J IRAN CHEM SOC (2013) 10:161–167
DOI 10.1007/s13738-012-0137-9

ORIGINAL PAPER

Nanocrystalline magnesium oxide as a solid base catalyst promoted one pot synthesis of *gem*-dichloroaziridine derivatives under thermal conditions

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Received: 17 December 2011 / Accepted: 5 July 2012 / Published online: 26 July 2012

Abstract In this study, efficient and mild synthesis of *gem*-dichloroaziridines from Schiff bases in the presence of nanocrystalline magnesium oxide/chloroform as a novel source of dichlorocarbene intermediate under thermal conditions have been described. The reaction is dramatically enhanced in the presence of nanocrystalline magnesium oxide, and any byproducts were detected during or after the reaction. The corresponding products have been obtained in excellent yields, high purity and short reaction times.

Keywords Aziridine · Nanocrystalline magnesium oxide · Schiff base · Dichlorocarbene · Catalyst

Introduction

Nanocrystalline oxides are of immense interest to scientist due to their enriched surface chemistry and high surface area. Ultrafine nanocrystallines of alkaline earth metal oxide such as magnesium oxide has many applications as catalyst [1–3], refractory materials [4], optically transparent ceramic windows [5, 6], etc. In the field of catalyst, MgO has strong basic properties, which are associated with catalysts by base in many organic reactions [7]. Asymmetric Henry and Michael reaction [8], synthesis of organic carbonates [9], aldol condensation [10], as a base catalyst for Claisen–Schmidt condensation reaction [11], an efficient catalyst for the synthesis of formamides [12], etc., are some of well-studied reactions catalyzed by MgO nanoparticles.

Gem-dichloroaziridines are valuable precursors for the preparation of pharmacologically active compounds such as indolinones [13], analogs of natural alkaloids such as isoquinolinones [14] and isoquinolines [15] and nitrogen-containing building blocks such as amidines [16] and aziridinones [17]. As a result, several methods for synthesis of *gem*-dichloroaziridines have been reported. The preparation has been accomplished by the addition of dichlorocarbene generated from chloroform, hexachloroacetone or ethyl trichloroacetate with the appropriate bases to imines [18–26]. Among these, dichlorocarbene, which is generated from chloroform under phase transfer catalyzed conditions, is most frequently used for the synthesis of *gem*-dichloroaziridines because the yields are acceptable. Also effective reaction between sodium hydroxide and chloroform to produce dichlorocarbene normally requires the use of phase transfer catalyst. Recently, Komatsu and co-workers [27] have been reported novel synthesis of *gem*-dichloroaziridines from imines via the KF/Al_2O_3 generation of dichlorocarbene from chloroform. Thus, herein we wish to report on the use of nanocrystallines MgO as an efficient base for preparation of *gem*-dichloroaziridine (**2**) from imine (**1**) and chloroform under thermal conditions.

Electronic supplementary material The online version of this article (doi:10.1007/s13738-012-0137-9) contains supplementary material, which is available to authorized users.

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Experimental

Chemicals and apparatus

All the materials were of commercial reagent grade. All the Schiff bases have been prepared according with previously reported procedure [28, 29].

IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FTIR spectrophotometer. ^{1}H and ^{13}C NMR were recorded in DMSO/$CDCl_3$ solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as internal reference. Mass spectra were recorded on a Finnigan MAT 44S by electron ionization (EI) mode with an ionization voltage of 70 eV. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer. Melting points obtained with a Yanagimoto micro melting point apparatus are uncorrected. The N_2 adsorption/desorption analysis (BET) was performed at −196 °C using an automated gas adsorption analyzer (Tristar 3000, Micromeritics). XRD analysis was performed with an X-ray diffractometer (PAnalytical X'Pert-Pro) using a Cu–Ka monochromatic radiation source and a Ni filter. Transmission electron microscopy (TEM) was performed with a Jeol JEM-2100UHR, operated at 200 kV. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

Preparation of nanocrystalline MgO

Nanocrystalline MgO was prepared by means of a procedure reported elsewhere [30]. In short, poly(vinyl alcohol) (PVA, MW70,000) was dissolved in water at 90 °C under vigorous stirring to form a transparent solution. $Mg(NO_3)_2{\cdot}6H_2O$ was dissolved in water containing PVA. The metal ion-to-PVA monomer unit molar ratio (M/PVA) was chosen as 1:3. Aqueous ammonia (25 % w/w) was added drop wise at room temperature to the resulting viscous liquid mixture, with rapid stirring, to achieve careful pH adjustment to 10.5. After precipitation, the slurry was stirred for another 30 min and then heated under reflux at 80 °C for 20 h under continuous stirring. The mixture was cooled at room temperature, filtered, and washed with hot deionized water for effective removal of the PVA. The final product was dried at 80 °C for 24 h and calcined at 700 °C.

Typical procedure for the synthesis of 1,3-bis (4-chlorophenyl)-2,2-dichloroaziridine

Measured quantities of nanocrystalline MgO (0.02 mol, 0.8 g) and *N*-[1-(4-chlorophenyl)-methylidene]-*N*-[4-chlorophenyl] amine (0.01 mol, 3.3 g) were poured into mortar and mix thoroughly together. The reaction mixture was introduced to a 50 mL flask and mechanically stirred. Then, 8 mL chloroform was added and uniformly was heated at 60 °C. The progress of the reaction was monitored by TLC. After the completion of the reaction, reaction mixture was washed with dichloromethane (3 × 10 mL). The organic solvent was stripped in a vacuum evaporator. The white solid, 1,3-bis(4-chlorophenyl)-2,2-dichloroaziridine, was obtained in 98 % yield, m.p. 139–141 °C. All of the diarylaziridine products were identified by physical and spectroscopic data as following:

1,3-Diphenyl-2,2-dichloroaziridine (**2a**): pale yellow solid; m.p. 100–102 °C (m.p. 98–99 °C) [20, 21, 25, 26].

1-(4-Bromophenyl)-2,2-dichloro-3-phenylaziridine (**2b**): white solid; m.p. 143–145 °C; IR (KBr): ν (cm^{-1}) 3,100, 2,914, 1,600, 1,524 (C=C, Ar); ^{1}H NMR (DMSO): δ 4.40 (s, 1H, HCN), 7.107.80 (m, 9H, Ar); ^{13}C NMR (DMSO): δ 53.4, 77.1, 120.9, 121.3, 127.8, 128.1, 130.1, 131.2, 135.2, 142.1; MS: *m/z* 348 (M^{+6} + 6, 10), 346 (M^{+4} + 4, 25), 344 (M^{+2} + 2, 45), 342 (M^{+}, 30), 309 (85), 307 (100), 233 (65), 230 (45), 152 (80), 77 (85); Anal. Calcd for $C_{14}H_{10}BrCl_2N$: C, 49.02; H, 2.94; N, 4.08 %. Found: C, 49.15; H, 2.95; N, 4.12 %.

1-(4-Chlorophenyl)-2,2-dichloro-3-phenylaziridine (**2c**): pale yellow solid; m.p. 72–74 °C (m.p. 71–72 °C) [22].

1,3-Bis(4-chlorophenyl)-2,2-dichloroaziridine (**2d**): white solid; m.p. 139–141 °C; IR (KBr): ν (cm^{-1}) 3,085, 2,910, 1,600, 1,504 (C=C, Ar); ^{1}H NMR (DMSO): δ 4.34 (s, 1H, HCN), 7.137.55 (m, 8 H, Ar); ^{13}C NMR (DMSO): δ 53.2, 75.9, 122.3, 128.9, 129.3, 129.7, 130.1, 132.2, 134.1, 143.6; MS: *m/z* 341 (M^{+8} + 8, 4), 339 (M^{+6} + 6, <3), 337 (M^{+4} + 4, 4), 335 (M^{+2} + 2, 10), 333 (M^{+}, 20), 298 (70), 296 (65), 174 (60), 172 (95), 161 (80), 159 (100), 89 (50), 77 (55); Anal. Calcd for $C_{14}H_9Cl_4N$: C, 50.49; H, 2.72; N, 4.20 %. Found: C, 50.48; H, 2.74; N, 4.21 %.

1-(4-Bromophenyl)-2,2-dichloro-3-(4-chlorophenyl)aziridine (**2e**): white solid; m.p. 134–136 °C; IR (KBr): ν (cm^{-1}) 3,100, 2,920, 1,598, 1,509 (C=C, Ar); ^{1}H NMR (DMSO): δ 4.33 (s, 1H, HCN), 7.077.59 (m, 8H, Ar); ^{13}C NMR (DMSO): δ 53.1, 75.9, 122.7, 128.9, 129.7, 130.1, 132.2, 132.6, 134.1, 144.1; MS: *m/z* 384.5 (M^{+8} + 8, 6), 382.5 (M^{+6} + 6, <2), 380.5 (M^{+4} + 4, 10), 378.5 (M^{+2} + 2, 35), 376.5 (M^{+}, 25), 341.5 (75), 339.5 (67), 217.5 (89), 205.5 (78), 204.5 (100), 202.5 (90), 89 (60); Anal. Calcd for $C_{14}H_9BrC_{13}N$: C, 44.54; H, 2.40; N, 3.71 %. Found: C, 44.64; H, 2.42; N, 3.73 %.

1-(4-Bromophenyl)-2,2-dichloro-3-(4-nitrophenyl)aziridine (**2f**): white solid; m.p. 141–143 °C; IR (KBr): ν (cm^{-1}) 3,080, 2,924, 1,600, 1,522 (C=C, Ar); ^{1}H NMR ($CDCl_3$): δ 4.09 (s, 1H, HCN), 7.228.55 (m, 8H, Ar); ^{13}C NMR ($CDCl_3$): δ 53.9, 74.5, 121.2, 121.5, 123.7, 128.9, 132.3, 139.6, 143.3, 148.4; MS: *m/z* 392 (M^{+6} + 6, 5), 390

(M^{+4} + 4, 8), 388 (M^{+2} + 2, 18), 386 (M^{+}, 10), 353 (100), 351 (89), 307 (94), 305 (85), 153 (90), 77 (60); Anal. Calcd for $C_{14}H_9BrCl_2N_2O_2$: C, 43.33; H, 2.34; N, 7.22 %. Found: C, 43.43; H, 2.35; N, 7.24 %.

2,2-Dichloro-1-(4-methylphenyl)-3-(4-nitrophenyl)aziridine (**2g**): yellow solid; m.p. 140–142 °C; IR (KBr): ν (cm^{-1}) 3,090, 2,918, 1,589, 1,490 (C=C, Ar); ^{1}HNMR ($CDCl_3$): δ 2.36 (s, 3H, CH_3), 3.89 (s, 1H, HCN), 6.598.31 (m, 8H, Ar); ^{13}C NMR ($CDCl_3$): δ 21.2, 53.5, 75.2, 119.6, 123.7, 128.9, 129.9, 134.6, 140.3, 141.7, 148.3; MS: *m/z* 326 (M^{+4} + 4, 20), 324 (M^{+2} + 2, 29), 322 (M^{+}, 40), 289 (90), 287 (100), 243 (60), 241 (80), 154 (70), 152 (82), 91 (92); Anal. Calcd for $C_{15}H_{12}Cl_2N_2O_2$: C, 55.74; H, 3.74; N, 8.67 %. Found: C, 55.75; H, 3.74; N, 8.67 %.

1-(4-Bromophenyl)-2,2-dichloro-3-(4-methylphenyl)aziridine (**2h**): yellow solid; m.p. 146–148 °C; IR (KBr): ν (cm^{-1}) 3,100, 2,898, 1,600, 1,500 (C=C, Ar); ^{1}H NMR ($CDCl_3$): δ 2.31 (s, 3H, CH_3), 3.75 (s, 1H, HCN), 7.047.48 (m, 8H, Ar); ^{13}C NMR ($CDCl_3$): δ 20.1, 55.1, 74.1, 117.9, 121.9, 128.4, 129.6, 132.1, 134.1, 138.2, 141.1; MS: *m/z* 362 (M^{+6} + 6, <2), 360 (M^{+4} + 4, 10), 358 (M^{+2} + 2, 27), 356 (M^{+}, 15), 323 (100), 321 (89), 234 (84), 232 (70), 91 (97); Anal. Calcd for $C_{15}H_{12}BrCl_2N$: C, 50.46; H, 3.39; N, 3.92 %. Found: C, 50.59; H, 3.39; N, 3.94 %.

2,2-Dichloro-3-(4-chlorophenyl)-1-(4-methylphenyl)aziridine (**2i**): white solid, m.p. 128–130 °C; IR (KBr): ν (cm^{-1}) 3,090, 2,920, 1,600, 1,508 (C=C, Ar); ^{1}H NMR ($CDCl_3$): δ 2.35 (s, 3H, CH_3), 3.65 (s, 1H, HCN), 6.947.47 (m, 8H, Ar); ^{13}C NMR ($CDCl_3$): δ 22.1, 54.2, 76.1, 118.1, 121.5, 128.9, 129.6, 132.1, 134.1, 138.1, 144.1; MS: m/z 318 (M^{+6} + 6, <2), 316 (M^{+4} + 4, 7), 314 (M^{+2} + 2, 12), 312 (M^{+}, 15), 279 (85), 277 (95), 242 (70), 154 (90), 152 (100), 91 (95); Anal. Calcd for $C_{15}H_{12}Cl_3N$: C, 57.62; H, 3.87; N, 4.48 %. Found: C, 57.64; H, 3.88; N, 4.49 %.

Results and discussion

Detailed studies of the reaction of *N*-phenyl-*N*-(1-phenylmethylidene)amine (**1a**) with chloroform led to 1,3-diphenyl-2,2-dichloroaziridine (**2a**). It was shown that this addition reaction was influenced to a considerable extent by the base as shown in Scheme 1 and Table 1.

As can be seen in Table 1, the reaction of **1a** (0.035 mol) with chloroform (8 mL) in the presence of Na_2CO_3 or K_2CO_3 (0.3 g) as a base under thermal conditions did not proceed at all (Table 1, entries 1 and 2), while nanocrystalline MgO displays a substantially activity for this reaction (Scheme 1) than commercial MgO under the same condition (Table 1, entry 8 vs. 7).

Because of the O^{-2}, sites of MgO are influence to trigger the reaction; in this regard, the nanocrystalline MgO should have more reactive sites than commercial MgO (irregular morphology with a surface area of 27 m^2 g^{-1}) and consequently higher basic activity. Also nanocrystalline MgO has more edges and corners which can lead to higher performance of this basic catalyst. Also, presentation of Mg^{+2} as a Lewis acid can be activated imine bond in Schiff base. In addition to the above mentioned and with attention to special structure of nanocrystalline MgO, it seems that the organic species should be able to diffuse faster on the surface of this solid base catalyst rather than commercial MgO.

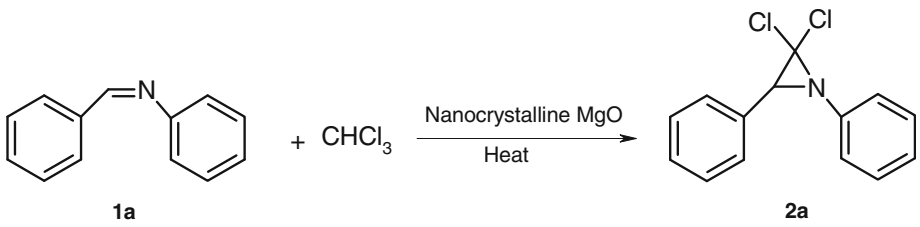
The crystallite sizes determined by XRD were between 12.8 and 17.5 nm (determined by use of the Scherrer equation), indicative of the nanocrystalline structure of the prepared MgO. In addition, the surface area was approximately 116 m^2 g^{-1}. The pore volume and pore size were also calculated from the N_2 adsorption result; the pore size was approximately 21.1 nm and the pore volume approximately 0.69 cm^3 g^{-1}. The theoretical particle size was also calculated from surface area, assuming spherical particles, from the equation:

$$D_{\mathrm{BET}} = \left(\frac{6,000}{\rho \times S}\right) \quad (1)$$

where D_{BET} is the equivalent particle diameter in nanometers, ρ is the density of the material in g cm^{-3}, and S is the specific surface area in m^2 g^{-1}. The particle size calculated from Eq. 1 was 14.4 nm, which confirmed the nanostructure of the MgO sample [30]. The TEM image of MgO is shown in Fig. 1. As can be seen, the sample has a nanocrystalline structure with a plate-like shape.

Subsequent efforts were focused on optimizing conditions to form the *gem*-dichloroaziridine using different amounts of nanocrystalline magnesium oxide and various temperatures (Table 2).

Scheme 1 Preparation of 1,3-diphenyl-2,2-dichloroaziridine from Schiff base (**1a**)



1a **2a**

Table 1 Survey of different bases effect in the reaction of schiff base with chloroform under thermal conditions at 40 °C

Entry	Base	Time (min)	Yield (%)
1	Na_2CO_3	25	–
2	K_2CO_3	25	–
3	ZnO	25	<10
4	CuO	25	15
5	BaO	25	15
6	CaO	25	25
7	MgO	25	35
8	Nanocrystalline MgO	25	75

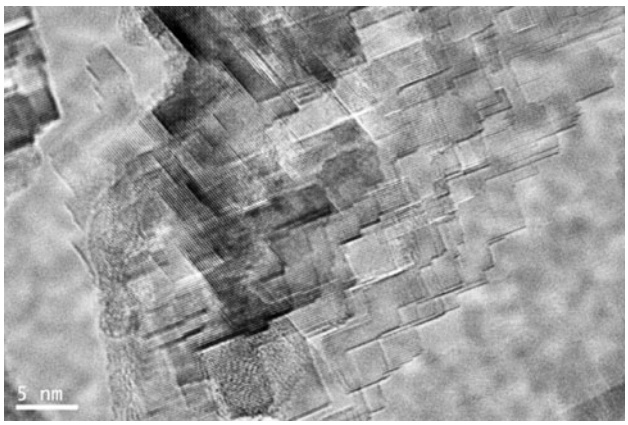


Fig. 1 TEM image of nanocrystalline MgO

Table 2 Optimizing amount of nanocrystalline MgO and reaction temperature conditions on the formation of **2a**

Entry	Amount of nanocrystalline MgO (g)	Time (min)	Temperature (°C)	Yield (%)
1	0.3	25	40	75
2	0.4	25	50	75
3	0.5	25	60	80
4	0.6	25	60	85
5	0.7	25	60	88
6	0.8	25	60	95
7	0.9	25	60	95
8	1.0	25	r. t	65

As indicated in this Table, the reaction showed an improvement in yield using 0.8 g nanocrystalline MgO as a solid base catalyst and the reaction temperature at 60 °C (entry 6 in Table 2). Also, as can be seen in this Table, by heating reaction mixture, the yield of desired product was increased. It seems that α-elimination reaction on $CHCl_3$ in the presence of nanocrystalline MgO under thermal conditions was occurred faster than the reaction at room temperature conditions. Thus, addition reaction of the generated CCl_2 to imine bond occurred in excellent yields.

With attention to the importance of *gem*-dichloroaziridine compounds, in continuation of our research, several different Schiff bases were selected and the reaction of them with nanocrystalline MgO (0.8 g) and $CHCl_3$ have been surveyed under thermal conditions (Scheme 2).

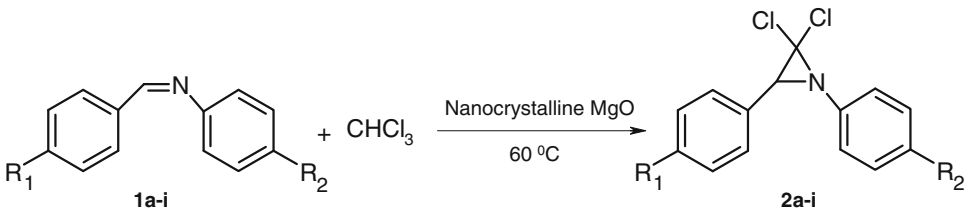
The corresponding results were summarized in Table 3. As can be seen in Table 3, all of the *gem*-dichloroaziridine products were obtained as crystalline solids in excellent yields and short reaction times.

The synthesis of *gem*-dichloroaziridines in the presence of nanocrystalline MgO and $CHCl_3$ was compared with previously reported methods. In this method, the used nanocrystalline MgO:Schiff base mole ratio was 2:1 and the desired product **2a** was obtained in excellent yield (95 %) after short reaction time (25 min) (Table 3, entry 1), while, in previously reported methods, the used $NaOCH_3$:Schiff base mole ratio was 4:1 and corresponding product was obtained in 55 and 61 % yields after several hours (Table 3, entries 2 and 5) [20, 21]. On the other hand, synthesis of this compound **2a** was reported in the presence of TEBAC as a PTC in which used NaOH:Schiff base mole ratio was 25:1 and the desired product was obtained in 74 and 88 % yields [25, 26]. Also preparation of product **2c** using this method was carried out in excellent yield and very short reaction time that is comparable with the reported method (Table 3, entry 7 vs. 8) [22].

It seems that this method for preparation of dichloroaziridines has some advantages such as novelty, high efficiency, appropriateness, being mild and useful in compare to previously reported methods (Table 3, entries 2–5 and 8).

With attention to the importance of *gem*-dichloroaziridine compounds, in continuation of our research, several

Scheme 2 Synthesis of *gem*-dichloroaziridines in the presence of nanocrystalline MgO



R_1 = H, CH_3, NO_2, Cl

R_2 = H, CH_3, Cl, Br

Table 3 Synthesis of 1,3-diaryl-2,2-dichloroaziridine in the presence of nanocrystalline MgO under thermal condition at 60 °C

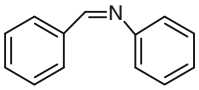
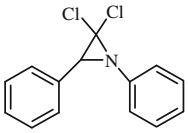
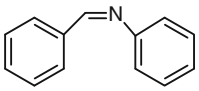
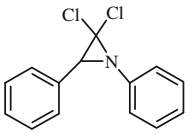
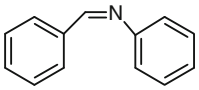
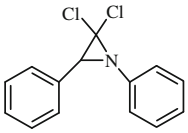
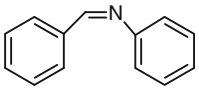
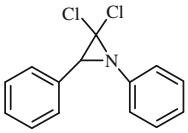
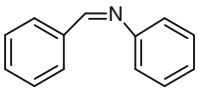
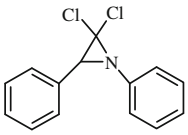
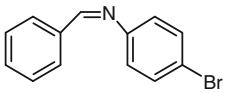
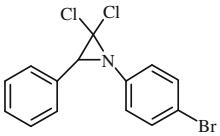
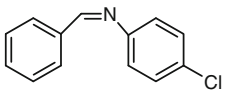
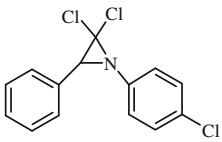
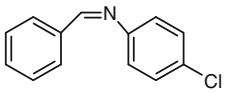
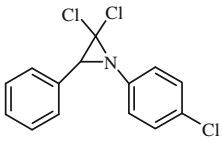
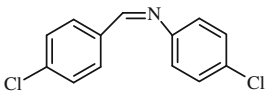
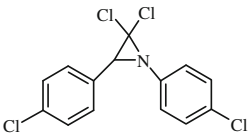
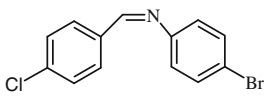
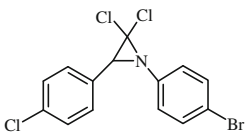
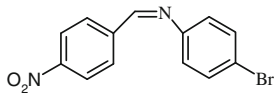
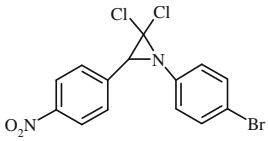
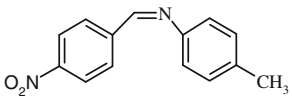
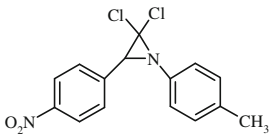
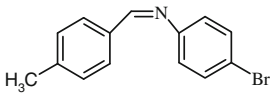
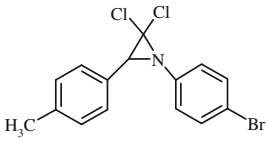
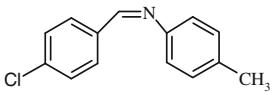
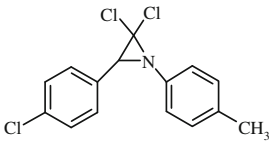
Entry	Substrate	Product	M.p. (°C)	Time (min)	Yield (%)[a]
1			100–102	25	95
2			98–99	360	61[b]
3			98–99	40	74[c]
4			99	–	88[d]
5			98–99	400	55[e]
6			143–145	20	94
7			72–74	20	95
8			71–72	960	68[f]
9			139–141	15	98
10			134–136	15	98

Table 3 continued

Entry	Substrate	Product	M.p. (°C)	Time (min)	Yield (%)[a]
11			141–143	20	93
12			140–142	15	92
13			146–148	15	96
14			128–130	15	97

[a] Isolated yields based on Schiff base

[b] By hexachloroacetone and sodium methoxide, Ref. [21]

[c] By sodium hydroxide (50 %), chloroform in the presence of PTC, Ref. [25]

[d] By sodium hydroxide (50 %), chloroform in the presence of PTC, Ref. [26]

[e] By sodium methoxide and chloroform, Ref. [20]

[f] By potassium *t*-butoxide and chloroform, Ref. [22]

different Schiff bases were selected and the reaction of them with nanocrystalline MgO (0.8 g) and $CHCl_3$ has been surveyed under thermal conditions. The results were summarized in Table 3.

The structure of products has been confirmed by physical and spectroscopic data such as IR, ^{1}H NMR, ^{13}C NMR, mass spectroscopy and C, H, N analyses. In the IR spectra, the stretching frequency of aromatic C=C is formed in the region between ν 1,490–1,600 cm^{-1}. The stretching vibration of C–H in the alkyl groups was appeared at region between ν 2,898–2,930 cm^{-1}. In the ^{1}H NMR spectra, one proton of CH–N has chemical shift in δ 3.65–4.40.

The signals around δ 6.59–8.55 are assigned by protons of CH=CH of aromatic rings. In the ^{13}C NMR spectra, one carbon of C–N has chemical shift in δ 52.1–55.1 and the signal around δ 74.1–77.1 is assigned by one carbon of CCl_2 of aziridine ring.

Conclusions

We have reported efficient and novel method for synthesis of *gem*-dichloroaziridines from Schiff base using nanocrystalline MgO as a base and $CHCl_3$. The corresponding products have been obtained in excellent yields, high purity and short reaction times. The obtained diarylaziridines have been characterized by physical and spectroscopic data such as IR, ^{1}H NMR, ^{13}C NMR, mass spectroscopy and C, H, N analyses.

Acknowledgments The authors are grateful to University of Kashan for supporting this work by Grant No. 159148/3.

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