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Original Research Article

Synthesis and Characterization of 1, 9-Diazahomoadamantane-4, 6-dione Derivatives

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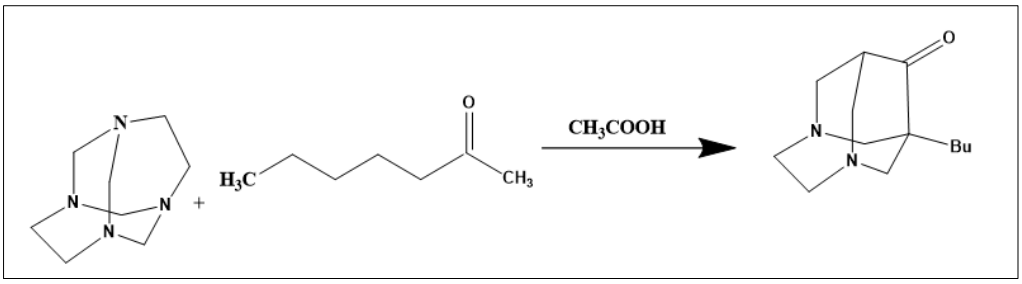
Abstract: This study explores the synthesis and characterization of a series of 1,9-diazahomoadamantane-4,6-dione derivatives, compounds with extremely interesting pharmacological properties. Several new derivatives were prepared through reactions involving condensation and hydrazine treatment to obtain Schiff bases and hydrazone derivatives, respectively. The identity of the molecules in the samples was determined based on data from the infrared (IR) and nuclear magnetic resonance (NMR) spectroscopy. The synthesis of [(4E,6E)-4,6-dihydrazineylidene-1,9-diazatricyclo[5.4.1.1^{3},^{9}]tridecane] was done by the reaction of 1,9-diazahomoadamantane-4,6-dione with hydrazine hydrate, and Schiff bases were produced by condensation with substituted aromatic amines. The structures of the prepared materials were supported by their IR and NMR spectra which depicted their characteristics, such as carbonyl, imine, and hydrazine groups which are among the biological active groups. These findings pave the way for the creation of new molecules that could be pharmacologically active with possible applications in the development of new drug candidates based on the diazahomoadamantane scaffold.

Keywords: 1,9-Diazahomoadamantane-4,6-Dione Derivatives, Pentane-2,4-Dione, Hydrazine Hydrate, Mannich's Reaction, Schiff Base.

INTRODUCTION

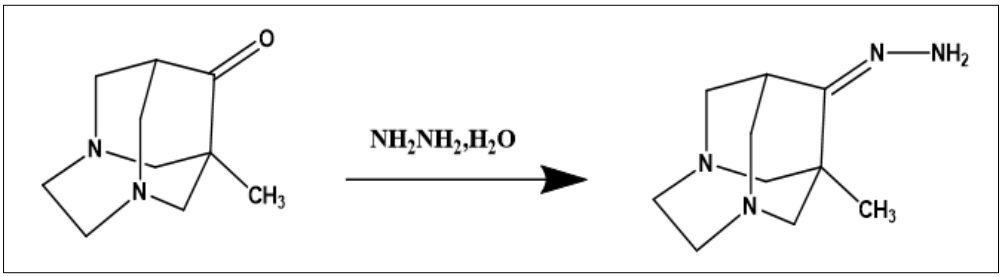
The synthesis of heterocyclic compounds has garnered significant attention in the field of organic chemistry due to their diverse pharmacological and biological activities. Besides, the diazahomoadamantane derivatives are one of the areas of research that have been drawing attention due to the simple but astonishing features of the structure and the potential use of these compounds in pharmacology. Among these, the 1,9-diazahomoadamantane-4,6-dione derivatives have turned out to be an interesting issue of research on the basis of their chemical reactivities and biological properties including antimicrobial, anti-inflammatory and anticancer activities [1, 2]. Since the adamantane's core is a strong, three-dimensional structure, it has been widely used to create molecules with biological activity when enriched structurally with nitrogen atoms such as in the diazahomoadamantane ring system. The new compounds exhibit more remarkable stability in chemical and reactivity [3]. Furthermore, the 4,6-dione group introduction into the framework also largely contributes to their multifunctionality, for which they foresee a great potential for the development of new therapeutic agents [4].

The various step of 1,9-diazahomoadamantane-4,6-dione derivatives synthesis is often based on reactions like nucleophilic substitution, cyclization, and displacing heteroatoms for precursor compounds application. Practically, these approaches have played a major role in obtaining the new derivatives with differentiated substitution patterns, aiming at influencing the chemical and biological properties of the same compounds [5]. The knowledge of the connection between the structure of these derivatives and their biological activities is the key to the informed planning of the upcoming drugs. As part of our ongoing research on the synthesis of novel 3,6-diazahomoadamantane derivatives, condensation of heptane-2-one was achieved with diethylene tetra methylene tetra mine [6], as depicted in Scheme 1. This method allowed the synthesis of 1-butyl-3,6-diazahomoadamantan-9-one.



Scheme 1: Synthesis of 1-butyl-3,6-diazahomoadamantan-9-one.

Derivatives of adamantane have been widely utilized in the development of treatments due to their diverse range of biological activities [7]. Diazahomoadamantane is a complex chemical compound with a polycyclic structure. First, it is necessary to comprehend how several fundamental molecules are brought together to create a larger one. It is well known that diethylene tetramethylene tetramine can be used to condense a cyclic ketone. In the presence of acetic acid, Mannich's reaction can yield 3,6-diazahomoadaman-9-one and its derivatives with substituents at the nodal position [8]. The ketone 1-phenylsulfanylpropan-2-one was used as the starting material. Its condensation with diethylene tetramethylene tetramine produced 1-phenylsulfanyl-3,6-diazahomoadamantan-9-one with a yield exceeding 60% [9], as in below. Furthermore, the compound shown in Scheme 2 was obtained via the reaction of a ketone with hydrazine hydrate [10, 11].



Scheme 2: The reaction of a ketone with hydrazine hydrate

Schiff bases are formed when aromatic or aliphatic amines condense with aromatic or aliphatic aldehydes or ketones, resulting in a carbon–nitrogen double bond (>C=N). They are used in chemistry on a large scale, in which the nitrogen atom's lone pair of electrons and the electron-donating properties of the C=N bond plays a key role. Due to these structural attributes, Schiff base is an eminent class of compounds in different medical and pharmaceutical fields from which it has been acknowledged to show a wide variety of biological activities [12-14].

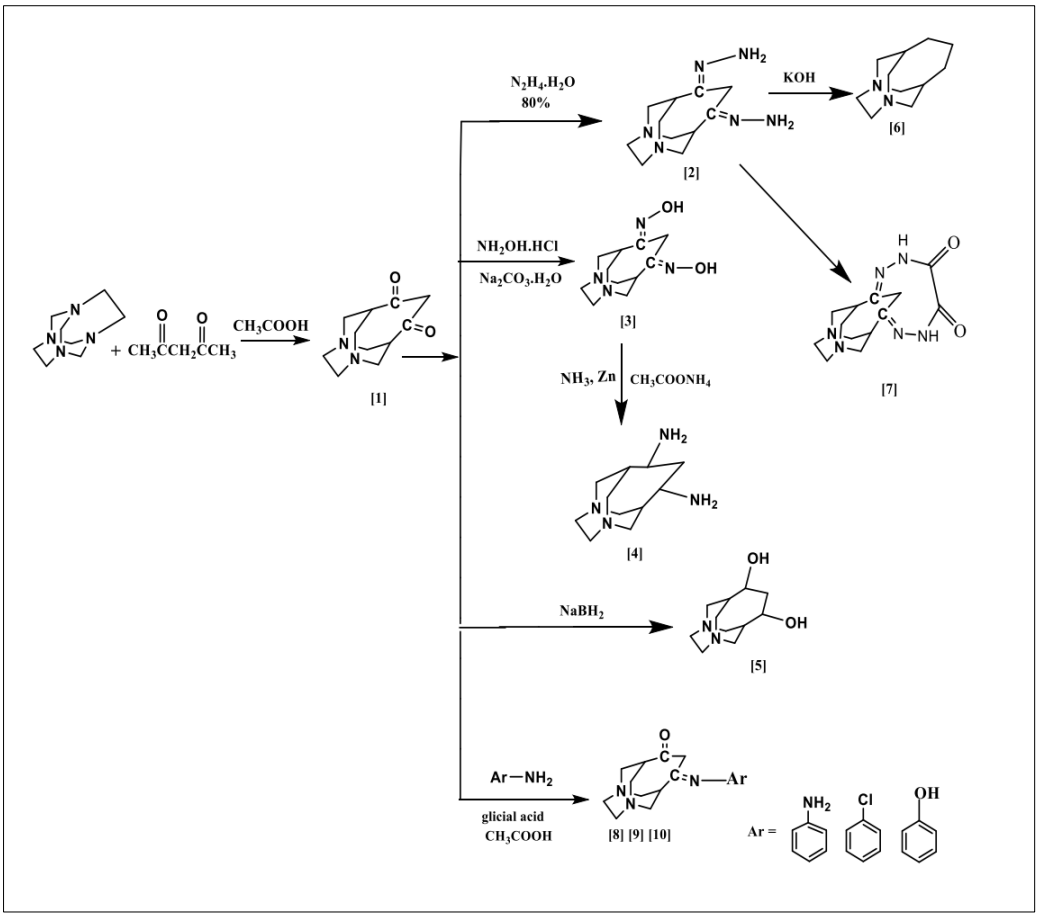
Under azeotropic reflux conditions, a hemiaminal intermediate is usually generated as a result of the nucleophilic addition of the $–NH_2$ group to the carbonyl group (C=O) of the aldehyde. Simultaneously, water is removed, and the intermediate undergoes dehydration, resulting in the synthesis of an imine [15]. Schiff bases are necessary for the synthesis of many different compounds with applications in industry and medicine. They are one of the key participants in the substitution, cycloaddition, and cyclization processes. On top of that, these substances are expected to reveal such a wide range of biological activities as a better-defined anti-inflammation, anti-allergy, radical scavenging, analgesia, and antioxidant (pharmacologic) action [16].

The present study aimed to synthesize and characterize a series of 1,9-diazahomoadamantane-4,6-dione derivatives, providing valuable insights into their chemical structures and potential biological activities. The synthesized compounds were identified by the means of advanced spectroscopic and analytical methods, including nuclear magnetic resonance (NMR) and mass spectrometry (MS). By investigating the relationship between the structure and activity of these derivatives, this study hopes to contribute to the development of novel pharmacologically active molecules with potential therapeutic applications.

EXPERIMENTAL SECTION

Sources of Chemicals

All chemicals were supplied by Fluke and Merck (local market). The Fourier Transform Infrared (FTIR) spectrum was obtained using a Shimadzu FTIR-8300 spectrophotometer (Shimadzu Corporation, Japan) with the KBr disk technique at the University of Baghdad, Iraq. Micro-elemental analysis (C, H, N, S) as well as the proton nuclear magnetic resonance (^{1}H-NMR) and Carbon-13 Nuclear Magnetic Resonance (^{13}C-NMR) spectra were conducted at Al-Bayt University in Amman, Jordan.



Scheme 3: Synthetic route for the preparation of the target compounds

Synthesis of [1,9-diazahomoadamantane-4,6-dione] „„„ (1)

In 50 milliliters of isopropyl alcohol, 8.40 grams (50 mmol) of diethylene tetramethylene tetraamine, 2.50 grams (25 mmol) of pentane-2,4-dione, and 9.00 grams (150 mmol) of acetic acid were heated for three hours at 60 to 70°C. After the mixture was concentrated under reduced pressure, hot heptane (4 × 40 mL) was used to remove the viscous substance. The residue was recrystallized from heptane. A white crystalline solid was isolated with a 46% yield of 3.90 g, which melted at 66–68°C. Peaks at 1720 and 1749 cm^{-1} indicating C=O were observed from the IR spectrum. The ^{1}H NMR spectrum (δ, ppm) represented: 3.63 m (4H, CH), 3.06-3.25 m (4H, NCH_2CH_2N), 2.96 m (4H, NCH_2C), 3.75-3.87 m . The ^{13}C-NMR spectrum (δC, ppm) had: 189.60 (C4, C6), 47.99 (C5), 49.07 (C7, C3), 52.87 (CH_2N), and 62.96 (1-CH_2CH_2). The molecular formula was $C_{11}H_{16}N_2O_2$, with calculated percentages: C, 63.44; H, 7.74; N, 13.45; O, 15.36. The molecular weight is 208.26 [6].

Synthesis of [(4E,6E)-4,6-dihydrazineylidene-1,9-diazatricyclo [5.4.1.1^{3,9}] tridecane] …. (2)

A mixture of 10 mL of 80% hydrazine hydrate and 5 mmol (1.04 g) of [1,9-diazahomoadamantane-4,6-dione] was heated and refluxed for three hours. Toluene was used to recrystallize the residue after the solution was raised. A white crystalline solid was isolated with a 0.78 g (75%) yield, which melted at 87–88°C. The IR spectrum of the compound showed NH_2 groups at 3189, 3288, 3311, and 3499 cm^{-1} and C=N groups at 1655 and 1678 cm^{-1}. The ^{1}H-NMR spectrum (δ, ppm) indicated: 2.14 m (4H, N=CCH), 2.36 m (4H, NCH_2CH_2N), 2.94 m (4H, NCH_2C), 3.01–3.30 m (2H, CCH_2C), and 4.93 s (4H, NH_2). ^{13}C-NMR (δC, ppm) had components of: 161.38(C4, C6), 26.10 (C5), 39.67 (C7, C3), 49.07 (CH_2N), and 55.44 (1-CH_2CH_2). The substance is composed of $C_{11}H_{20}N_6$ and the percentages calculated are: C, 55.91; H, 8.53; N, 35.56. The molecular weight is 236.32 [7].

Synthesis of [(4E,6E)-1,9-diazatricyclo [5.4.1.1^{3,9}] tridecane-4,6-dione dioxime] ……..... (3)

An aliquot of 20 mmol (4.16 g) of compound (1) was taken to which 9 mL of distilled water was added at room temperature. Next, a solution containing 50 mmol (3.47 g) of hydroxylamine hydrochloride in 9.9 mL of distilled water was added. The reaction was carried out at 80–90°C with stirring. Over 15 minutes, a solution containing 50 mmol (5.3 g) of sodium carbonate dissolved in 19.8 mL of distilled water was added. The reaction was only allowed to go on for an hour and then the mixture was evaporated, and the residue was washed with methanol. White crystals weighing 3.3 g (79%) were initially collected, and the crystals were found to melt at 86–88°C. Two C=N bands for oxime at 1634 and 1645 cm^{-1},

along with OH bands at 3456 and 3476 cm^{-1}, were observed using infrared spectroscopy. The ^{1}H-NMR spectrum (δ, ppm) showed the subsequent signals: 1.663 m (4H, N=CCH), 2.85 m (4H, NCH_2CH_2N), 3.03 m (4H, NCH_2C), 3.23–3.78 m (2H, CCH_2C, J = 13.9 Hz), and 5.95 s (2H, OH). The substance is composed of $C_{11}H_{18}N_4O_2$ and the elemental analysis data (percentage) are: C, 55.45; H, 7.61; N, 23.51; O, 13.43. The molecular weight is 238.29 [8].

Synthesis of 1,9-diazatricyclo [5.4.1.1^{3,9}] tridecane-4,6-diamine …….... (4)

An aliquot of 7.782 millimoles (1.85 g) of compound (3) were added to 45 mL of 25% ammonia, followed by the addition of 9 mL of absolute ethanol. The mixture was stirred for 15 minutes at room temperature. Next, 38.91 millimoles (2.529 g) of zinc were added to the mixture over a 15-minute period. The temperature was gradually raised to 50°C, at which point effervescence occurred. The first step was the reaction of a mixture which was stirred for 4 hours and then allowed to cool to the room temperature. Then, 25 mL of ethyl acetate was added to the mixture, after that the solvent was evaporated, and the precipitate formed was collected and recrystallized with toluene. A yield of 1.3 g (70%) of white crystals with melting point of 120–122°C was achieved. The IR spectrum shows the bands that correspond to NH_2 and OH groups combining at 3320, 3335, 3455, and 3478 cm^{-1}. The ^{1}H-NMR spectrum (δ, ppm) delivers the subsequent signals: 2.85 m (4H, NCH_2CH_2N), 1.863 m (2H, CH_2CNH_2), 2.34 m (4H, NCH_2CH), 3.00–3.35 m (2H, NCH_2CH, J = 13.9 Hz), and 5.39 s (4H, NH_2). The ^{13}C-NMR spectrum (δC, ppm) gathered these signals: 43.12 (C4, C6), 42.11 (C5), 38.22 (C7, C3), 51.11 (CH_2N), and 59.98 (1-CH_2CH_2). The molecular formula is $C_{11}H_{22}N_4$, with calculated percentages: C, 62.82; H, 10.54; N, 26.64. The molecular weight is 210.32 [8].

Synthesis of [(1s,9s)-1,9-diazatricyclo [5.4.1.1^{3,9}] tridecane-4,6-diol] ……. (5)

An isopropanol solution of 15 mL was made containing 1.04 g (5 mmol) of compound (1). The mixture was then shaken for 30 minutes at room temperature, and subsequently, over the period of 30 minutes, a solution composed of 0.38 g (10.2 mmol) of sodium borohydride dissolved in 10 mL of isopropanol was progressively added. The mixture was heated for 5 hours. Afterward, 5 mL of the solvent was evaporated, and 5 mL of distilled water was added to the mixture. The mixture was then extracted with 50 mL of toluene. The remaining solvent was evaporated, and the product was recrystallized from toluene. White crystals of 0.73 g (70%) were prepared with a melting point of 96–98°C. Two OH groups at 3368 cm^{-1} and 3425 cm^{-1} were indicated by the IR spectrum. The ^{1}H-NMR spectrum (δ, ppm) is as follows: 2.40 m (4H, NCH_2CH_2N), 2.25 d (4H, NCH_2C), 1.30 m (6H, HO-CCH), 1.35 m (2H, CCH_2C), and 4.49 s (2H, OH). The ^{13}C-NMR spectrum (ΔC, ppm) included 69.44 (C4, C6), 42.07 (C5), 38.82 (C7, C3), 42.07 (CH_2N), and 59.59 (1-CH_2CH_2). The molecular formula is $C_{11}H_{20}N_2O_2$, and the respective percentages were calculated as: C, 62.24; H, 9.50; N, 13.20; O, 15.07. The molecular weight is 212.29 [9].

Synthesis of (1s,9s)-1,9-diazatricyclo [5.4.1.1^{3,9}] tridecane ……. (6)

Following thorough mixing, 1.2 g (11 mmol) of potassium hydroxide and 0.47 g (2 mmol) of compound (2) were heated to 220–240°C for two hours. After the mixture was allowed to cool, it was extracted with toluene (3 × 20 mL). The solvent was removed by evaporation and the residue was recrystallized with toluene and 0.30 g (63%) of white crystals with melting point 64–66°C were obtained. The IR spectrum indicated the presence of aliphatic C-H stretches at 2850 and 2947 cm^{-1}. The ^{1}H-NMR spectrum (δ, ppm) included: 1.12–1.49 m (8H, $CHCH_2CH_2CH_2CH$), 1.82-1.84 m (4H, N=CCH), 3.87–3.83m (4H, NCH_2CH_2N), 2.98-2.95 d (4H, NCH_2C), 2.34 d (4H, NCH_2C), and 2.33–2.27 m (2H, CCH_2C). The ^{13}C-NMR spectrum (δC, ppm) consisted of 26.10 (C4, C6), 39.26 (C5), 40.09 (C7), 55.23 (CH_2N), and 62.96 (CH_2CH). The molecular formula is $C_{11}H_{20}N_2$, with calculated elemental analysis (%, mass) C, 73.28; H, 11.18; N, 15.54. The molecular weight is 180.30 [11].

Synthesis of (1E,10E)-4,7,11,12,15,16-hexaazatetracyclo [8.6.1.1^{2,7}.1^{4,9}] nonadeca-1(16),10-diene-13,14-dione …. (7)

Compound (2), 3.25 g of oxalic acid, and 30 mL of 4 N HCl were mixed together and the solution was heated to boil for 2 hours. A crystalline solid was obtained as a result of the reaction. After cooling the reaction mixture, the solid was separated, washed with water, and dried. The compound obtained was dissolved in alkali and then acidified with HCl to give the pure compound. The compound was recrystallized from toluene. A melt point of 233–231°C of 5 g of white crystals was obtained. The IR indicated C=O stretches at 1703 and 1711 cm^{-1}, N-H stretches at 3333 and 3329 cm^{-1}, and C=N stretches at 1644 and 1653 cm^{-1}. The ^{1}H-NMR spectrum (δ, ppm) included: 1.33–1.39 m (8H, $CHCH_2CH_2CH_2CH$), 1.56 m (4H, N=CCH), 3.54–3.62 m (4H, NCH_2CH_2N), 3.22 d (4H, NCH_2C), 3.11 d (4H, NCH_2C), 2.52–2.63 m (2H, CCH_2C), and 12.5 s (NH). The molecular weight is 290.33 [17].

Synthesis of Schiff base compounds (8, 9, and 10)

Schiff bases were synthesized through a condensation reaction between substituted aromatic amines and compound (1). In a typical procedure, compound (1) (0.6 g, 3.0 mmol) was dissolved in 5 mL of ethanol in a round-bottom flask. The requisite aromatic amine (3.0 mmol) and three drops of glacial acetic acid were added as a catalyst. The reaction mixture was heated under reflux for 3–4 hours to facilitate the formation of the imine bond between the amine and the carbonyl carbon of compound (1). Upon completion, the mixture was allowed to cool to ambient temperature, and then

gradually placed into an ice water bath. After the solvent evaporated, the residue was recrystallized from toluene [18-20]. The physical properties are summarized in Table 1.

Table 1: Structures and physical properties of the synthesized compounds (8, 9, and 10)

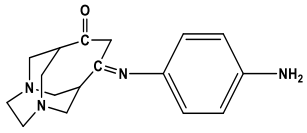
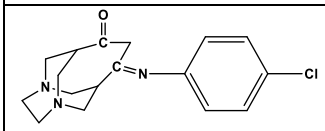
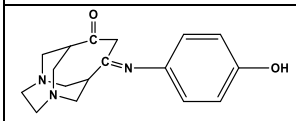
Compound	Color	MP (°C)	Yield	Elemental analysis
 8	White	112-114	68%	C: 68.43, H: 7.43, N: 18.78, O: 5.36
 9	White	124-126	73%	C: 64.25, H: 6.34, Cl: 11.15, N: 13.22, O: 5.03
 10	White	116-118	77%	C: 68.20, H: 7.07, N: 14.04, O: 10.69

Table 2: The ^{1}H-NMR spectral data (δ, ppm) and FTIR spectral data (υ, cm^{-1}) of compounds (8, 9, and 10)

Compound Chemical NO Shifts	C-H Aromatic	C-H Aliphatic	C=O	C=N	C=C	Other spectral
8 6.84 (S, 2H, NH_2), 6.86-7.73 (d, 4H, H-Ar), 1.86 (m, 1H, CHC=N), 2.51-3.50 (m, 16H, CH_2 and CH-CH_2 group)	3070	2916	1712	1635	1450	NH_2 3330, 3350
9 6.81-7.13 (d, 4H, H-Ar), 1.43 (m, 1H, CHC=N), 2.34-3.35 (m, 16H, CH_2 and CH-CH_2 group)	3054	2923	1718	1642	1488	C-Cl 673
10 9.87 (s, 1H,OH), 6.83-7.22 (d, 4H, H-Ar),1.47 (m, 1H, CHC=N),2.45-3.38 (m, 16H, CH_2, and CH-CH_2 group)	3013	2928	1711	1648	1469	C-OH 3553

RESULTS AND DISCUSSION

One reaction converted diethylene tetra methylene tetraamine and pentane-2,4-dione into one compound, [1,9-diazahomoadamantane-4,6-dione]. The IR of the resultant chromatographic sample represented two specific bands at 1720 cm^{-1} and 1749 cm^{-1}, which allowed the identification of the C=O stretching vibrations from the first and the second carbonyls, respectively. Concerning the ^{1}H-NMR spectrum, (Figure 2) conveys the carbon framework, and the signals coming from it are multiplets in the region of 2.96–3.87 ppm. The ^{13}C-NMR data provide evidence for one carbonyl group at positions 4 and 6 (189.60 ppm), and for the methylene groups (CH_2) that are bonded to nitrogen at 52.87 ppm. The peaks at 47.99 ppm and 49.07ppm give the possibility to the carbons in 5 and 3, respectively. The one at 62.96 ppm is the signal of an ethyl group (CH_2CH_2) at position 1(Figure 2).

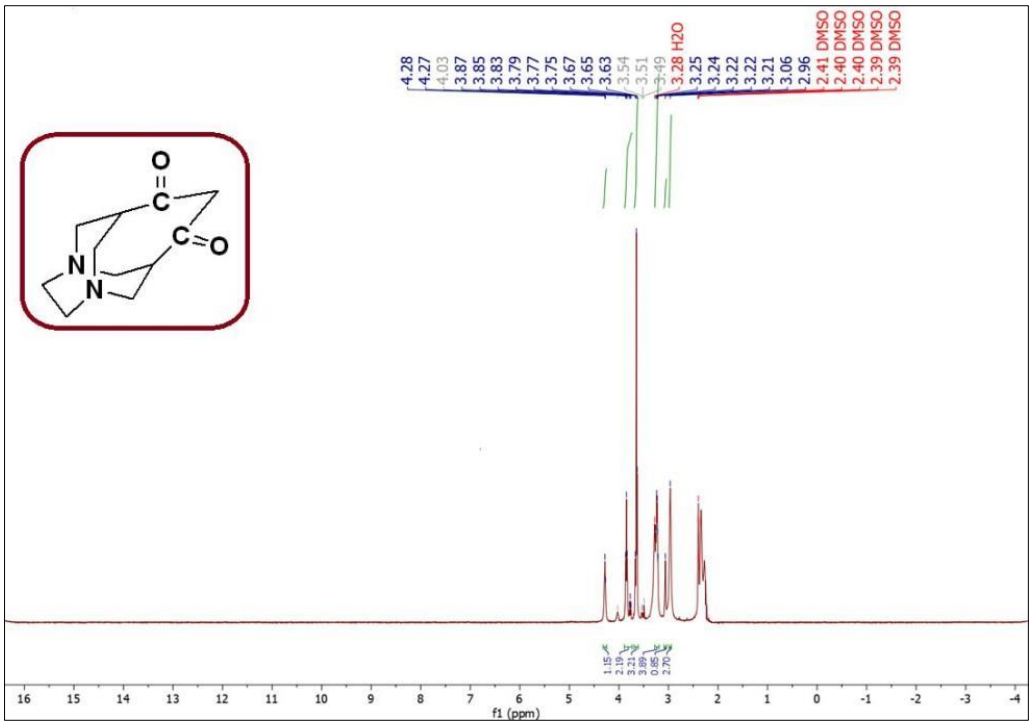


Figure 1: ^{1}H-NMR spectrum of compound (1).

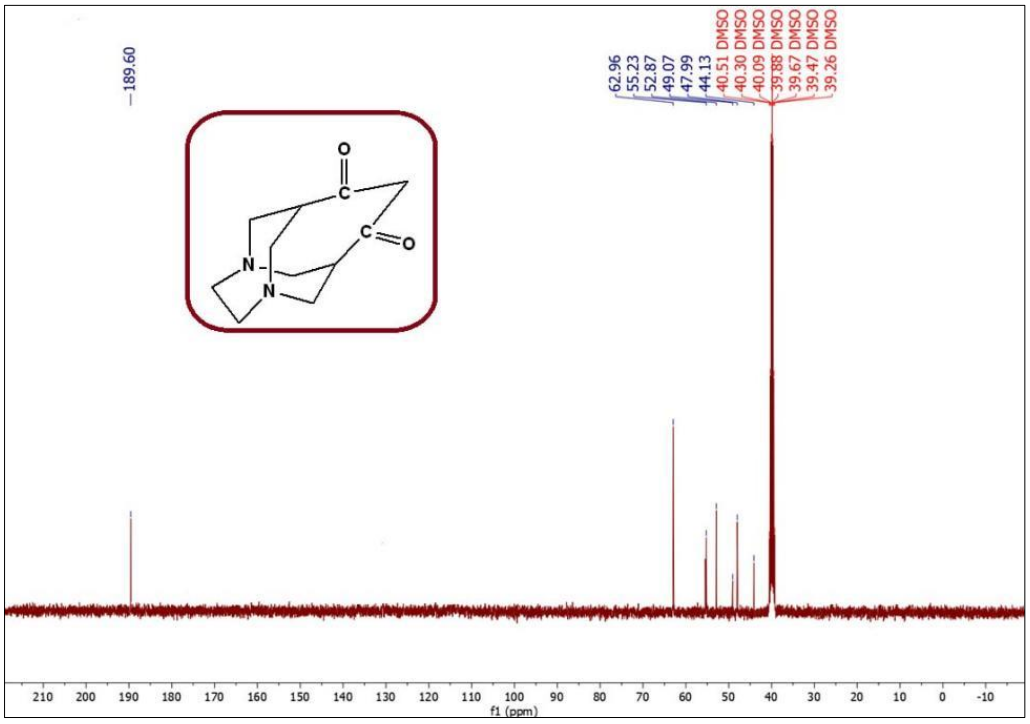


Figure 2: ^{13}C-NMR spectrum of compound (1).

Hydrazine hydrate was used to react with compound (1) to prepare compound (2), [(4E,6E)-4,6-dihydrazineylidene-1,9-diazatricyclo[5.4.1.1^{3},^{9}]tridecane]. One of the most important pieces of evidence for the success of the reaction is the IR spectral data collected. In particular, the disappearance of carbonyl bands has been reported, as well as the occurrence of typical NH_2 stretching vibrations at 3189 and 3288 cm^{-1}, and 3311 and 3499 cm^{-1}, which suggest that amino groups have been generated. In addition, the progress of the reaction from hydrazine moieties was verified by the simultaneous appearance of absorption bands at 1655 and 1678 cm^{-1}, the latter corresponding to C=N bonds. The complete condensation of the carbonyl functionalities has led compound (2) to be of the structure with two hydrazine groups.

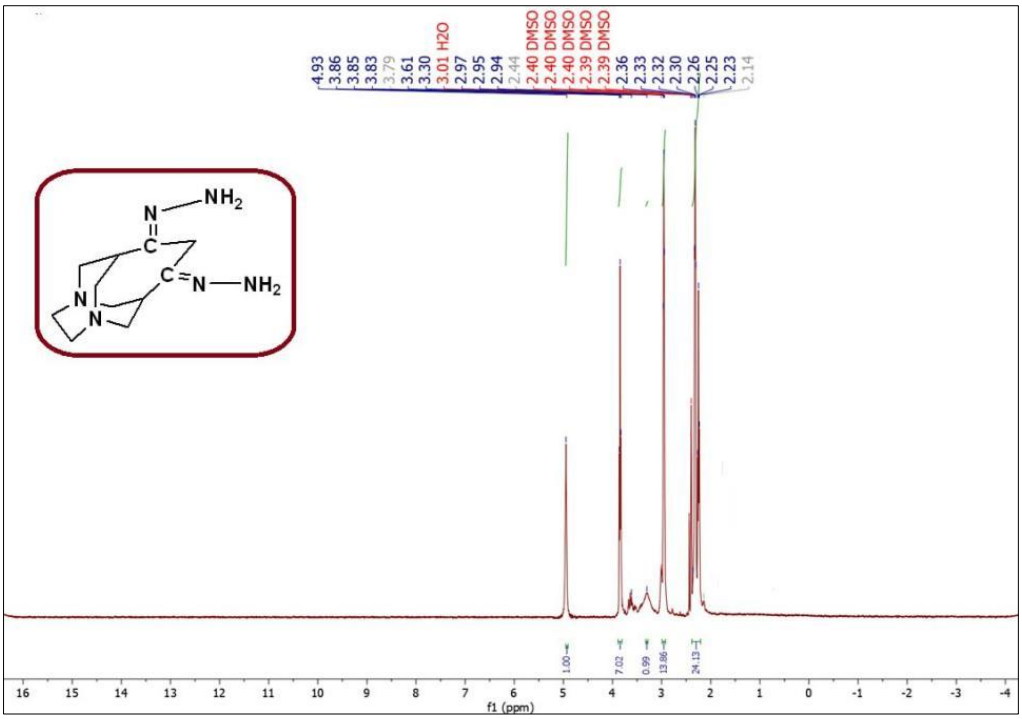


Figure 3: ^{1}H-NMR spectrum of compound (2).

There is a wide signal centred at 4.93 ppm in the ^{1}H-NMR spectrum that is associated with the two NH_2 groups. The number of protons corresponding to the intensity of this signal is four, and hence, it can be concluded that the structure of compound (2) contains two terminal amino groups. (Figure 4) makes the point very clear. Figure 4 contains the ^{13}C-NMR spectrum of compound (2), through which various structural signals were identified: Peaks derived from C4 and C6 at 161.38 ppm were assigned to the conjugated system around the C=N bonds in the two hydrazine units. A signal at 26.10 ppm for C5 is consistent with a methyl carbon or a carbon bonded to an atom of low electronegativity. The two peaks at 39.67 ppm that are for C7 and C3 show that these are aliphatic surroundings of the ring. One signal at 49.07 ppm for CH_2N confirms the carbon bonded to nitrogen in the hydrazine linkage. The peak at 55.55 ppm for the 1-CH_2-CH_2 group suggests the presence of methylene bridging two carbon centre's.

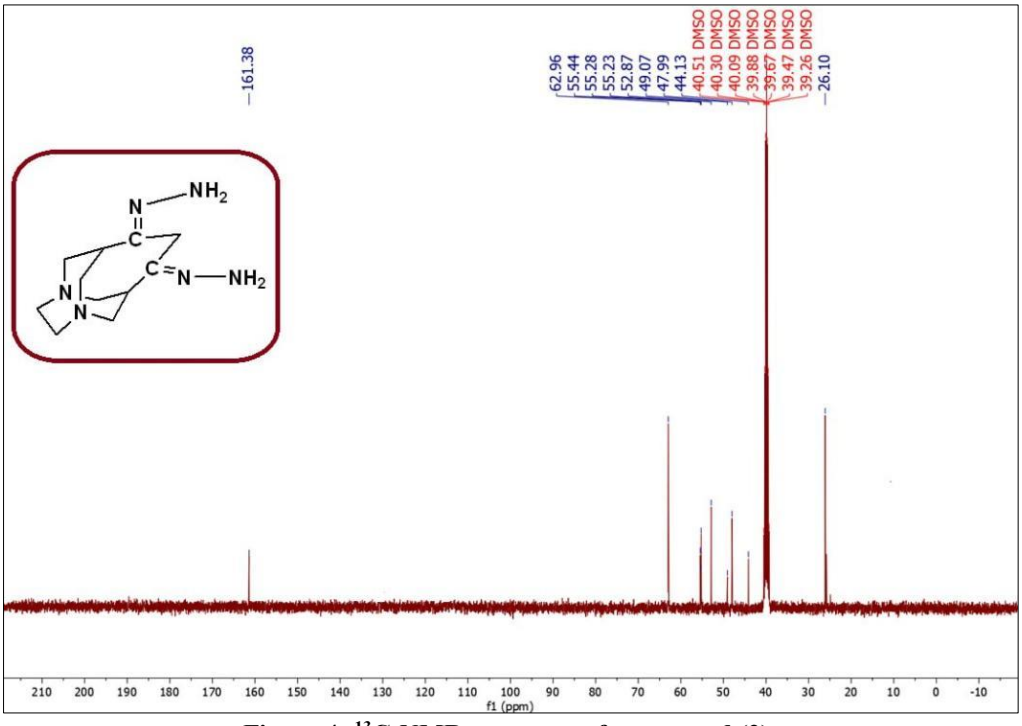


Figure 4: ^{13}C-NMR spectrum of compound (2).

The complete shift of the two carbonyl groups in compound (1) can be seen from the subsequent product's IR spectrum. Carbonyl (C=O) absorption bands, which normally appear at around 1720 and 1749 cm^{-1}, have disappeared, showing that these groups have already been converted. Newly formed absorption bands at 1634 and 1645 cm^{-1} indicate the stretching vibrations of the C=N double bond. Moreover, the bands at 3456 and 3476 cm^{-1} represent the O–H stretching vibrations of the oxime group. The changes in the spectra give very strong evidence that the two carbonyl groups have been converted into two oxime groups (–C=NOH), thus the reaction's advancement and the generation of compound (3) with the expected structure are confirmed. A broad singlet at δ 5.95 ppm was the ^{1}H-NMR spectrum feature. The protons of the two hydroxyl groups bound to the oxime moieties (–C=NOH) give this signal which is a confirmation of the presence of two oxime functionalities in the molecular structure. The δ 1.663 ppm (4H, m) signal can be explained as methylene protons next to the imine bond in the –N=C–CH_2– units, thus it is in line with the proposed oxime structure. Furthermore, based on the two signals at δ 2.85 ppm and δ 3.03 ppm (each 4H, m), one can conclude that the methylene protons of CH_2 groups that are attached to nitrogen atoms are there, which means that the molecule has an aliphatic –N–CH_2–CH_2–N– chain.

A separate multiplet at δ 3.23–3.78 ppm (2H, m, J = 13.9 Hz) is the central CH_2 group flanked by two carbon atoms (–C–CH_2–C–) that we are talking about. The value of coupling constant found here (J = 13.9 Hz) happens to be for symmetrical magnetic coupling and this is one of the reasons structural symmetries in the molecular framework is ensured. The IR spectrum of compound (4) shows the characteristic bands of the oxime groups (–C=NOH) that were present in the previous compound (3) no longer exist. Besides, new absorption bands at 3320, 3335, 3455, and 3478 cm^{-1} can be seen that correspond to the N-H bond stretching vibrations in the primary amine groups (–NH_2). These spectral changes represent the conversion of oxime groups into amine groups by reduction with zinc in a basic medium (aqueous ammonia solution), a common method in organic chemistry for the production of amines from oximes. The ^{1}H-NMR signals indicate the presence of primary amine groups, e.g., at δ 5.39 ppm, a singlet corresponding to NH_2 protons, and several resonances for CH_2 groups attached to nitrogen, exhibiting different chemical environments for these groups. Besides, conversion of oxime to amine while keeping the underlying molecular structure has been confirmed by ^{13}C-NMR spectra showing signals for aliphatic carbon atoms directly bonded to or next to nitrogen atoms.

The IR spectrum of compound (5) is shows additional sharp bands at 3368 and 3425 cm^{-1} that correspond to the stretching vibrations of hydroxyl groups (O–H) in place of the carbonyl absorption band near 1700 cm^{-1}. Such a change affirms that the two carbonyl groups have been converted into two alcohol groups by treatment with sodium borohydride ($NaBH_4$) in isopropanol. Decrease in ^{1}H-NMR and ^{13}C-NMR data show that the compound is a saturated aliphatic with two terminal hydroxyl groups and a number of methylene groups linked to nitrogen atoms as shown in Figure 5 and Figure 6. The observation of hydroxyl signals coupled with the lack of carbonyl signals is a strong indication that the compound is a product of two carbonyl groups' reduction to two primary alcohols, consistent with both the synthetic pathway and elemental analysis for compound $C_{11}H_{20}N_2O_2$ with a molecular weight of 212.29.

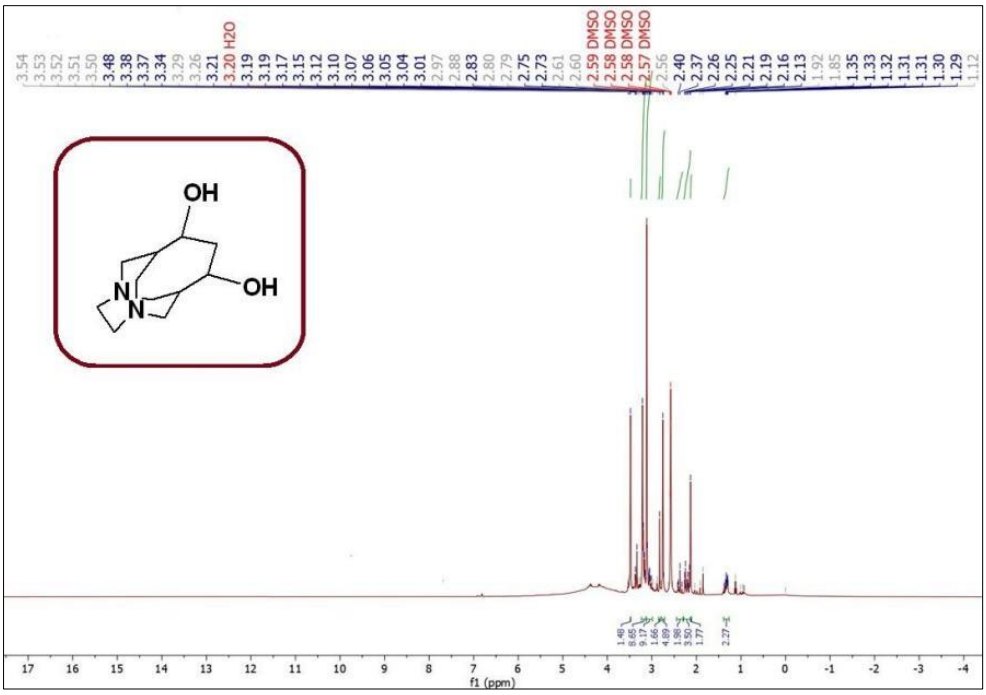


Figure 5: ^{1}H-NMR spectrum of compound (5)

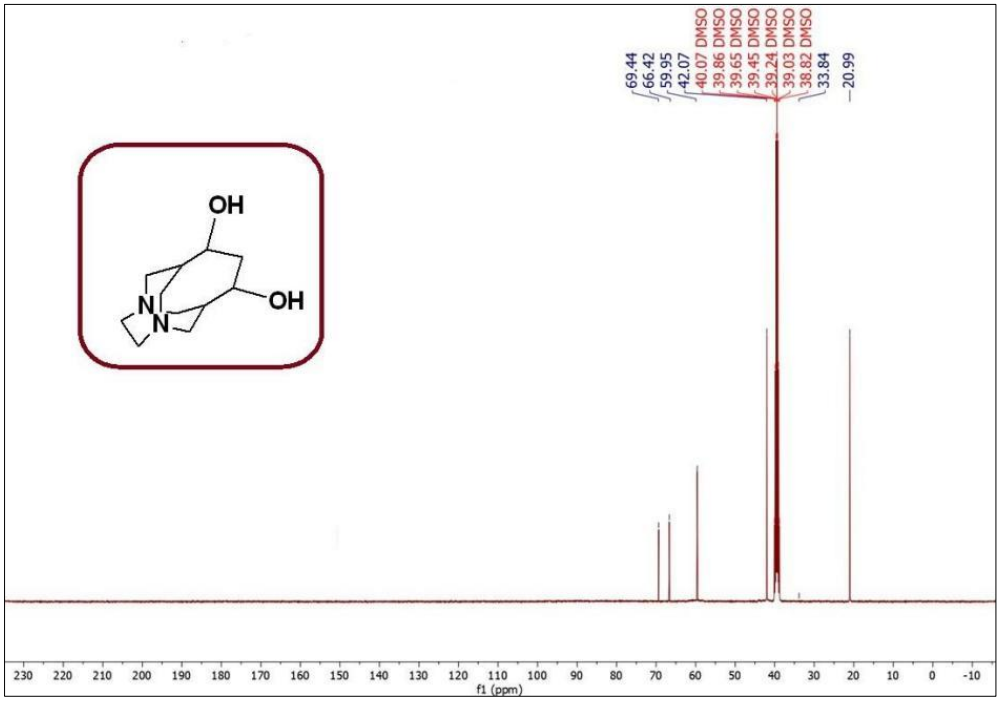


Figure 6:[13]C-NMR spectrum of compound (5)

The peaks of the IR spectrum are displayed coming from the sample at 2850 and 2947 cm-[1], which are the main features of the vibrations of aliphatic C–H bonds. The region ~3300–3500 cm-[1], where N–H stretching vibrations of amines are frequently found, had absolutely no peaks. In other words, the disappearance of the $–NH_2$ group is confirmed. The present IR indicated only the presence of aliphatic C–H bonds and no impurities, such as carbonyl or hydroxyl, were found in the sample.

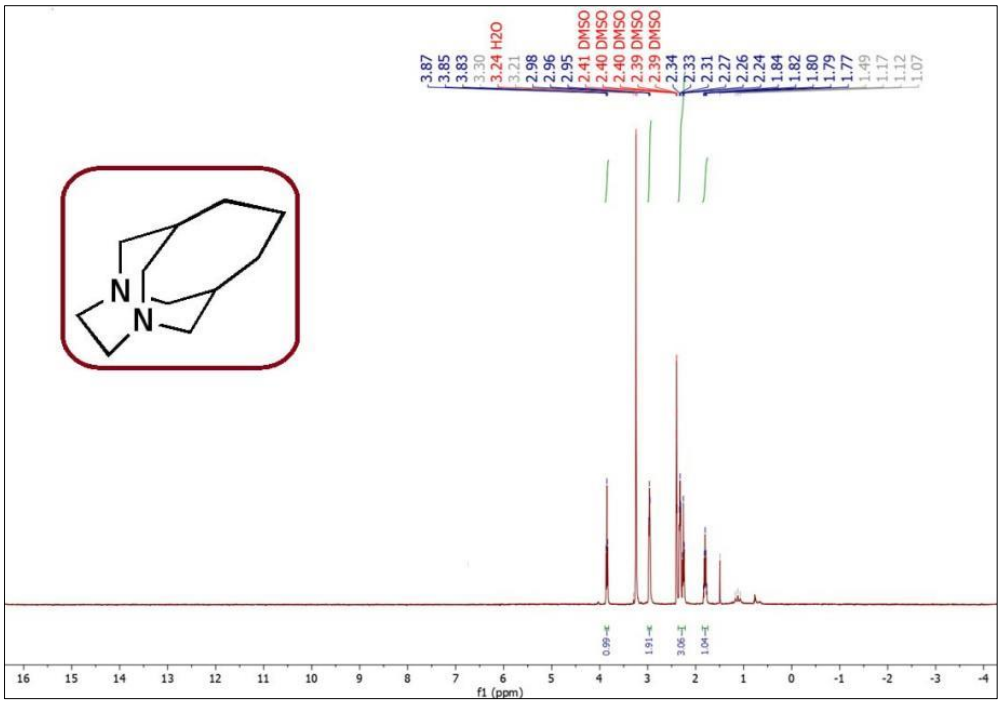


Figure 7: [1]H-NMR spectrum of compound (6)

The [1]H-NMR spectrum shows the sample consisting of aliphatic chains, and those CH_2 groups that are bonded to two nitrogen atoms, thereby it is a saturated cyclic structure with methylene bridges and an imine group, as demonstrated in Figure 7.

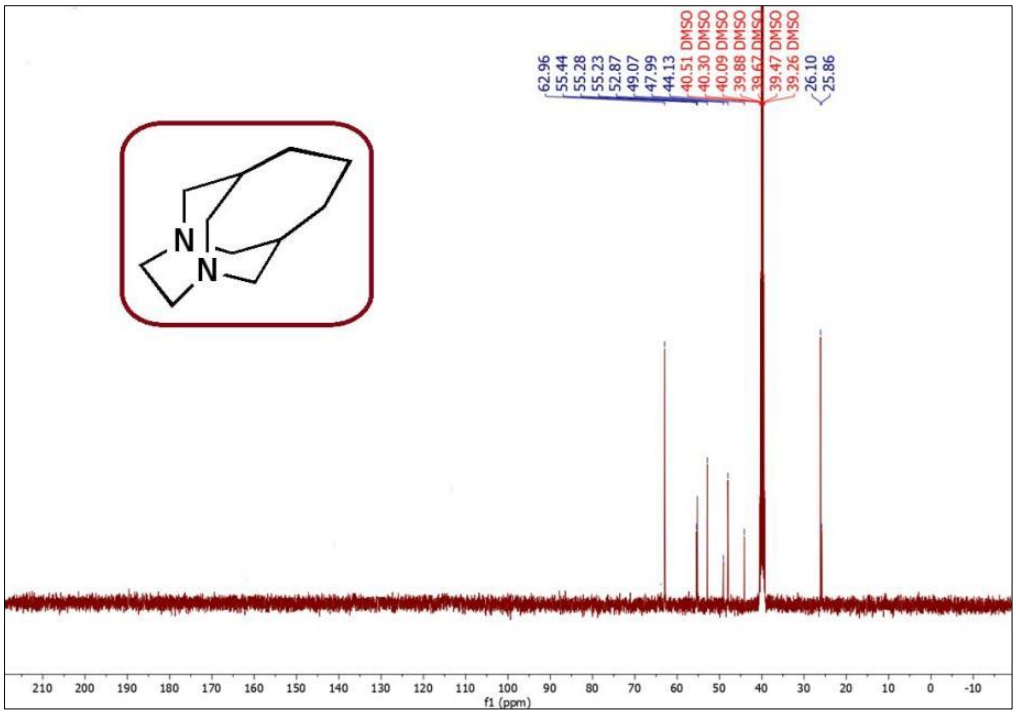


Figure 8: ^{13}C-NMR of compound (6)

The three Schiff bases (compounds 8, 9, and 10) were prepared by the interaction of compound (1) with primary amines. Compound (1) is a compound with two carbonyl groups, and the process is shown in Scheme 8. The reaction, however, goes only through one carbonyl group, thus the imine (C=N) formation becomes typical. The IR spectra of the samples were very much alike and showed the imine stretching vibrations at 1635, 1642, and 1648 cm^{-1}, respectively, besides stretching vibrations of the C=O and C=C bonds.

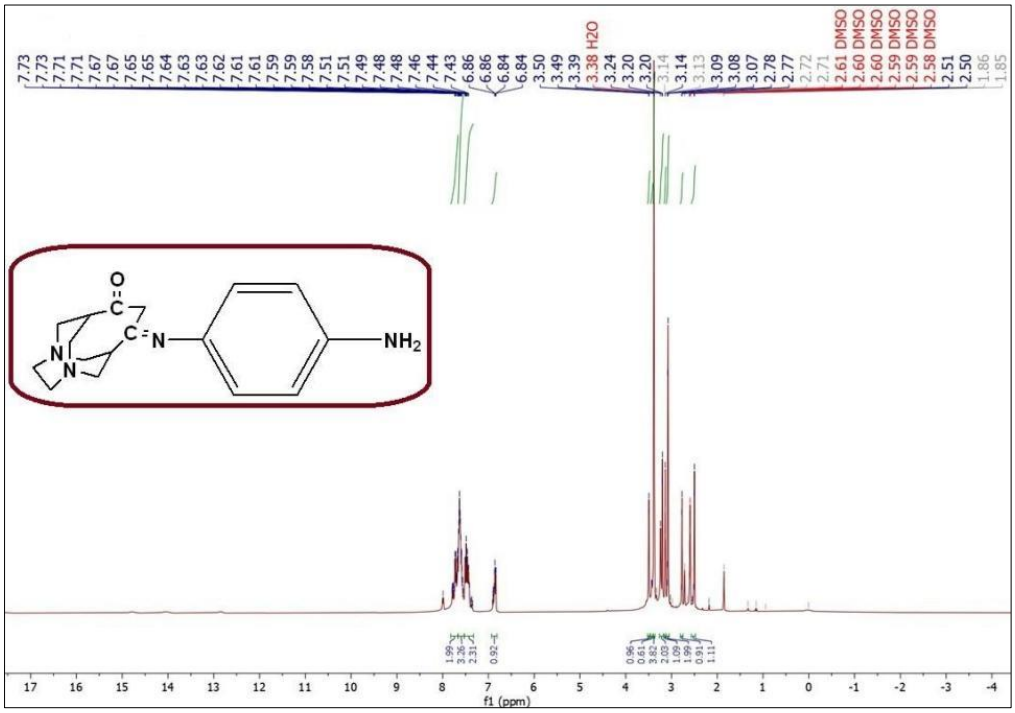


Figure 9: ^{1}HNMR spectrum of compound (8)

The ^{1}H-NMR spectra of compounds (8, 9, and 10) indicate the presence of one proton at 1.4–1.5 ppm attached to the carbon adjacent to the imine bond (CH–C=N), thus the formation of the imine linkage in these compounds was confirmed. These compounds also show some signals in the range of 6.8–7.3 ppm coinciding with the aromatic protons on the aromatic ring and hence the aromatic structure remains intact. In compound (8), in the region of 6.86-7.73ppm, the

signal at 6.84 ppm due to the primary amine group (NH_2) is quite broad, thus partially converted during the condensation reaction is the fraction of the amine group that remains unreacted. There are many signals with intensities from 2.3 to 3.6 ppm and the signals in these regions can be assigned to methylene groups and aliphatic side chains, respectively, the latter being the result of high molecular complexity and close proximity of the molecule to the reactive centre's. Figure 9 shows the compound (8) ^{1}H-NMR spectrum where broad NH_2 signal at 6.84 ppm, aromatic proton region between 6.86 and 7.73 ppm, CH adjacent to the imine at 1.86ppm, and aliphatic methylene signals between 2.51 and 3.50 ppm are indicated Figure 9. Figure 10 illustrates the formation of compound 8, as shown in the ^{13}C NMR spectrum.

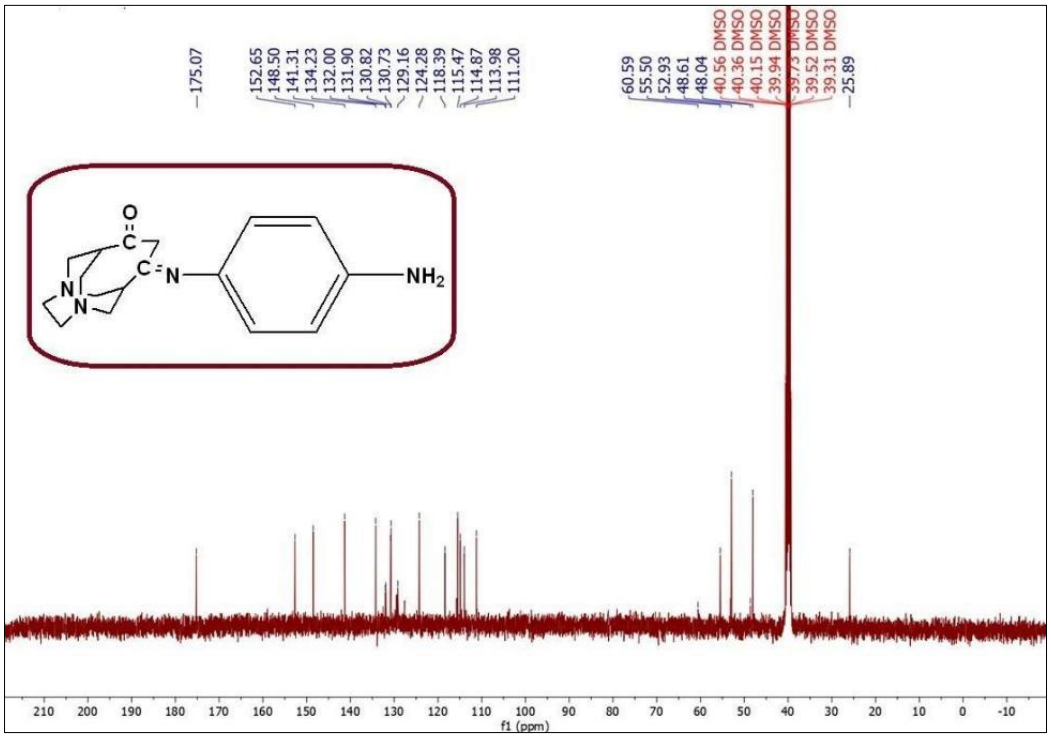


Figure 10: ^{13}C-NMR spectrum of compound 8

CONCLUSION

The compound, 1,9-diazahomoadamantane-4,6-dione was made with the reaction of diethylene tetramethylene tetraamine with pentane-2,4-dione in an acidic alcoholic medium, followed by recrystallization. This substance was the main constituent of the Schiff base synthesis. The derivatives of [(4E,6E)-4,6-dihydrazone-1,9-diazatricyclo [5.4.1.13,9] tridecane] were made via the reaction of compound (1) with hydrazine hydrate, under reflux. Contrarily, hydroxylamine hydrochloride reaction was used to synthesize [(4E,6E)-1,9-diazatricyclo [5.4.1.13,9] tridecane-4,6-dione dioxime]. The zinc reduction of compound (3) resulted in 1,9-diazatricyclo [5.4.1.1^{3},^{9}] tridecane-4,6-diamine and sodium borohydride was used to reduce compound [1] hence the formation of [(1s,9s)-1,9-diazatricyclo [5.4.1.1^{3},^{9}] tridecane-4,6-diol] was observed. The treatment of compound (2) with potassium hydroxide and oxalic acid resulted in pure crystalline compounds. The method for obtaining the Schiff bases involved the condensation of the corresponding amines. All the components were identified through several spectroscopic methods.

Conflict of Interest: No conflict of interest.

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