal. calc. for $C_{14}H_{11}BrN_2O_4$ (351.17): C 47.88, H 3.15, N 7.98; found: C 48.10, H 3.20, N 8.03.

REFERENCES

[1] B.-C. Chen, M. S. Bednarz, R. Zhao, J. E. Sundeen, P. Chen, Z. Shen, A. P. Skoumbourdis, J. C. Barrish, *Tetrahedron Lett.* **2000**, *41*, 5453.

[2] a) K. Kobayashi, S. Nagato, M. Kawakita, O. Morikawa, H. Konishi, *Chem. Lett.* **1995**, *24*, 575; b) A. Jackson, O. Meth-Cohn, *J. Chem. Soc., Chem. Commun.* **1995**, 1319.

[3] I. M. Downie, M. J. Earle, H. Heaney, K. F. Shuhaibar, *Tetrahedron* **1993**, *49*, 4015.

[4] A. Kakehi, S. Ito, S. Hayashi, T. Fujii, *Bull. Chem. Soc. Jpn.* **1995**, *68*, 3573.

[5] B. B. Lohray, S. Baskaran, B. S. Rao, B. Y. Reddy, I. N. Rao, *Tetrahedron Lett.* **1999**, *40*, 4855.

[6] G. Petit, M. Kalnins, T. Liu, E. Thomas, K. Parent, *J. Org. Chem.* **1961**, *26*, 2563.

[7] S. Kobayashi, K. Nishio, *J. Org. Chem.* **1994**, *59*, 6620.

[8] S. Kobayashi, M. Yasuda, I. Hachiya, *Chem. Lett.* **1996**, *25*, 407.

[9] M. A. Kraus, *Synthesis* **1973**, 361.

[10] Y. Han, L. Cai, *Tetrahedron Lett.* **1997**, *38*, 5423.

[11] I. Ugi, *Angew. Chem., Int. Ed.* **1982**, *21*, 810; J. Waki, J. Meienhofer, *J. Org. Chem.* **1977**, *42*, 2019.

[12] F. Effenberger, J. Eichhorn, *Tetrahedron: Asymmetry* **1997**, *8*, 469.

[13] U. Schöllkopf, *Angew. Chem., Int. Ed.* **1977**, *16*, 339.
[14] P. Wu, Z. Wang, M. Cheng, L. Zhou, J. Sun, *Tetrahedron* **2008**, *64*, 11304.
[15] F. F. Blicke, C.-J. Lu, *J. Am. Chem. Soc.* **1952**, *74*, 3933; F. M. F. Chen, N. L. Benoiton, *Synthesis* **1979**, 709.
[16] H. Yale, *J. Org. Chem*. **1971**, *36*, 3238; L. Kisfaludy, L. Ötvös Jr., *Synthesis* **1987**, 510; W. Duczek, J. Deutsch, S. Vieth, H.-J. Niclas, *Synthesis* **1996**, 37.
[17] M. Mihara, Y. Ishino, S. Minakara, M. Komatsu, *Synthesis* **2003**, 2317.
[18] B. Das, M. Krishnaiah, P. Balasubramanyam, B. Veeranjaneyulu, D. N. Kumar, *Tetrahedron Lett*. **2008**, *49*, 2225; P. G. Reddy, G. D. K. Kumar, S. Bhaskaran, *Tetrahedron Lett.* **2000**, *41*, 9149.
[19] J. B. Sweeney, *Chem. Soc. Rev.* **2002**, *31*, 247.
[20] V. H. Dahanukar, L. A. Zavialov, *Curr. Opin. Drug Discovery Dev.* **2002**, *5*, 918.
[21] A. Padwa, S. S. Murphree, *Arkivoc* **2006**, *iii*, 6.
[22] 'Aziridines and Epoxides in Organic Synthesis', Ed. A. K. Yudin, Wiley-VCH, Weinheim, 2006.
[23] D. M. Hodgson, P. G. Humphreys, S. P. Hughes, *Pure Appl. Chem*. **2007**, *79*, 269.
[24] L. Marzorati, G. C. Barazzone, M. A. Bueno Filho, B. Wladislaw, C. D. Vitta, *Tetrahedron Lett*. **2007**, *48*, 6509.
[25] G. S. Singh, M. D'hooghe, N. De Kimpe, *Chem. Rev.* **2007**, *107*, 2080.
[26] H. Naeimi, K. Rabiei, *Chin. Chem. Lett.* **2011**, *22*, 1273.
[27] A. F. Khlebnikov, M. S. Novikov, M. V. Golovkina, P. P. Petrovskii, A. S. Konev, D. S. Yufit, H. Stoeckli-Evans, *Org. Biomol. Chem*. **2011**, *9*, 3886.
[28] M. Mihara, Y. Ishino, S. Minakata, M. Komatsu, *J. Org. Chem*. **2005**, *70*, 5320.
[29] R. R. Kostikov, A. F. Khlebnikov, K. A. Oglobin, *Zh. Org. Khim*. **1977**, *13*, 1857.
[30] A. F. Khlebnikov, K. A. Oglobin, *Zh. Org. Khim*. **1973**, 9, 2346.
[31] G. A. Yost, L. L. Miller, *J. Agric. Food Chem*. **1976**, *24*, 724.
[32] a) E. K. Fields, S. M. Sandri, *Chem. Ind*. (*London*) **1959**, 1216; b) A. G. Cook, E. K. Fields, *J. Org. Chem*. **1962**, *27*, 3686.
[33] M. K. Meilahn, D. K. Olsen, W. J. Brittain, R. T. Anders, *J. Org. Chem*. **1978**, *43*, 1346; J. Graefe, *Z. Chem*. **1974**, *14*, 469.
[34] M. Amanullah, S. K. Sadozai, W. Rehman, Z. H. A. Rauf, M. Iqbal, *African J. Biotechnol.* **2011**, *10*, 209.
[35] S. J. Chung, D. H. Kim, *Tetrahedron* **1995**, *51*, 12549; M. S. Goodman, A. D. Hamilton, J. Weiss, *J. Am. Chem. Soc*. **1995**, *117*, 8447.
[36] A. F. Khlebnikov, T. Y. Nikiforova, M. S. Novikov, R. R. Kostikov, *Synthesis* **1997**, 677.
[37] M. K. Meilahn, L. L. Augenstein, J. L. McManaman, *J. Org. Chem.* **1971**, *36*, 3627.
[38] Y. Ohshiro, H. Ohnishi, M. Komatsu, *J. Jpn. Oil Chem. Soc.***1987**, *36*, 884.
[39] H. Naeimi, F. Salimi, K. Rabiei, *J. Mol. Catal. A: Chem.* **2006**, *260*, 100; H. Naeimi, H. Sharghi, F. Salimi, K. Rabiei, *Heteroatom Chem.* **2008**, *19*, 43.
[40] H. Schiff, *Ann. Chem. Pharm. Suppl*. **1864**, *3*, 343.
[41] K. Rabiei, Ph.D. Thesis, University of Kashan, Iran, 2010.

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