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Ultrasonic assisted synthesis of *gem*-dichloroaziridine derivatives using Mg/CCl_4 under neutral conditions

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ABSTRACT

A novel and convenient method for synthesis of *gem*-dichloroaziridine derivatives was reported in that was utilized Mg powder with CCl_4 for dichlorocarbene generation under ultrasonic irradiation. In this clean and efficient reaction procedure, the desired products were purely obtained in excellent yields. The other advantages of this method are availability of reagents, very short reaction times, simplicity of the method, easily work up, neutral reaction medium and high purity of products.

1. Introduction

The use of ultrasonic waves is a convenient technique in organic synthesis [1–13]. Sonochemistry is also becoming more and more important for a variety of synthetic organic reactions as an energy source to generate radicals and initiate the electron transfer process. Many metal-involved organic reactions have been accelerated under ultrasound [14,15]. Sonochemical generation of dichlorocarbene has also been reported [16,17]. Recently, syntheses of fluoro aziridines in the presence of phase transfer catalyst under ultrasonic irradiation have been reported [18]. The advantages of ultrasound procedures, such as; good yields, short reaction times and mild reaction conditions are well documented [19]. Aziridines are an important class of three-membered heterocycles containing one nitrogen atom with a rapidly expanding scope of applications in organic synthesis [20]. 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 and can be opened in a stereo controlled manner with various nucleophiles, providing access to a wide range of important nitrogen-containing products [21–25].

Since the first synthesis of an aziridine reported by Gabriel in 1888, the synthetic scope of aziridine chemistry has blossomed in recent years [26–30]. Many synthetic compounds of biological interest contain the aziridine framework in their structure. 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 [31–33]. 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 [34–38].

In continuation of our research onto preparation of dichloroaziridines [39–41], we hope to report a novel procedure for preparation of these compounds using the addition reaction of dichlorocarbene generated through the reaction of Mg powder and CCl_4 under ultrasonic irradiation to Schiff base compounds.

2. Experimental section

2.1. Materials and apparatus

All the materials were of commercial reagent grade. All the Schiff bases have been prepared in laboratory in accordance to the previously reported method in literature [42].

IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FTIR spectrophotometer. ^1H NMR and ^{13}C NMR were recorded in $DMSO/CDCl_3$

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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. The BANDELIN ultrasonic HD 3200 with probe model US 70/T, 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. 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).

2.2. The power measurement by calorimetric method

We assessed the cavitational energy applied by ultrasonication calorimetrically with water. The piezoelectric transducer was connected to the frequency generator, HD-3200 (with frequency; 20 kHz). The probe (US 70 T) 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 Jg^{-1} k^{-1}), ΔT is the difference in temperature (k) and t is the sonication time (s).

2.3. General procedure for the synthesis of 2,2-dichloro-1,3-diphenylaziridines

A mixture of magnesium powder (80 mmol), Schiff base compounds **1a** (40 mmol) and CCl_4 (80 mmol) in anhydrous tetrahydrofuran (8 mL) was placed in flask equipped with ultrasonic prob. The mixture was then treated with ultrasonic irradiation until all magnesium was consumed. To the reaction mixture was then added 10% NH_4Cl solution (30 mL) and the aqueous layer was extracted with diethyl ether (3 × 10 mL). The combined organic layer was dried over anhydrous sodium sulfate. The solvent was evaporated and desired product, *gem*-dichloroaziridine, was obtained in excellent yield. All of the diarylaziridine products were identified by physical and spectroscopic data as following and were consistent in comparison with authentic samples [34,36–38].

2,2-dichloro-1,3-diphenylaziridine (***2a***); pale yellow solid; m.p. = 101–103 °C (m.p. = 98–99 °C) [34,37,38].

2,2-dichloro-1-(4-bromophenyl)-3-phenylaziridine (***2b***); white solid; m.p. = 145–147 °C; IR (KBr)/$\upsilon(cm^{-1})$: 3100, 2914, 1600, 1524 (C=C, Ar); ^{1}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, 4 H, Ar); ^{13}C NMR/(DMSO)/δ ppm: 50.0, 71.0, 119.4, 122.7, 128.9, 129.0, 131.9, 132.3, 136.2, 151; 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 $C_{14}H_{10}NBrCl_2$: 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. = 73–75 °C (m.p. = 71–72 °C) [36].

2,2-dichloro-1,3-bis(4-chlorophenyl)aziridine (***2d***); white solid; m.p. = 141–143 °C; IR (KBr)/$\upsilon(cm^{-1})$: 3085, 2910, 1600, 1504 (C=C, Ar); ^{1}H NMR (DMSO)/δ ppm: 4.34 (s, 1 H, HCN), 7.14 (d, 2 H, Ar), 7.45 (d, 2 H, Ar), 7.50–7.58 (m, 4 H, Ar); ^{13}C NMR/(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 (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 $C_{14}H_9NCl_4$: 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. = 136-138 °C; IR (KBr)/$\upsilon(cm^{-1})$: 3100, 2920, 1598, 1509 (C=C, Ar); ^{1}H NMR (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}C NMR/(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*: 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 $C_{14}H_9NBrCl_3$: 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. = 143–145 °C; IR (KBr)/$\upsilon(cm^{-1})$: 3080, 2924, 1600, 1522 (C=C, Ar); ^{1}H NMR ($CDCl_3$)/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}C NMR/(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 (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 $C_{14}H_9N_2O_2BrCl_2$: 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. = 142–144 °C; IR (KBr)/$\upsilon(cm^{-1})$: 3090, 2918, 1589, 1490 (C=C, Ar); ^{1}H NMR (DMSO)/δ ppm: 2.29 (s, 3 H, CH_3) 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}C NMR/(DMSO)/δ ppm: 20.9, 53.5, 75.1, 119.6, 123.7, 128.9, 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 $C_{15}H_{12}N_2O_2Cl_2$: 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. = 148–150 °C; IR (KBr)/$\upsilon(cm^{-1})$: 3100, 2898, 1600, 1500 (C=C, Ar); ^{1}H NMR ($CDCl_3$)/δ ppm: 2.38 (s, 3 H, CH_3) 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}C NMR/($CDCl_3$)/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*: 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 $C_{15}H_{12}NBrCl_2$: 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. = 130–132 °C; IR (KBr)/$\upsilon(cm^{-1})$; 3090, 2920, 1600, 1508 (C=C, Ar); ^{1}H NMR ($CDCl_3$)/δ ppm: 2.36 (s, 3 H, CH_3) 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}C NMR/($CDCl_3$)/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*: 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 $C_{15}H_{12}NCl_3$: C, 57.60; H, 3.84; N, 4.48, Found: C, 57.64; H, 3.88; N, 4.49.

3. Results and discussion

Here, the ultrasonic irradiation catalyzed reaction of dichloro aziridination of Schiff base compounds under neutral conditions has been studied. In order to enhance the yields of gem-dichloroaziridin derivatives, it is necessary to control the molar ratios of reactants. Firstly, we have carried out this reaction in the presence of various molar ratios of Mg, CCl_4 and Schiff base under ultrasonic irradiation (60 W). The corresponding results are indicated in Table 1.

As can be seen in this Table, the desired product was obtained with good yield using Mg: CCl_4: Schiff base with 2:2:1 molar ratio.

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Table 1
Enhancement of ultrasonic irradiation (60 W) on the formation of 2,2-dichloro-1-(4-methylphenyl)-3-(4-nitrophenyl) aziridine in the presence of various molar ratio of Mg/CCl_4.

Entry	Mg:CCl_4:Schiff base (molar ratio)	Time (min)	Yield[a] (%)
1	1:1:1	30	45
2	1.2:1.2:1	30	55
3	1.4:1.4:1	30	60
4	1.6:1.6:1	30	70
5	1.8:1.8:1	30	75
6	2:2:1	30	80
7	2.2:2.2:1	30	80
8	2:2:1	30	<10[b]

[a] Isolated yields.
[b] Isolated yield under silent conditions.

In order to determine enhancement of ultrasound irradiation with performance of this reaction under silent condition and magnetic stirring, the products were obtained in very low yield (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-nitrophenyl)methylene(4-methylphenyl)amine as model reaction in order to optimize the best suited reaction conditions. It was observed that the reaction in the presence of ultrasonic irradiation with power 75 W was afforded the best result as obtained product with 93% isolated yield during 11 min (Table 2, entry 5).

Table 2
Survey the effect of ultrasonic irradiation on the formation of 2,2-dichloro-1-(4-methylphenyl)-3-(4-nitrophenyl) aziridine.

Entry	Power (W)	Time (min)	Yield[a] (%)
1	60	30	80
2	64	30	85
3	68	20	85
4	72	15	90
5	75	11	93
6	80	11	93

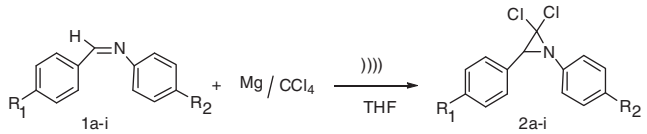
[a] Isolated yields.

3.1. Survey the effect of ultrasonic irradiation

Sonication is able to effect reactions proceeding through radicals, and ultrasound has also provided a distinct alternative to the initiation and enhancement of synthetic reaction involving metals as a reagent. Also, ultrasound irradiation accelerates the reaction by ensuring a better contact between the reactants. By occurring the reaction under sonication conditions dichloro carbene 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.

Then to ascertain the scope and limitation of the present reaction, several Schiff base compounds were reacted with CCl_4 in the presence of Mg powder under ultrasonic irradiation and the desired dichloroaziridine derivatives were prepared (Scheme 1).



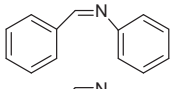
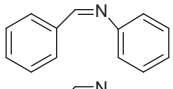
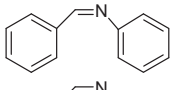
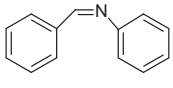
R_1 = H, CH_3, NO_2, Cl

R_2 = H, CH_3, Cl, Br

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

The results are summarized in Table 3. As shown in this Table, the reaction of the various Schiff bases with CCl_4 and Mg powder, 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. The main advantages of this method is that the reaction carried out in a neutral medium, some side reactions caused by the strong base in phase transfer catalyst system can be avoided and highly efficiently, appropriately, mildly and useful in compare to previously reported methods [34,36–38] (Table 3, entries 2, 3, 4, 5 and 7).

Table 3
Synthesis of *gem*-dichloro-1,3-diarylaziridines by using Mg powder/CCl_4 under ultrasonic irradiation with 75 (W) power and neutral conditions.

Entry	Substrate	Product	M.P. (°C)	Time (min)		Yield[a] (%)	
				Silent	Ultrasonic	Silent	Ultrasonic
1		**2a**	100–102	960	12	30	96
2		**2a**	98–99	360	–	61[b]	–
3		**2a**	98–99	40	–	74[c]	–
4		**2a**	99	–	–	88[d]	–

(*continued on next page*)

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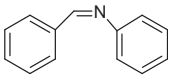
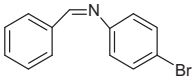
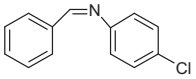
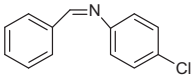
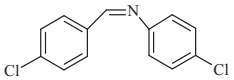
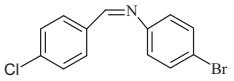
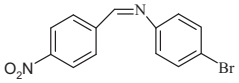
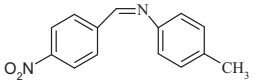
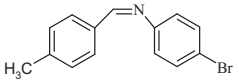
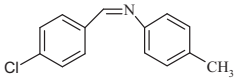
Table 3 (*continued*)

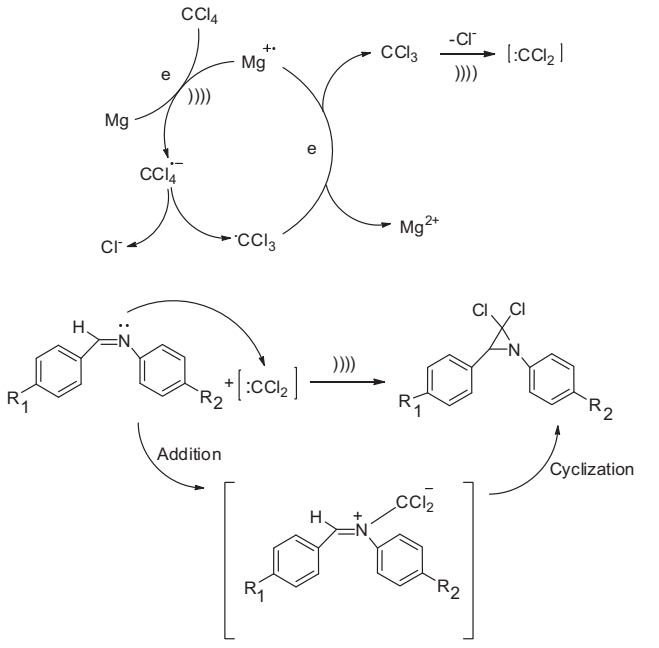
Entry	Substrate	Product	M.P. (°C)	Time (min)		Yield[a] (%)	
				Silent	Ultrasonic	Silent	Ultrasonic
5		**2a**	98–99	400	–	55[e]	–
6		**2b**	143–145	1000	9	26	95
7		**2c**	72–74	1050	9	29	96
8		**2c**	71–72	960	–	68[f]	–
9		**2d**	139–141	900	7	39	98
10		**2e**	134–136	900	7	40	97
11		**2f**	141–143	940	7	30	94
12		**2g**	140–142	955	11	26	93
13		**2h**	146–148	900	8	36	97
14		**2i**	128–130	900	7	39	98

[a] Isolated yields based on Schiff base.
[b] By hexachloroacetone and sodium methoxide, Ref. [34].
[c] By sodium hydroxide(50%), chloroform in the presence of PTC [37].
[d] By sodium hydroxide(50%), chloroform in the presence of PTC [38].
[e] By sodium methoxide and chloroform, Ref. [35].
[f] By potassium *t*-butoxide and chloroform, Ref. [36].

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 magnesium and CCl_4 into the Schiff base was 2, while in other reports, high alkaline medium conditions was applied for dichloroaziridination of Schiff bases. Also, the yields of products were higher and the reaction time was shorter than the other previously reported methods [34,36–38].

3.2. The proposed reaction mechanism

The proposed reaction mechanism for the preparation of dichloroaziridines via formation of carbene intermediate was shown in Scheme 2. The reaction between Mg and CCl_4 may be involving a single electron transfer from magnesium to carbon tetrachloride. Sonication of the reaction through cavitations is able to affect the reaction proceeding through generated radicals from metal as reagent. In the case of volatile molecules, cavities are believed to act as microreactor. By using ultrasonic irradiation, treatment of Mg with CCl_4, the activated surface of Mg can be achieved due to the clean and oxide free of Mg surface. Dichlorocarbene in situ generated was trapped by Schiff base compound and corresponding product was obtained.



Scheme 2. Proposed mechanism for the preparation of 2,2-dichloro-1,3-diarylaziridines.

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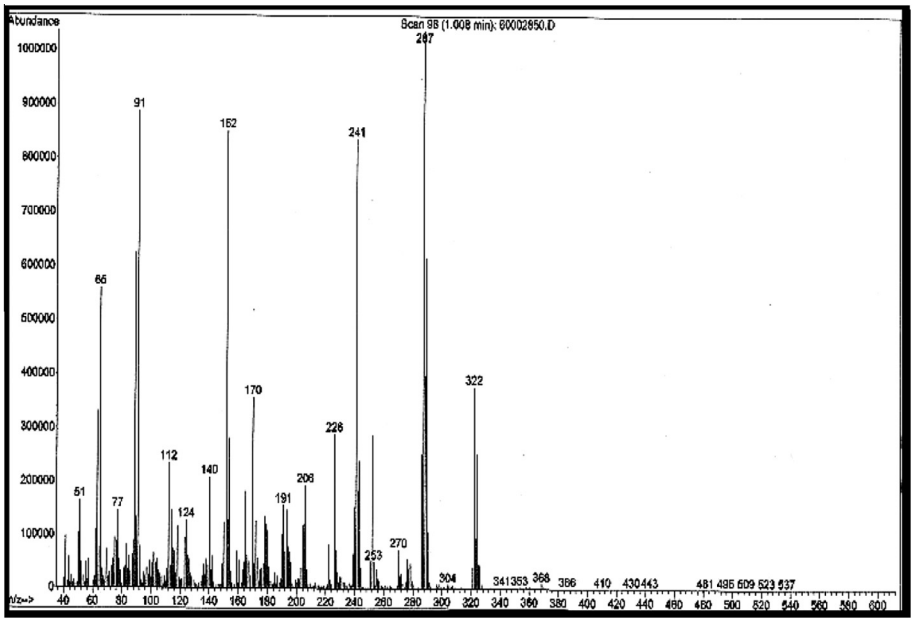

Fig. 1. The mass spectrum of compound (**2g**).

The structures of products were 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 = 1490–1600 cm^{-1}. The stretching vibration of C–H in the alkyl groups was appeared at region between ν = 2898–2930 cm^{-1}. In the ^{1}H NMR spectra, one proton of CH–N has chemical shift in δ = 3.65–4.40 ppm. 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 ppm and the signal around δ = 74.1–77.1 is assigned by one carbon of CCl_2 of aziridine ring. The Mass spectrum of product (**2g**) is indicated in Fig. 1. The peak with m/z = 322 related to molecular ion was confirmed the structure of this compound.

4. Conclusion

In this study, we have synthesized *gem*-dichloroaziridine derivatives through the reaction of various Schiff base compounds with Mg/CCl_4 under ultrasonic irradiations. The corresponding products have been obtained in excellent yields, high purity and short reaction times under neutral conditions. The products have been confirmed by physical and spectroscopic data such as; IR, ^{1}H NMR, ^{13}C NMR, MS spectroscopy and C. H. N. analyses.

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