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A NEWLY SYNTHESIS AND IDENTIFICATION OF 5-NITRO-2,4,6-TRIPHENYL HEXAHYDRO PYRIMIDINE DERIVATIVES STRUCTURES

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ABSTRACT

The Mannich reaction was used to synthesise new hexahydro pyrimidine derivatives with good to excellent yields of eleven derivatives. The reaction was based on the condensation of three major components, nitromethane, ammonium acetate, and mono- or di-substituted benzaldehyde compounds in the ratio 1:2:3, which were mixed with n-butanol as a solvent system. All products were characterized using the fundamental techniques of FT-IR, 1H-NMR, and mass spectroscopy.

Keywords: Condensation, Mannich reaction, nitro-hexahydro pyrimidine

I. INTRODUCTION

One of the significant applications of hexahydro pyrimidine derivatives is in pharmaceuticals and medicinal chemistry. Hexahydropyrimidines are a type of heterocycle compounds with biologically effective and a wide area of physiological activity, such as cytotoxic (Ibraheem *et al.*, 2018), antitumoral (Durga devi, Manivarman and Subashchandrabose, 2017), antimicrobial (Murahari *et al.*, 2017), antibacterial (Shamim *et al.*, 2018), antiviral (Rivera, Miranda-Carvajal and Ríos-Motta, 2018) and antiarrhythmic (Devi *et al.*, 2011). The compounds in this class are physiologically active in a wide range and can also be subsequently modified to make syntheses of the large libraries of new heterocycles possible (Muravyova *et al.*, 2007; Agbaje *et al.*, 2011; Gibadullina *et al.*, 2018). The reaction of aldehydes or 1,3-dicarbonyl with urea or thiourea (Zohdi, Rateb and Elnagdy, 2011; Abu-Obaid *et al.*, 2015); aldehydes or ketones with 1,3-diamine which called Biginelli reactions (Katritzky, Singh and He, 2002) (Senkus, 1946); or Mannich CH condensation of aldehyde and amine (Dandia, Jain and Sharma, 2012), (Urbański, 1974), (Cox, 1968) are commonly used in the built of a hexahydropyrimidone ring. Tetrahydropyrimidine derivatives can also be made using multicomponent processes and chemical synthesis with ultrasound activation. Some nitro groups are formed by using a Mannich condensation reaction which carried out by mixing the suitable primary amine, primary nitroalkanes or nitro olefins, and formaldehyde (Dabrowska-Urbanska, AR and T, 1969; Petzold *et al.*, 2001). In addition, hexahydropyrimidone and several of its derivatives produced nitrogen donor polydentate complexes in mono-, di-, and polycoordination, coordinating forms of transition cations. Hexahydro pyrimidine derivatives are used to stabilise polymers (Mayr and Buchmeiser, 2004; Schmidt, Wiedemann and Grohmann, 2011; Wang *et al.*, 2017). In this paper, we describe new 5-nitro-2,4,6-triphenyl hexahydro pyrimidine derivative compounds and their characterization in order to present them in future applications. Furthermore, using FT-IR, 1HNMR, and MS characterizations, we discuss the effects of substitute groups on the hexahydro pyrimidine ring.

II. MATERIALS AND METHODS

Ammonium acetate (99%) was supplied by Chem-Lab. 4-Methyl benzaldehyde (98%), and 4-hydroxy-3-methoxy benzaldehyde (99%) were supplied by EXIR. Nitromethane (98%), 4-nitro benzaldehyde (99%), 2-nitro benzaldehyde (98%), 4-methoxy benzaldehyde (98%), 4-chloro benzaldehyde (97%), 4-bromo benzaldehyde (98%), 4-floro benzaldehyde (99%), 2,4-dichloro benzaldehyde (98.9%), 4-pyridine carboxaldehyde (99%) and 2-chloro-4-floro benzaldehyde (97%) were supplied by Aldrich. The Stuart (SMP 30) apparatus was used to determine melting points. Proton nuclear magnetic resonance (^{1}H NMR) was used (a solvent is DMSO-d_6 and an internal standard is TMS) Bruker 500 MHz-Avance III to collect spectra. Fourier-transform infrared (FT-IR) was

used to determine function groups by using the Bruker Tensor-27 (ATR) technique. Finally, the mass spectrometry (Electron Impact 70 eV) 5973 Network Mass Selective Detector spectrometers

Synthesis of 5-nitro-2,4,6-tri substituted phenyl hexahydro-pyrimidine in basic procedure (1a-g)

In 35 ml n-BuOH, 0.03 mol of benzaldehyde derivatives, 0.02 mol of ammonium acetate, and 0.01 mol of nitromethane were mixed. The refluxed time of the mixture was 15-75 min at 90 °C until the formation of the suspended solution. Reactions were followed by thin-layer chromatography (TLC) (using a Benzene: Acetone mixture in a 9:1 ratio). The reaction mixture was set aside for the following day. The precipitate was filtered and recrystallized from toluene after it had precipitated.

5-Nitro-2,4,6-tris(4-nitrophenyl) hexahydro pyrimidine (1a)

4-nitrobenzaldehyde (0.03mol; 3.18g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 82% yield and a melting point of 140 °C; $C_{22}H_{18}N_6O_8$. FT-IR (ATR) (ν, cm-1): 3418.6 (N-H), 1522.6 & 1343.9 (-NO_2), 3106 (sp^2-C-H), 3060 (sp^3-C-H), 1587.1 & 1448.8 (C=C). ^{1}H NMR (DMSO-d_6; 500MHz), δ, ppm: 2.09 (s, 2H, N-H), 7.31 (d, 4$H_{2,4,5,6}$, 8.63, C-Hpyrimidine), 7.96 (d, 6$H_{aa,a'a',2'2'}$, 8.74, Ar-H), 8.33 (m, 2$H_{1'1'}$, Ar-H), 8.42 (m, 2$H_{bb,b'b'}$, Ar-H). Mass spectrum m/z: 431.4 $[C_{22}H_{18}N_6O_4]^{++}$, 401.4 $[C_{22}H_{19}N_5O_3]^{++}$, 355.4 $[C_{22}H_{19}N_4O]^{++}$, 279.4 $[C_{16}H_{16}N_4O]^{+}$, 163.3 $[C_{10}H_{13}N_2]^{+}$.

5-Nitro-2,4,6-tris(2-nitrophenyl) hexahydro pyrimidine (1b)

2-Nitrobenzaldehyde (0.03mol; 3.18g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 84% yield and a melting point of 242 °C; $C_{22}H_{18}N_6O_8$. FT-IR (ATR) (ν, cm^{-1}): 3111.6 (N-H), 1519.8 & 1344 (-NO_2), 3087.1 (sp^2-C-H), 3036 (sp^3-C-H), 1551.9 & 1443 (C=C). ^{1}H NMR (DMSO-d_6; 500MHz), δ, ppm: 2.10 (s, 2H, N-H), 5.09 (m, H_6, C-$H_{pyrimidine}$), 5.27 (dd, H_4, (3.53, 13.35) C-$H_{pyrimidine}$), 5.66 (m, H_5, C-$H_{pyrimidine}$), 5.83 (m, 2$H_{c,c'}$, (8.74), Ar-H), 6.24 (m, $H_{6'}$, Ar-H), 6.34 (t, H_2, (7.97), Ar-H), 7.77 (m, $H_{2'5',dd',ee'}$, Ar-H), 8.59 (s, $H_{3'}$, Ar-H), 8.76 (d, $H_{ff'}$, Ar-H). Mass spectrum m/z: 494.4 $[C_{22}H_{18}N_6O_8]^{++}$, 478.4 $[C_{22}H_{18}N_6O_7]^{+}$, 431.4 $[C_{22}H_{19}N_6O_4]^{+}$, 298.3 $[C_{16}H_{16}N_3O_3]^{+}$, 135.2 $[C_9H_{13}N]^{+}$, 104.1 $[C_8H_8]^{++}$.

5-Nitro-2,4,6-tris(4-methoxyphenyl) hexahydro pyrimidine (1c)

4-Methoxybenzaldehyde (0.03mol; 4.1g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 76% yield and a melting point of 90 °C; $C_{25}H_{27}N_3O_5$. FT-IR (ATR) (ν, cm^{-1}): 3101.1 (N-H), 1591.6 & 1490.3 (-NO_2), 2931.6 (sp^2-C-H), 2831.6 (sp^3-C-H), 1565.1 & 1423 (C=C), 1211.8 (C-O). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.09 (s, 2H, N-H), 3.84 (s, 9H, -OCH_3), 7.05 (m, 4$H_{2,4,5,6}$, C-$H_{pyrimidine}$), 7.84 (m, 6Hc,$c_{,3'}$, Ar-H), 8.12 (m, 6$H_{2',b,b'}$, Ar-H). Mass spectrum m/z: 432.4 $[C_{24}H_{22}N_3O_5]^{\bullet++}$, 358.4 $[C_{22}H_{18}N_2O_3]^{\bullet++}$, 233.2 $[C_{16}H_{13}N_2]^{\bullet++}$, 159.2 $[C_{10}H_{11}N_2]^{\bullet++}$, 132.1 $[C_9H_{10}N]^{\bullet+}$, 90.1 $[C_7H_6]^{\bullet+}$, 505.6 $[C_{35}H_{27}N_3O]^{\bullet+++}$.

5-Nitro-2,4,6-tris(4-chlorophenyl) hexahydro pyrimidine (1d)

4-Chlorobenzaldehyde (0.03mol; 4.2g), ammonium acetate (0.02mol; 1.54g), and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 83% yield and a melting point of 162 °C; $C_{22}H_{18}C_{l3}N_3O_2$. FT-IR (ATR) (ν, cm^{-1}): 3323.5 (N-H), 1550.1 & 1370.3 (-NO_2), 2977 (sp^2-C-H), 2900 (sp^3-C-H), 1508 (C=C), 834 (C-Cl). ^{1}H NMR (DMSO-d_6; 500MHz), δ, ppm: 2.96 (t, 2H, (9.53), N-H), 4.55 (t, 2$H_{4,6}$, (9.87), C-$H_{pyrimidine}$), 4.96 (d, 2$H_{2,5}$, (13.90), C-$H_{pyrimidine}$), 7.46 (m, 10$H_{bb',cc',3'}$, Ar-H), 7.61 (d, 2$H_{2'}$, (8.52), Ar-H). Mass spectrum m/z: 398.3 $[C_{22}H_{21}Cl_2N_3]^{++}$, 289.7 $[C_{15}H_{14}ClN_2O_2]^{\bullet+}$, 262.2 $[C_{16}H_{12}N_3O]^{++}$, 138.5 $[C_8H_7Cl]^{\bullet+}$, 76.1 $[C_6H_4]^{\bullet++}$.

5-Nitro-2,4,6-tris(4-florophenyl) hexahydro pyrimidine (1e)

4-Florobenzaldehyde (0.03mol; 3.7g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 78% yield and a melting point of 192 °C; $C_{22}H_{18}F_3N_3O_2$. FT-IR (ATR) (ν, cm^{-1}): 3306.1 (N-H), 1540.1 & 1406.8 (-NO_2), 2979 (sp^2-C-H), 2902.6 (sp^3-C-H), 1480 (C=C), 825.7 (C-F). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 1.24 (s, 2H, N-H), 1.07 (d, 2$H_{4,6}$, 8.50, C-$H_{pyrimidine}$), 2.10 (m, H_5, C-$H_{pyrimidine}$), 7.20 (m, 5$H_{cc',e,e',2}$, Ar-H;C-$H_{pyrimidine}$), 7.36 (t, 2$H_{3',5'}$, 8.86, Ar-H), 7.51 (ddd, 2$H_{b',f'}$, (3.51,5.56,8.81), Ar-H), 7.63 (dd, H_b, (5.56,8.61), Ar-H), 7.97 (m, H_f, Ar-H), 8.20 (dd, $H_{2',6'}$, Ar-H). Mass spectrum m/z: 411.3 $[C_{22}H_{16}F_3N_3O_2]^{++}$, 389.3 $[C_{22}H_{13}F_2N_3O_2]^{\bullet+++}$, 350.3 $[C_{22}H_{12}N_3O_2]^{\bullet+++}$, 227.2 $[C_{14}H_{12}FN_2]^{\bullet+++}$, 122.1 $[C_7H_{10}N_2]^{\bullet+++}$.

5-Nitro-2,4,6-tris(4-hydroxy-3-methoxyphenyl) hexahydro pyrimidine (1f)

4-hydroxy-3-methoxybenzaldehyde (0.03mol; 4.56g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 71% yield and a melting point of 170 °C; $C_{25}H_{27}N_3O_8$. FT-IR (ATR) (ν, cm^{-1}): 3112.2 (N-H), 3464 (O-H), 1595.3 & 1477.6 (-NO_2), 2978.9 (sp^2-C-H), 2908.8 (sp^3-C-H), 1512.4 & 1423.9 (C=C), 1243.7 (C-O). ^{1}H NMR (DMSO-d_6; 500MHz), δ, ppm: 2.09 (s, 2H, N-H), 10.05 (s, H, O-H), 3.84 (s, 9H, -OCH_3), 6.86 (d, $2H_{4,6}$, (8.16) C-$H_{pyrimidine}$), 7.31 (dd, $3H_{5'e,,e'}$, Ar-H), 7.49 (d, $2H_{2,5}$, (2.01), C-$H_{pyrimidine}$), 8.03 (m, $3H_{2',b,b'}$, Ar-H), 8.17 (m, $H_{6',f,f'}$, Ar-H). Mass spectrum m/z: 195.2 $[C_{10}H_{15}N_2O_2]^{\bullet+++}$, 148.2 $[C_9H_{12}N_2]^{++}$, 133.1 $[C_8H_9N_2]^{++}$, 88.1 $[C_7H_4]^{\bullet+}$.

5-Nitro-2,4,6-tris(2,4-chlorophenyl) hexahydro pyrimidine (1g)

2,4-Clorobenzaldehyde (0.03mol; 5.2g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 94% yield and a melting point of 200 °C; $C_{22}H_{15}Cl_6N_3O_2$. FT-IR (ATR) (ν, cm^{-1}): 3081.1 (N-H), 1550.1 & 1381.3 (-NO_2), 2918 (sp^2-C-H), 2870 (sp^3-C-H), 1582 & 1466.6 (C=C), 862.6 (C-Cl). ^{1}H NMR (DMSO-d_6; 500MHz), δ, ppm: 2.09 (s, 2H, N-H), 5.67 (dd, $2H_{4,6}$, (8.03;32.48) C-$H_{pyrimidine}$), 6.30 (t, H_5, (10.37), C-$H_{pyrimidine}$), 7.54 (m, $7H_{2,3',5',cc',ee'}$, Ar-H), 7.90 (d, $H_{6'}$, (8.52), Ar-H), 8.62 (d, $H_{f,f'}$, (20.61), Ar-H). Mass spectrum m/z: 502.6 $[C_{22}H_{19}Cl_5N_3]^{\bullet+++}$, 359.8 $[C_{22}H_{18}ClN_3]^{\bullet+}$, 298.5 $[C_{14}H_{10}Cl_3N_3]^{\bullet+}$, 159.2 $[C_{10}H_{11}N_2]^{\bullet+++}$.

5-Nitro-2,4,6-tris(4-methylphenyl) hexahydro pyrimidine (1h)

4-Methylbenzaldehyde (0.03mol; 3.5g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 73% yield and a melting point of 80 °C; $C_{25}H_{27}N_3O_2$. FT-IR (ATR) (ν, cm^{-1}): 3300.8 (N-H), 1540.2 & 1253.7 (-NO_2), 2978.9 (sp^2-C-H), 2905.3 (sp^3-C-H), 1444.9 (C=C). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.31 (s, 2H, N-H), 2.29 (s, 9H, -CH_3), 4.45 (t, $2H_{4,6}$, (10.25) C-$H_{pyrimidine}$), 4.93 (m, $2H_{2,5}$, C-$H_{pyrimidine}$), 7.49 (m, $6H_{3',c,c'}$, Ar-H), 7.33 (d, $4H_{,b,b'}$, (8.05), Ar-H), 8.47 (d, $2H_{2'}$, Ar-H). Mass spectrum m/z: 338.3 $[C_{22}H_{16}N_3O]^{\bullet+++}$, 236.3 $[C_{16}H_{16}N_2]^{\bullet+++}$, 118.1 $[C_8H_8N]^{\bullet+}$.

5-Nitro-2,4,6-tris(4-pyridine) hexahydro pyrimidine (1i)

4-Pyridinecarboxaldehyde (0.03mol; 3.2g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 84% yield and a melting point of 300 °C; $C_{19}H_{18}N_6O_2$. FT-IR (ATR) (ν, cm^{-1}): 3192.4 (N-H), 1588.6 & 1407.7 (-NO_2), 2979.2 (sp^2-C-H), 2901 (sp^3-C-H), 1488.9 (C=C), 1252.3 & 1222.8 (C=N). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.09 (s, 2H, N-H), 7.56 (m, $10H_{2,4,5,6,cc,c'c'}$, Ar-H; C-$H_{pyrimidine}$), 8.03 (m, $4H_{bb,b'b'}$, Ar-H), 8.71 (m, $2H_{2'2'}$, Ar-H). Mass spectrum m/z: 298.3 $[C_{15}H_{16}N_5O_2]^{\bullet+++}$, 271.3 $[C_{14}H_{15}N_4O_2]^{\bullet+}$, 167.2 $[C_8H_{13}N_3O]^{++}$.

5-Nitro-2,4,6-tris(4-bromophenyl) hexahydro pyrimidine (1j)

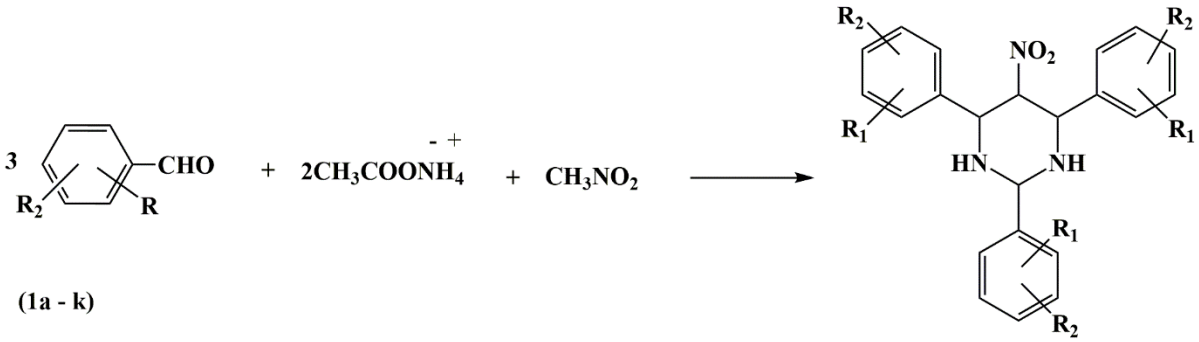
4-Bromobenzaldehyde (0.03mol; 5.5g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 69% yield and a melting point of 180 °C; $C_{22}H_{18}Br_3N_3O_2$. FT-IR (ATR) (ν, cm^{-1}): 3044.2 (N-H), 1550.3 & 1363.6 (-NO_2), 2946.6 (sp^2-C-H), 2900 (sp^3-C-H), 1699.5 & 1484.9 (C=C), 820.5 (C-Br). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.10 (s, 2H, N-H), 7.55 (m, $10H_{2,4,5,6,2',3',bb,b'b'cc,c'c'}$, Ar-H; C-$H_{pyrimidine}$). Mass spectrum m/z: 593.0 $[C_{22}H_{15}Br_3N_3O_2]^{\bullet+++}$, 546 $[C_{22}H_{14}Br_3N_2]^{\bullet+++}$, 390.3 $[C_{22}H_{18}BrN_2]^{\bullet+++}$, 352 $[C_{14}H_{10}Br_2N]^{\bullet+}$, 183.2 $[C_{13}H_{13}N]^{\bullet+}$.

5-Nitro-2,4,6-tris(2-chloro-4-florophenyl) hexahydro pyrimidine (1k)

2-Chloro-4-bromobenzaldehyde (0.03mol; 4.7g), ammonium acetate (0.02mol; 1.54g) and nitromethane (0.61 g, 0.01 mol) were combined to yield (1a). Yellow colour powder with an 92% yield and a melting point of 216 °C; $C_{22}H_{15}Cl_3F_3N_3O_2$. FT-IR (ATR) (ν, cm^{-1}): 3081.6 (N-H), 1551.3 & 1370.5 (-NO_2), 3046.4 (sp^2-C-H), 2946 (sp^3-C-H), 1596.8 & 1485.6 (C=C), 859.5 (C-Cl), 817.7 (C-F). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 8.45 (s, 2H, N-H), 5.36 (d, $2H_{4,6}$, (7.72) C-$H_{pyrimidine}$), 6.15 (t, H_5, (7.75), C-$H_{pyrimidine}$), 7.26 (t, $2H_{e,e'}$, (8.45) Ar-H), 7.37 (d, $2H_{2,5'}$, (5.28), Ar-H; C-$H_{pyrimidine}$), 7.47 (d, $2H_{c,c'}$, (6.19), Ar-H), 7.56 (d, $H_{3'}$, (6.28), Ar-H), 7.68 (m, $2H_{f,f'}$, Ar-H), 8.04 (m, $H_{6'}$, Ar-H). Mass spectrum m/z: 515.7 $[C_{22}H_{14}Cl_3F_3N_3O_2]^{+}$, 454.7 $[C_{22}H_{13}Cl_3F_3N_3]^{\bullet++}$, 298.3 $[C_{21}H_{18}N_2]^{\bullet+++}$, 204.2 $[C_{15}H_{10}N]^{\bullet+}$, 143.1 $[C_{10}H_9N]^{\bullet+}$.

III. RESULTS AND DISCUSSION

The Mannich reaction was used to create the 5-Nitro-2,4,6-tris(phenyl) hexahydro pyrimidine derivatives (1a-k) (Scheme 1). Shukkur's research was used to derive these starting materials (Hamed, 2020). The yields of the reactions ranged from good to excellent depending on the substituted group on the phenyl rings, while the melting points of the prepared derivatives with different substituted groups, as well as the position of the substituted group on the ring (for the same substituted group), showed a large variation. Because of the presence of an electron-withdrawing group on the ring, which decreased the electron density and increased the nucleophilic attack on the carbonyl group of the aldehyde. So that, the yields of the nitro-containing derivatives 1a and 1b were very good (this is true for the pyridine ring.). The yields are clearly lower when compared to the electron-donating groups (1c-OCH_3, 1f-OH -OCH_3, 1h-CH_3). The position of the substituted group, on the other hand, played a significant role in changing the melting points. We can see that the 1b derivative has a higher antagonist point than 1a, and the 1g compound has a higher melting point than 1d. This could be due to the substituted group's proximity to the hexahydro pyrimidine ring and the formation of intramolecular hydrogen bonds during bond movement, which increases the stability of the compounds.



1a= (R_1)-H/(R_2)4-NO_2 , **1b=** (R_1)2-NO_2/(R_2)-H , **1c=** (R_1)-H/(R_2)4-OCH_3
1d= (R_1)-H/(R_2)4-Cl , **1e=** (R_1)-H/(R_2)4-F , **1f=** (R_1)3-OCH_3/(R_2)4-OH
1g= (R_1)2-Cl/(R_2)-4-Cl , **1h=** (R_1)-H/(R_2)4-CH_3 , **1i=** (R_1)-H/(R_2)4-Py
1j= (R_1)-H/(R_2)4-Br , **1k=** (R_1)2-Cl/(R_2)4-F ,

Scheme 1. The general procedure of 5-nitro-2,4,6-triphenyl hexahydro pyrimidine derivatives synthesis (1a-k)

Characterization of compounds (1a-k)

Fourier-transform infrared spectroscopy (FT-IR)

FT-IR spectra are depicted in Figure 1. The vibrations of the N-H group appear at 3,418.6 cm^{-1} in 1a and 3111.6 cm^{-1} in 1b. Furthermore, the majority of the peaks in the 1b spectrum are significantly or slightly displaced when compared to 1a. Because the positions of nitro groups have changed, the properties of bonds have changed, as have bond vibrations. In 1f's spectrum, the peaks attributed to O-H and N-H were 3,464 cm^{-1} and 3112.2 cm^{-1}, respectively. The C-O bond, on the other hand, vibrated at a frequency of 1243.7 cm^{-1}. It's worth noting that the vibrations in the spectrum of the 1f compound are nearly identical to the vibrations in the spectrum of the 1c compound.

When the phenyl ring's substituent group is halides, the distinct appearance of the amine peak in 1d, 1e, 1g, 1j, and 1k compounds varies at 3323.5, 3306.1, 3081.1, 3044.2, and 3081.6 cm^{-1}, respectively. Furthermore, because of the long bonds between carbon and halide, the vibration of C-X bonds (X is the halide) appears in the low-energy regions of the IR spectrum in the ranges 800-865 cm^{-1}. The IR spectrum of compound 1i, on the other hand, shows the presence of peaks representing the functional groups of the prepared derivative, which are represented by the amine group N-H, the nitro group-NO2, and the imine group at 3192.4, 1588.6 & 1407.7, and 1252.3-1222.8 cm^{-1}, respectively.

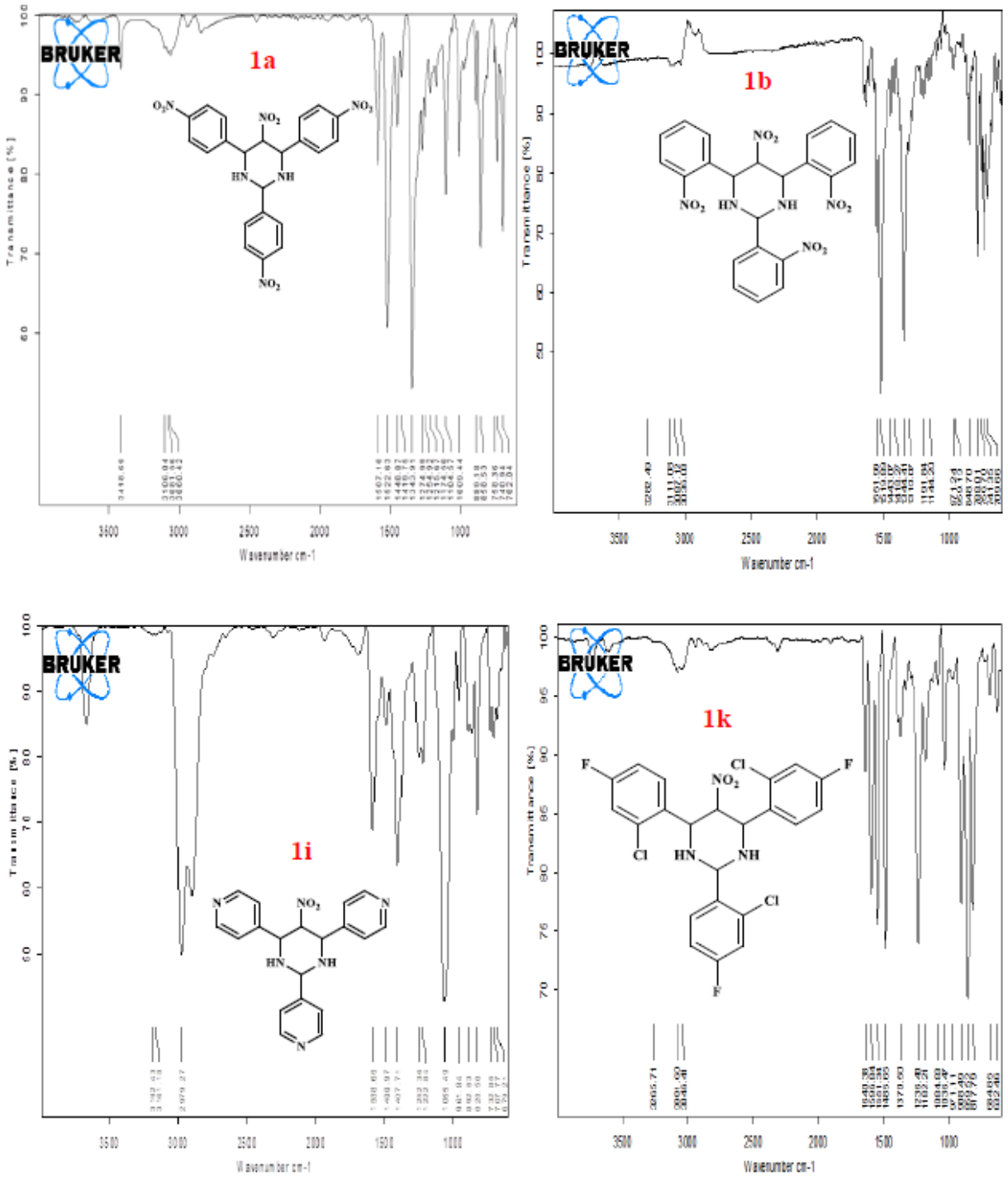


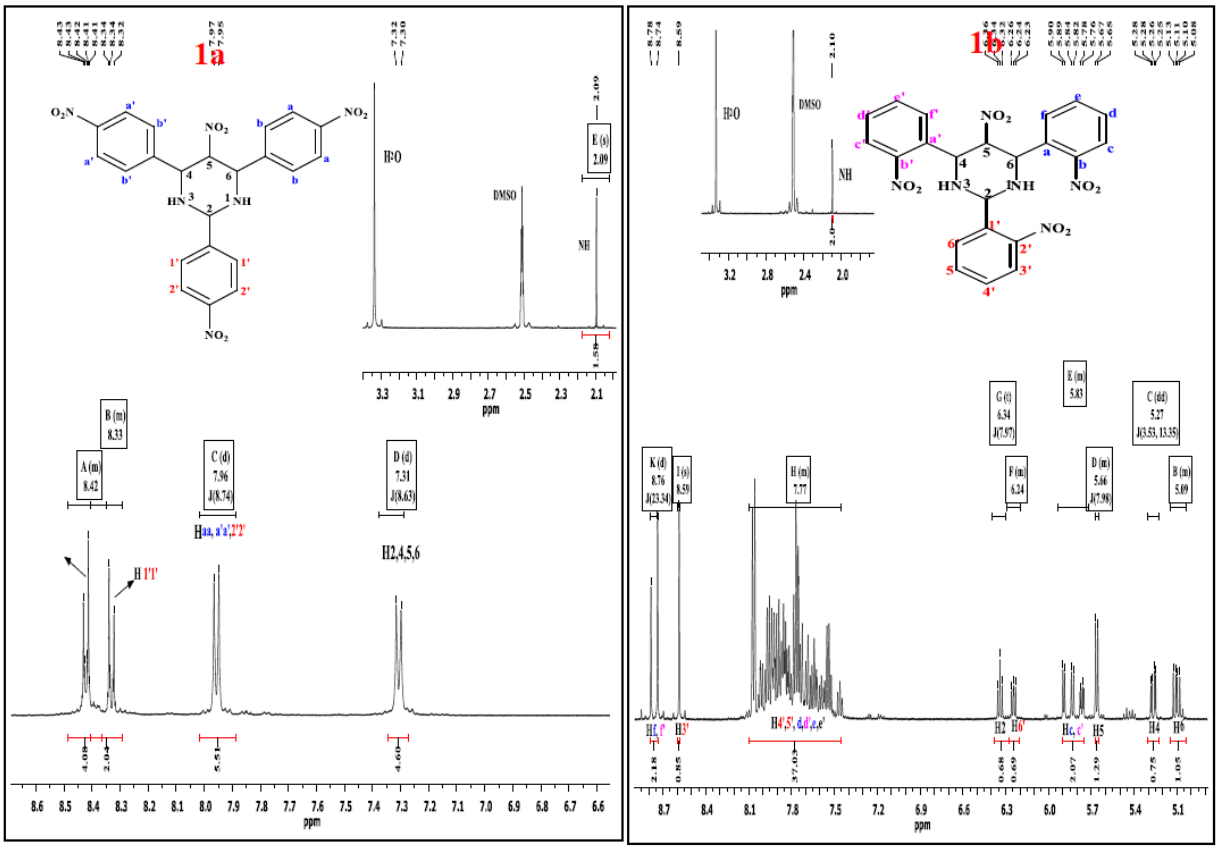
Figure 1. FT-IR spectra for specific compounds (1a, b, i and k)

Proton nuclear magnetic resonance ^{1}H-NMR

^{1}H-NMR spectra (Figure 2) revealed the effect of the compensated aggregates on the hexahydro pyrimidine ring, as well as changes in the shape and location of the apparent proton peaks. The ^{1}H-NMR spectra revealed peaks for the N-H protons mostly in the range of 1.2-3 ppm, with the exception of the 1k compound at 8.45 ppm. We discovered that the withdrawing groups effect of the electronic density of the nitro group causes the peaks to be displaced towards the downfield by spectral tracking of compounds 1a and 1b. Peaks of the compounds 1a $H_{2,4,5,6}$, $H_{aa,a'a',2'2'}$, $H_{1'1'}$, and $H_{bb,b'b'}$ combine in equal environments at 7.31, 7.96, 8.33, and 8.42 ppm, respectively. The presence of the nitro group on the ortho site in the 1b compound, on the other hand, had an effect on the proton environment of the hexahydro pyrimidine and phenyl rings. The environment of the protons in compound 1i, on the other hand, was nearly identical to that of the compounds containing nitro groups. This shows that the nitrogen

atom in the pyridine ring has the same effect on the protons as the nitro group. Furthermore, the electron-donating groups (-OCH3, -CH3, -OH) of (1c, 1f, and 1h) compounds had the opposite effect on the electron-withdrawing groups as the electron-containing compounds. Furthermore, the presence of these groups on the ortho position has resulted in a distinct difference in the environments, but only in the upfield.

Halogen substituents are unique in that they can deactivate while still directing ortho- and para-. This is due to the fact that they both withdraw inductive electrons and donate resonance at the same time (due to their electronegativity) (lone pair donation). We observe the effect of lone electron pairs on halides by entering them into the aromatic ring resonance by tracing the values of the apparent peaks in the compounds containing halides 1d, 1e, 1g, 1i, and 1k. In the 1k compound, increasing the electro density on the phenol rings resulted in the formation of intramolecular hydrogen bonds between the chloro and amine hydrogens (Cl---H-N) in the pyrimidine ring. This configuration has resulted in a lower electronic density on the amine proton N-H and a downfield shift of 8.24 ppm.



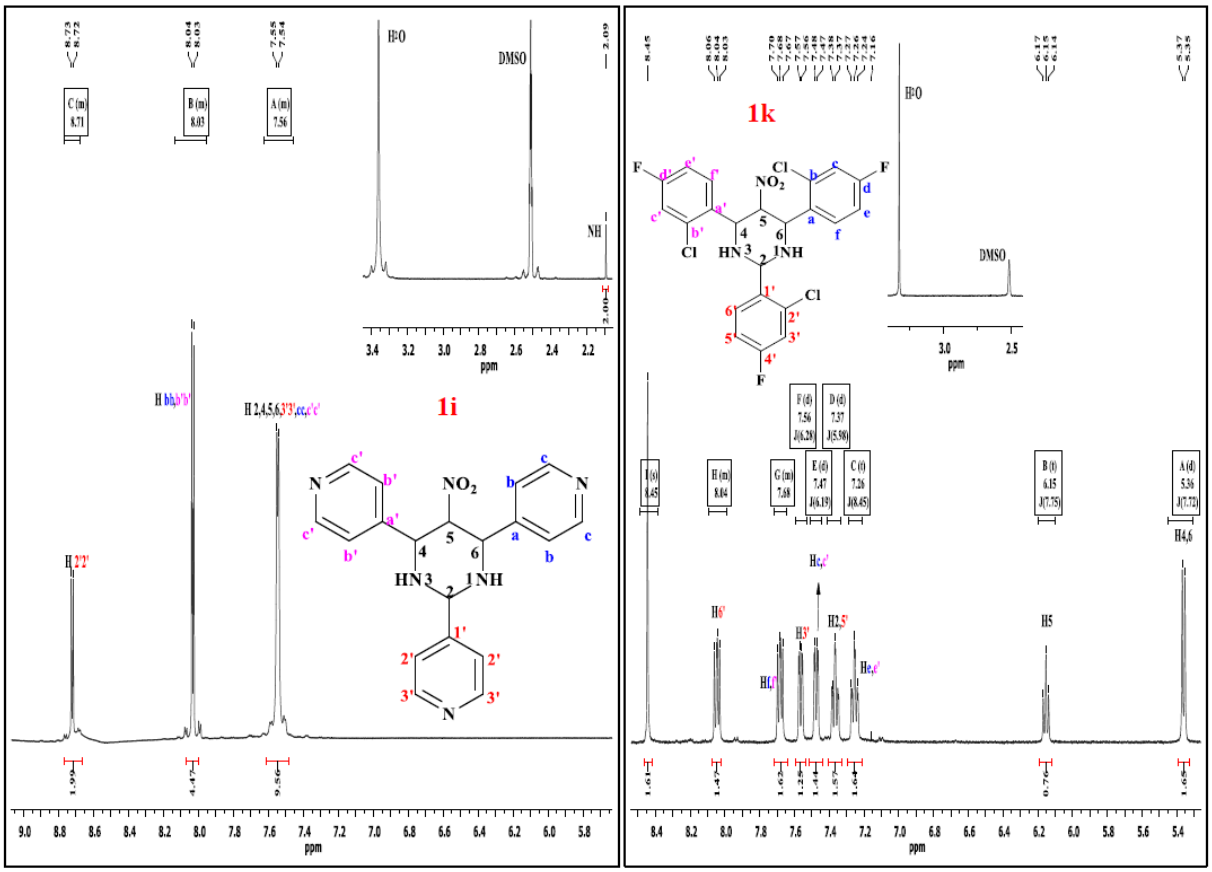


Figure 2. [1]H-NMR spectra for specific compounds (1a, b, i and k)

Mass spectrometry

Figure 3 depicts mass spectra used to compare theoretically calculated molecular masses to all molecular ions with experimentally measured peak m/z [M+]. The structures of the prepared derivatives were confirmed. As previously stated, the position of the substitute group has a clear impact on melting points; however, tracking the MS spectra of nitro derivatives 1a and 1b reveals that this factor plays an important role. The nitro group at the Ortho position increases compound stability by forming a relative abundance peak at 494 in compound 1b, whereas there is no peak at 494 of m/z axis in compound 1a, as shown in Figure 4.

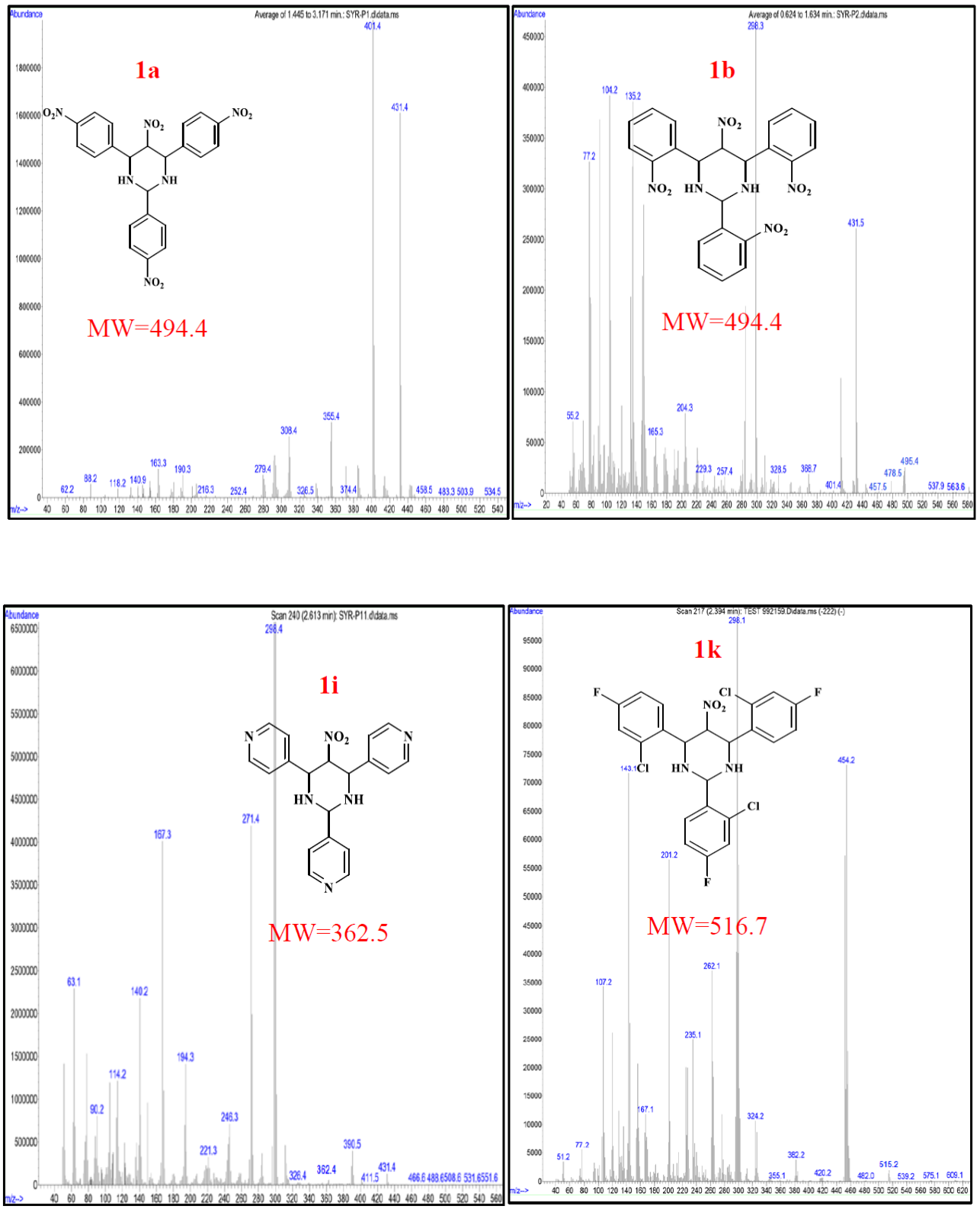


Figure 3. Mass spectra for specific compounds (1a, b, i and k)

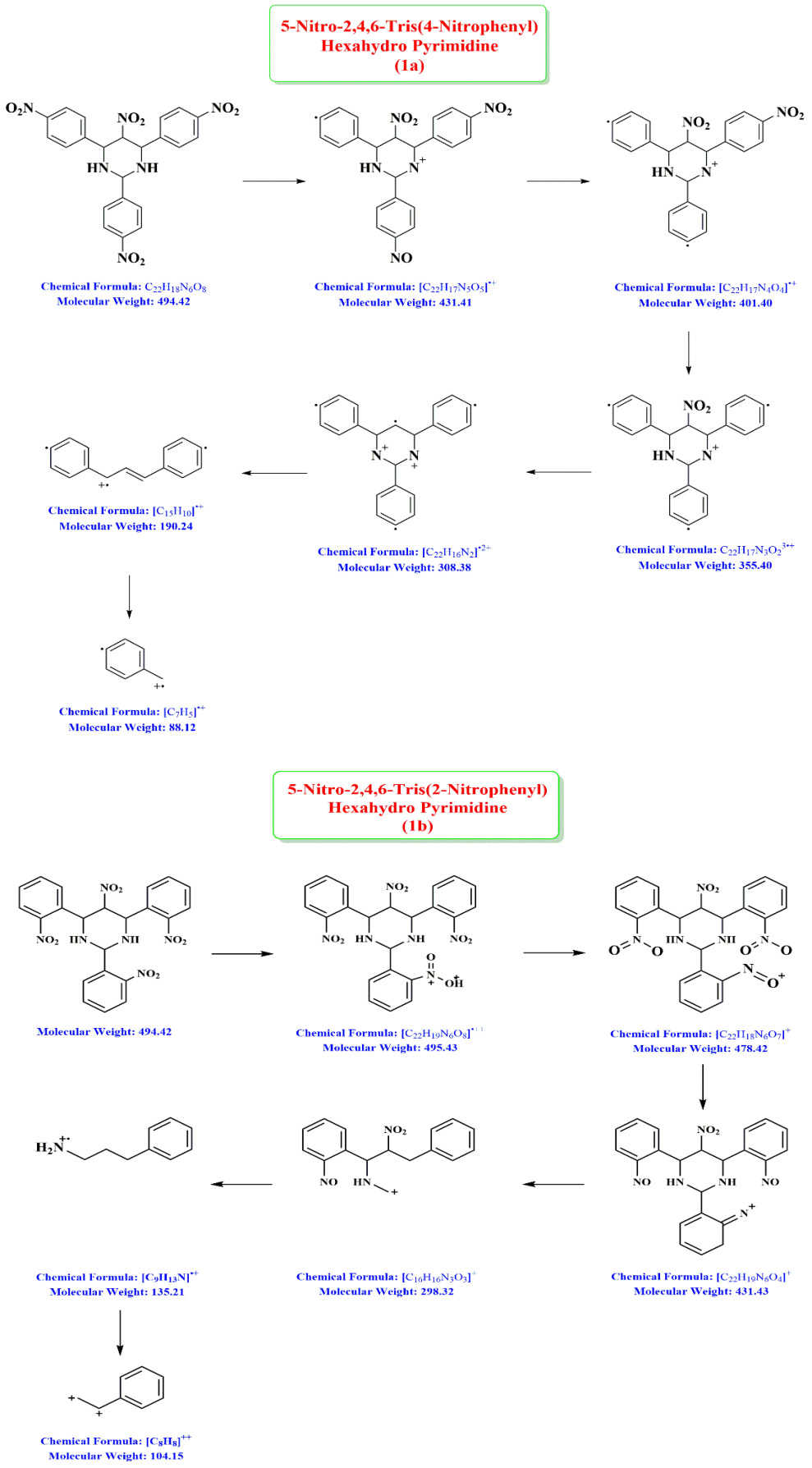


Figure 4. Mass spectrometry fragmentation of 1a and 1b compounds (Electron Impact 70 eV)

IV. CONCLUSION

The eleven new 5-nitro-2,4,6-triphenylhexahydro-pyrimidine derivatives produced good results with yields ranging from good to excellent. According to Mannich's reaction, the reactants went through a condensation reaction. The effect of the substituted groups on the aromatic rings was evident in the reaction route and even in the yield amount. By comparing the melting points of the prepared derivatives, the position of substituted groups played an important role in productivity, stability, and thermal resistance. FTIR, ^{1}H NMR, and mass spectroscopy were used to characterize the products.

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