



Sonocatalyzed facile and mild one pot synthesis of *gem*-dichloroaziridine derivatives under alkaline conditions

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ABSTRACT

In this research, rapid and efficient preparation of 2,2-dichloro-1,3-diarylaziridines through the reaction of Schiff base compounds with dichlorocarbene yielded in situ from chloroform and sodium hydroxide without any phase transfer catalyst under ultrasonic irradiation is described. The advantages of this reaction are very short reaction times, excellent product yields, simplicity of the method and high purity of products.

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1. Introduction

Aziridines, which are extremely important synthetic building blocks, are nitrogen equivalents of epoxides, and can be similarly opened in a stereo controlled manner with various nucleophiles, providing access to a wide range of important nitrogen-containing products [1]. These compounds are among the most fascinating intermediates in organic synthesis, acting as precursors of many complex molecules due to the strain incorporated in their skeletons [2]. Since the first synthesis of an aziridine reported by Gabriel in 1888 [3], the synthetic scope of aziridine chemistry has blossomed in recent years. Thus, obtaining aziridines, has become of great importance in organic chemistry. In particular, the antitumor and antibiotic properties of a great number of aziridine-containing compounds are of high significance among other biological properties, which make them attractive synthetic targets in their own right [4]. 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 ethyltrichloroacetate with the appropriate base under phase transfer catalyst, to imines [5–10].

The use of ultrasonic waves in organic synthesis has been boosted in recent years [11–21]. Ultrasound is known to accelerate diverse types of organic reactions and it is established as an important technique in organic synthesis [17,19,22]. Sonication also increases the reaction rate and avoids the use of high reaction

temperatures [19]. A number of organic reactions have been revisited by means of ultrasonic waves [17,19,22]. Also, some literatures on ultrasonically prepared dichlorocarbene have been reported [23–27]. Recently, syntheses of fluoro aziridines in the presence of phase transfer catalyst under ultrasonic irradiation have been reported [28]. The advantages of ultrasound procedures, such as, good yields, short reaction times and mild reaction conditions, are well documented [29].

In conjunction with ongoing work in our laboratory on the preparation of Schiff base derivatives [30–33], here we decided to report the synthesis of various dichloroaziridines through the reaction of various Schiff base compounds and chloroform without any phase transfer catalyst under ultrasonic irradiation.

2. Experimental section

2.1. Materials

All the materials were of commercial reagent grade. All the Schiff bases have been prepared according to the previously reported procedures [30,31].

2.2. Apparatus

IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FTIR spectrophotometer. ¹H NMR and ¹³C NMR were recorded in DMSO/CDCl₃ solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as internal reference. Mass spectra were recorded on a Finnigan MAT

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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. The BANDELIN ultrasonic HD 3200 with probe model KE 76, 6 mm diameter, was used to produce ultrasonic irradiation and homogenizing the reaction mixture. Piezoelectric crystal of this kind of probe normally works in the range of 700 kHz, but using through some proper clamps the output frequency of piezoelectric crystal have controlled and reduced to 20 kHz. Therefore, the induced frequency of probe to the reaction mixture is equal to 20 kHz. By changing the power of Tip the cavitations rate is displaced. Meaning the Tip frequency under all amount of power is constant. Melting points obtained with a Yanagimoto micro melting point apparatus are uncorrected. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

2.3. The power measurement by calorimetric method

We assessed the cavitation energy applied by ultrasonication calorimetrically with water. The piezoelectric transducer was connected to the frequency generator, HD-3200 (with frequency; 20 kHz). The probe (KE-76) was dipped in a jacketed cylindrical vessel. For calorimetric measurement, the jacket was empty and connected to vacuum to minimize heat losses. In this method, by measuring the rate of temperature increase due to the conversion of ultrasound energy into heat and calculating $P_{acoustic}$ according to: $P = mc\Delta T/t$, where m is the mass of water (g), c is the specific heat capacity of water ($4.18 \text{ Jg}^{-1} \text{ K}^{-1}$), ΔT is the difference in temperature (K) and t is the sonication time(s).

2.4. Typical procedure for the synthesis of 2,2-dichloro-1, 3-diphenylaziridine

Measured quantities of NaOH (0.075 mol, 3 g) were dissolved in 30 ml of water and the obtained solution was introduced to a 100 ml flask. The ultrasonic probe was immersed directly in the reactor. Then, Schiff base (organic reactant; 0.028 mol, 8.2 g) dissolved in chloroform (0.07 mol, 8.3 ml) was gradually added drop wise to the NaOH solution under ultrasonic irradiation with power 67 W. The progress of the reaction was monitored by TLC. After the completion of the reaction in 15 min, the solution was separated and the portion of aqueous solution was extracted by diethylether. Magnesium sulfate was also added to adsorb the residual water. The organic solvent and other residues were stripped in a vacuum evaporator. The pale yellow solid, 2,2-dichloro-1, 3-diphenylaziridine, was obtained in 96% yield, m.p. = 100–102 °C (reported [5,8–10], m.p. = 98–99 °C). All of the diarylaziridine products were identified by physical and spectroscopic data as following;

2,2-dichloro-1, 3-diphenylaziridine (2a); pale yellow solid; m.p. = 100–102 °C (m.p. = 98–99 °C) [5,8–10].

2,2-dichloro-1-(4-bromophenyl)-3-phenylaziridine (2b); white solid; m.p. = 143–145 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3100, 2914, 1600, 1524 (C=C, Ar); $^1\text{H NMR}$ (DMSO)/ δ ppm: 4.34 (s, 1 H, HCN), 7.14 (d, 2 H, Ar), 7.45 (d, 2 H, Ar), 7.50–7.55 (m, 5 H, Ar); $^{13}\text{C NMR}$ (CDCl₃) / δ ppm: 50.0, 71.0, 119.4, 122.7, 128.9, 129.0, 131.9, 132.3, 136.2, 151; MS: m/z: 347 ($\text{M}^+ + 6$, 8), 345 ($\text{M}^+ + 4$, 20), 343 ($\text{M}^+ + 2$, 45), 341 (M^+ , 27), 308 (80), 306 (100), 229 (75), 227 (50), 77 (85); Anal. Calcd. For C₁₄H₁₀NBrCl₂: C, 49.12; H, 2.92; N, 4.11. Found: C, 49.15; H, 2.95; N, 4.12.

2,2-dichloro-1-(4-chlorophenyl)-3-phenylaziridine (2c); pale yellow solid; m.p. = 72–74 °C (m.p. = 71–72 °C) [7].

2,2-dichloro-1, 3-bis(4-chlorophenyl)aziridine (2d); white solid; m.p. = 139–141 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3085, 2910, 1600, 1504 (C=C, Ar); $^1\text{H NMR}$ (400 MHz, DMSO)/ δ ppm: 4.34 (s, 1 H, HCN), 7.14 (d, 2 H, Ar), 7.45 (d, 2 H, Ar), 7.50–7.58 (m, 4 H, Ar); ^{13}C

NMR/ (100 MHz, DMSO)/ δ ppm: 53.15, 75.9, 122.3, 128.9, 129.3, 129.7, 130.1, 132.2, 134.1, 143.6; MS: m/z: 341 ($\text{M}^+ + 8$, 4), 337 ($\text{M}^+ + 6$, <3), 335 ($\text{M}^+ + 4$, 10), 333 ($\text{M}^+ + 2$, 25), 331 (M^+ , 15), 298 (70), 296 (65), 174 (60), 172 (98), 161 (80), 159 (100), 77 (55); Anal. Calcd. For C₁₄H₉NCl₄: C, 50.45; H, 2.70; N, 4.20. Found: C, 50.48; H, 2.74; N, 4.21.

2,2-dichloro-1-(4-bromophenyl), 3-(4-chlorophenyl) aziridine (2e); white solid; m.p. = 134–136 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3100, 2920, 1598, 1509 (C=C, Ar); $^1\text{H NMR}$ (400 MHz, DMSO)/ δ ppm: 4.34 (s, 1 H, HCN), 7.08 (d, 2 H, Ar), 7.50–7.56 (m, 4 H, Ar), 7.58 (d, 2 H, Ar); $^{13}\text{C NMR}$ (100 MHz, DMSO)/ δ ppm: 53.0, 76.9, 122.3, 129.1, 129.3, 130.1, 131.8, 134.5, 137.6, 150.1; MS: m/z: 381 ($\text{M}^+ + 6$, 6), 379 ($\text{M}^+ + 4$, 25), 377 ($\text{M}^+ + 2$, 34), 375 (M^+ , 14), 342 (60), 340 (46), 218 (60), 216 (94), 205 (82), 203 (100); Anal. Calcd. For C₁₄H₉NBrCl₃: C, 44.62; H, 2.39; N, 3.72. Found: C, 44.64; H, 2.42; N, 3.73.

2,2-dichloro-1-(4-bromophenyl), 3-(4-nitrophenyl) aziridine (2f); white solid; m.p. = 141–143 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3080, 2924, 1600, 1522 (C=C, Ar); $^1\text{H NMR}$ (400 MHz, CDCl₃)/ δ ppm: 3.79 (s, 1 H, HCN), 6.95 (d, 2 H, Ar), 7.50 (d, 2 H, Ar), 7.71 (d, 2 H, Ar), 8.31 (d, 2 H, Ar); $^{13}\text{C NMR}$ (100 MHz, DMSO)/ δ ppm: 53.2, 75.9, 122.3, 128.9, 129.2, 129.7, 130.2, 132.2, 134.1, 143.6; MS: m/z: 392 ($\text{M}^+ + 6$, 7), 390 ($\text{M}^+ + 4$, 20), 388 ($\text{M}^+ + 2$, 50), 386 (M^+ , 38), 353 (78), 351 (100), 307 (65), 305 (80), 218 (80), 216 (95), 77 (80); Anal. Calcd. For C₁₄H₉N₂O₂BrCl₂: C, 43.41; H, 2.33; N, 7.24. Found: C, 43.43; H, 2.35; N, 7.24.

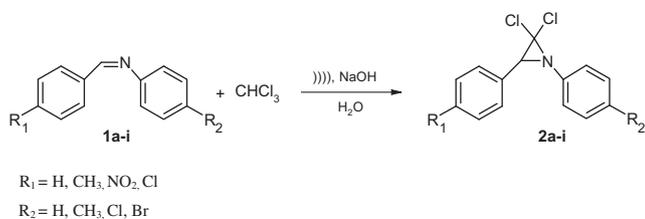
2,2-dichloro-1-(4-methylphenyl), 3-(4-nitrophenyl) aziridine (2g); yellow solid; m.p. = 140–142 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3090, 2918, 1589, 1490 (C=C, Ar); $^1\text{H NMR}$ (400 MHz, DMSO)/ δ ppm: 2.29 (s, 3 H, CH₃), 4.45 (s, 1 H, HCN), 7.02 (d, 2 H, Ar), 7.21 (d, 2 H, Ar), 7.80 (d, 2 H, Ar), 8.31 (d, 2 H, Ar); $^{13}\text{C NMR}$ (100 MHz, CDCl₃)/ δ ppm: 20.9, 53.5, 75.1, 119.6123.7, 128.9, 134.6, 140.3, 141.7, 148.3; MS: m/z: 326 ($\text{M}^+ + 4$, 6), 324 ($\text{M}^+ + 2$, 29), 322 (M^+ , 40), 289 (70), 287 (100), 243 (60), 241 (80), 154 (70), 152 (82), 91 (92); Anal. Calcd. For C₁₅H₁₂N₂O₂Cl₂: C, 55.73; H, 3.72; N, 8.67. Found: C, 55.75; H, 3.74; N, 8.67.

2,2-dichloro-1-(4-bromophenyl), 3-(4-methylphenyl) aziridine (2h); yellow solid; m.p. = 146–148 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3100, 2898, 1600, 1500 (C=C, Ar); $^1\text{H NMR}$ (400 MHz, CDCl₃)/ δ ppm: 2.38 (s, 3 H, CH₃), 3.41 (s, 1 H, HCN), 7.15 (d, 2 H, Ar), 7.21 (d, 2 H, Ar), 7.45 (d, 2 H, Ar), 7.84 (d, 2 H, Ar); $^{13}\text{C NMR}$ (100 MHz, CDCl₃)/ δ ppm: 20.9, 51.0, 72.9, 120.1, 129.8, 130.1, 135.0, 136.7, 137.8, 149.3; MS: m/z: 361 ($\text{M}^+ + 6$, <2), 359 ($\text{M}^+ + 4$, 24), 357 ($\text{M}^+ + 2$, 47), 355 (M^+ , 35), 322 (85), 320 (100), 218 (64), 216 (80), 91 (95); Anal. Calcd. For C₁₅H₁₂NBrCl₂: C, 50.56; H, 3.37; N, 3.93. Found: C, 50.59; H, 3.39; N, 3.94.

2,2-dichloro-1-(4-methylphenyl), 3-(4-chlorophenyl) aziridine (2i); white solid, m.p. = 128–130 °C; IR (KBr)/ $\nu(\text{cm}^{-1})$: 3090, 2920, 1600, 1508 (C=C, Ar); $^1\text{H NMR}$ (400 MHz, CDCl₃)/ δ ppm: 2.36 (s, 3 H, CH₃), 3.65 (s, 1 H, HCN), 6.95 (d, 2 H, Ar), 7.18 (d, 2 H, Ar), 7.36–7.47 (m, 5 H, Ar); $^{13}\text{C NMR}$ (100 MHz, CDCl₃)/ δ ppm: 21.5, 53.1, 73.1, 121.0, 128.5, 129.9, 130.1, 138.1, 138.5, 138.1, 139.0, 149.3; MS: m/z: 317 ($\text{M}^+ + 6$, 10), 315 ($\text{M}^+ + 4$, 20), 313 ($\text{M}^+ + 2$, 47), 311 (M^+ , 50), 283 (96), 281 (100), 154 (80), 152 (84), 91 (98); Anal. Calcd. For C₁₅H₁₂NCl₃: C, 57.60; H, 3.84; N, 4.48. Found: C, 57.64; H, 3.88; N, 4.49.

3. Results and discussion

In this research, the ultrasonic irradiation as a phase transfer catalyzed reaction of dichloro aziridination of Schiff base compounds has been studied. When 0.028 mol of Schiff base compound was reacted with dichlorocarbene intermediate obtained in situ from the reaction of chloroform and base under ultrasonic irradiation, corresponding products, 2,2-dichloro-1, 3-diarylaziri-



Scheme 1. Preparation of 2,2-dichloro-1,3-diarylaziridine compounds from Schiff bases.

Table 1

Enhancement of ultrasonic irradiation (52 W) on the formation of 2,2-dichloro-1-(4-bromophenyl), 3-(4-chlorophenyl) aziridine in the presence of various amount of NaOH.

Entry	Amount of NaOH (%)	Time (min)	Yield ^a (%)
1	5	90	53
2	5.6	70	60
3	6.7	60	64
4	8.3	50	72
5	9.3	45	80
6	10	40	90
7	12	37	90
8	10	840	40 ^b

^a Isolated yields.

^b Isolated yield under silent conditions.

Table 2

Survey the effect of ultrasonic irradiation on the formation of 2,2-dichloro-1-(4-bromophenyl),3-(4-chlorophenyl) aziridine.

Entry	Electric power (W)	Measured power (W)	Time (min)	Yield ^a (%)
1	50	47	10	63
2	55	52	10	70
3	60	56	10	80
4	65	61	10	85
5	68	63	10	90
6	72	67	10	98
7	75	70	10	98

^a Isolated yields under 10% NaOH.

Table 3

Synthesis of *gem*-dichloro-1,3-diarylaziridines and NaOH (10%) under ultrasonic irradiation with 67 W power.

Entry	Substrate	Product	M.P. (°C)	Time (min)		Yield ^a (%)	
				Ultrasound	Silent	Ultrasound	Silent
1			100–102	15	900	95	35
2			98–99	–	360	–	61 ^b
3			98–99	–	40	–	74 ^c

dine compounds were obtained under alkaline conditions (10% NaOH) (Scheme 1).

Firstly, we have carried out this reaction in the presence of various amount of NaOH under ultrasonic irradiation (52 W). The corresponding results are indicated in Table 1.

As can be seen in this Table, the desired product was obtained with high yield using NaOH (10%) under ultrasonic irradiation (52 W) (Table 1, entry 6). In order to determine enhancement ultrasonic irradiation with performance of this reaction under magnetic stirring, the product was obtained in low yield and long reaction times (Table 1, entry 8).

In continuation of this research, the effect of various powers of ultrasonic irradiation has been surveyed. Initially we carried out aziridination of (4-bromo-phenyl)methylene(4-chloro-phenyl)amine as model reaction in order to optimize the best suited reaction conditions. It was observed that the reaction in the presence of NaOH 10% and ultrasonic irradiation with power 67 W was afforded the best result as obtained product with 98% isolated yield during 10 min (Table 2, entry 6).

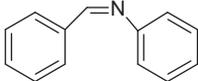
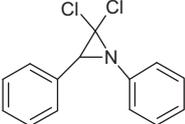
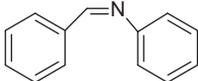
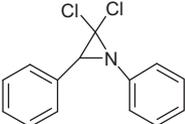
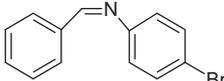
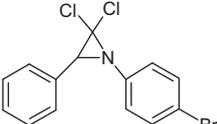
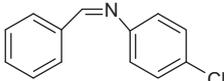
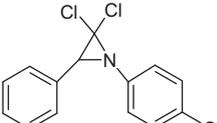
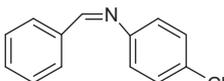
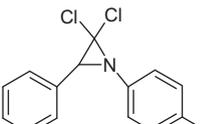
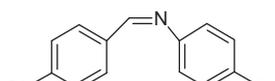
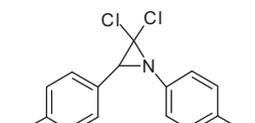
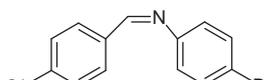
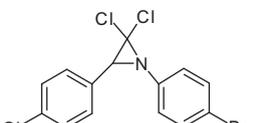
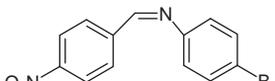
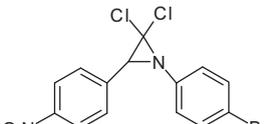
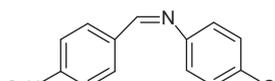
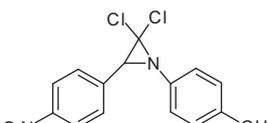
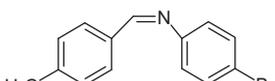
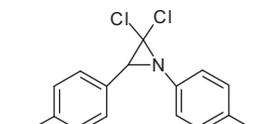
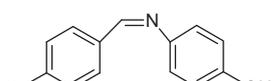
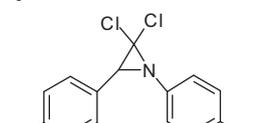
3.1. The effect of ultrasonic irradiation

Sonification of multiphasic systems accelerates the reaction by ensuring a better contact between the different phases. By occurring the reaction under sonication conditions dichlorocarbene intermediate was produced very fast and thus corresponding products were obtained in excellent yields and very short reaction times.

The effects of ultrasonic irradiation observed during organic reactions are due to cavitations. In the case of volatile molecules cavities are believed to act as a microreactor: as the volatile molecules enter the microbubbles and the high temperature and pressure produced during cavitations break their chemical bonds thus reacting with other specie [34–36]. The presence of ultrasonic irradiation in liquid–liquid system, cavitation collapse near the liquid–liquid interface disrupts the interface and impels jets of one liquid into the other forming fine emulsion, and leading to a dramatic increase in the interfacial contact area across which transfer of species can take place. Also, the ultrasonic irradiation increase the produce of :CCl_2 intermediate and cause the enhancement of addition reaction rate.

Then to ascertain the scope and limitation of the present reaction, several Schiff base compounds were reacted with CHCl_3 in

Table 3 (continued)

Entry	Substrate	Product	M.P. (°C)	Time (min)		Yield ^a (%)	
				Ultrasound	Silent	Ultrasound	Silent
4			99	-	-	-	88 ^d
5			98–99	400	Several hours	-	55 ^e
6			143–145	12	970	94	32
7			72–74	12	998	95	36
8			71–72	-	960	-	68 ^f
9			139–141	10	840	98	40
10			134–136	10	840	98	40
11			141–143	13	870	93	30
12			140–142	14	910	92	27
13			146–148	11	845	96	38
14			128–130	10	840	97	40

^a Isolated yields based on Schiff base.

^b By hexachloroacetone and sodium methoxide, Ref. [5].

^c By sodium hydroxide(50%), chloroform in the presence of PTC, Ref. [8].

^d By sodium hydroxide(50%), chloroform in the presence of PTC Ref. [9].

^e By sodium methoxide and chloroform, Ref. [6].

^f By potassium *t*-butoxide and chloroform, Ref. [7].

the presence and optimum amount of NaOH (10%) under ultrasonic irradiation and the desired dichloroaziridine derivatives were prepared. The results are summarized in Table 3. As shown in this Table, the reaction of the various Schiff bases with chloroform and NaOH (10%), were catalyzed by ultrasonic irradiation. The corresponding products were obtained in excellent yields and appropriate reaction times under ultrasonic irradiation. It seems this method for preparation of dichloroaziridines has some advantages such as; highly efficiently, appropriately, mildly and useful in compare to previously reported methods [5–9] (Table 3, entries 2–5 and 7). Among these, dichlorocarbene which is generated from CHCl_3 under PTC conditions is most frequently used because the yields are acceptable. In our report on synthesis of dichloroaziridine derivatives under ultrasonic irradiation, the mol ratio of NaOH and CHCl_3 into the Schiff base was ~ 2.5 , while in other reports were 4 and 10. Also, in our research, NaOH with 10% concentration was applied. The yields of products were higher and the reaction time was shorter than the other previously reported methods [6–9].

The structure of products has been confirmed by physical and spectroscopic data such as; IR, ^1H NMR, ^{13}C NMR, Mass spectroscopy and C. H. N. analyses. In the IR spectra, the stretching fre-

quency of aromatic $\text{C}=\text{C}$ is formed in the region between $\nu = 1490\text{--}1600\text{ cm}^{-1}$. The stretching vibration of $\text{C}-\text{H}$ in the alkyl groups was appeared at region between $\nu = 2898\text{--}2930\text{ cm}^{-1}$. In the ^1H NMR spectra, the disappearance of imine proton signal around the $\delta = 8.42\text{--}8.75$ in Schiff base compounds and followed to appear the signal of $\text{CH}-\text{N}$ around $\delta = 3.41\text{--}4.45$ ppm in *gem*-dichloroaziridines was confirmed the formation of them in this reaction (Fig. 1). The signals around $\delta = 6.95\text{--}8.31$ are assigned by protons of $\text{CH}=\text{CH}$ of aromatic rings. In the ^{13}C NMR spectra, one carbon of $\text{C}-\text{N}$ has chemical shift in $\delta = 52.1\text{--}55.1$ ppm and the signal around $\delta = 71.0\text{--}76.9$ is assigned by one carbon of CCl_2 of aziridine ring (Fig. 2).

4. Conclusion

In this study, we have synthesized *gem*-dichloroaziridine derivatives through the reaction of various Schiff base compounds with chloroform under ultrasonic irradiations as a phase transfer catalyst. The corresponding products have been obtained in excellent yields, high purity and short reaction times. The products have been confirmed by physical and spectroscopic data such as; IR, ^1H NMR, ^{13}C NMR, MS spectroscopy and C. H. N. analyses.

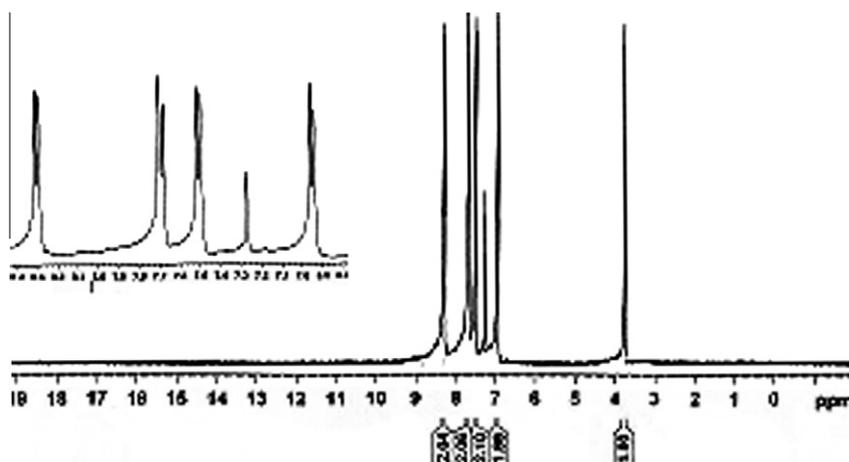


Fig. 1. ^1H NMR spectra of 2,2-dichloro-1-(4-bromophenyl), 3-(4-nitrophenyl)aziridine.

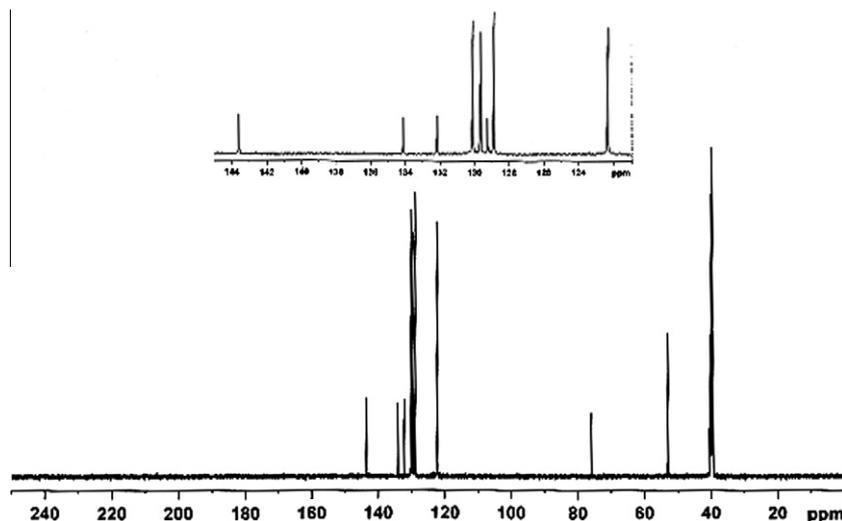


Fig. 2. ^{13}C NMR spectra of 2,2-dichloro-1-(4-bromophenyl), 3-(4-nitrophenyl)aziridine.

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