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A Novel Synthesis, Molecular Structure by X-ray Diffraction of 5-Nitro-2,4,6-Triphenylhexahydropyrimidine, and Some it's Derivatives

Shukkur A. Hamed

Department of Chemistry, College of Education for Pure Science, University of Anbar, Al-Anbar, Iraq
Corresponding author: Tel: +9647902650452
Email: ahmedsatori@uoanbar.edu.iq
ORCID ID: 0000-0003-4471-9191

ABSTRACT
A novel of the title compound and some its derivatives were synthesized in a good to excellent yields of seven derivatives, by condensation of three components: benzaldehyde or some various monosubstituted of its derivatives, ammonium acetate, and nitromethane with ratio 3:2:1 respectively, in presence *n*-butanol, by type Mannich reaction in one–pot. The products were characterized by FTIR, ^{1}H NMR, and Mass spectroscopy. The molecular structure of 5-nitro-2,4,6-triphenylhexahydropyrimidine was affirmed by X-ray crystallography analysis of single-crystal.

Keywords: Condensation, Mannich reaction, nitrohexahydropyrimidine, X-ray crystallography analysis.

Correspondence:
Shukkur A. Hamed
Department of Chemistry, College of Education for Pure Science, University of Anbar, Al-Anbar, Iraq
Corresponding author: Tel: +9647902650452
Email: ahmedsatori@uoanbar.edu.iq
ORCID ID: 0000-0003-4471-9191

INTRODUCTION
Recent research respecting the synthetic molecular design of organic compounds has been concerned with obtaining extended molecular groups by assembling them from two or more small molecules being used as building blocks. Pyrimidine and hydropyrimidine are compounds that have six-membered heterocyclic rings, which contain two nitrogen atoms at 1,3-positions. The classical synthetic pathways for synthesis of derivatives pyrimidine, di-, tetra-, hexahydropyrimidine were occurred by different reactions which involve multicomponent synthesis for example condensation of substituted 1,3-diaminopropane with various aldehydes and ketones, urea/thiourea with chalcones, coupling of guanidine/imidamide with different reagents, cyclization of isocyanate/nitrile derivatives (1–5). Also, some of their derivatives contain nitro groups, such as 5-nitro-1,3-disubstituted hexahydropyrimidine and 5-nitro-1,3-disubstituted-1,2,3,4-tetra-hydropyrimidine, which were synthesized by a condensation reaction type Mannich between the primary nitroalkane or nitroolefin, formaldehyde, and the convenient primary amine (6–9). Pyrimidine and reduced rings are essential fragments in a number of natural remedies and drugs like folic acid, riboflavin, and barbitone (10,11). They exhibit a broad spectrum of pharmacological activities, being particularly effective in terms of their anti-viral, anti-bacterial, anti-fungal, antioxidant and anti-cancer properties (12–16). Moreover, hexahydropyrimidine and many of its derivatives made poly-dentate complexes by nitrogen donor, it's coordinating the transition cations in mono-, di- and poly-coordination form (17–20). Some derivatives of hexahydropyrimidine used as polymer stabilizers (21). Till now, the similar structures of synthesized compounds are not available in the CCDC database survey. So, in this work, we synthesized novel compounds of 5-nitro-2,4,6-triphenylhexahydropyrimidine and its derivatives which allowing new applications. The molecular structure was confirmed by using spectroscopy methods and X-ray crystallography analysis.

MATERIALS AND METHODS
All materials were supplied by Merck Co. (Germany) and Romil Co. (UK). Melting points were measured by the Stuart (SMP 30) apparatus. FTIR, ^{1}H NMR and Mass spectra were recorded on Bruker Tensor-27 as (ATR) technique, Bruker 500 MHz- Avance III (DMSO-d6 as a solvent, TMS as internal standard) and 5973 Network Mass Selective Detector (Electron Impact 70 eV) spectrometer, respectively, while X-ray diffraction of single-crystal was measured by using a Stoe Stadi Vari Pilatus 100 K diffractometer.

General procedure for synthesis 5-nitro-2,4,6-trisubstituted phenylhexahydro-pyrimidine (1a-g)
Benzaldehyde or monosubstituted its derivatives 0.03 mol, ammonium acetate 0.02 mol and nitromethane 0.01 mol, were mixed in 15 mL *n*-BuOH, then the mixture was refluxed at 90 °C with stirring for 15-75 min, until a turbid solution was formed. The mixture was left at room temperature for 12 hours. The precipitate was filtered, and the filtrate was vaporized in low pressure and collected as crystals, the crude product was re-crystallized from toluene or benzene.

Properties and characterization of synthesized compounds 1a-g

5-nitro-2,4,6-triphenylhexahydropyrimidine (1a)
The reaction mixture of (3.18 g, 0.03 mol) benzaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1a). White colour powder yield 2.9 g, 86%; mp 198–199 °C. $C_{22}H_{21}N_3O_2$. FTIR (ATR) (ν, cm^{-1}): 3307 (N—H), 2983 (C—H $_{aromatic}$), 2903 (C—H $_{aliphatic}$), 1536, 1364 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.80 (t, 2H$_{1,3}$, N—H), 4.53 (t, 2H$_{4,6}$, C—H), 4.99, 5.01 (dd, 2H$_{2,5}$, J^2=10 Hz, C—H), 7.31–7.61 (m, 15H,

3(Ar—H ring)). Mass spectrum *m/z*: 358 $[C_{22}H_{20}N_3O_2]^+$, 312 $[C_{22}H_{20}N_2]^{++}$, 282 $[C_{16}H_{16}N_3O_2]^+$, 254 $[C_{15}H_{14}N_2O_2]^{++}$, 208 $[C_{10}H_{14}N_3O_2]^+$, 194 $[C_9H_{12}N_3O_2]^+$, 106 $[C_7H_8N]^+$, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$.

The single crystal was obtained by allowing the saturated dry toluene solution of compound 1a to stand for five days at room temperature. The details of X-ray crystallographic data were shown in Tables 1–5.

4,4',4''-(5-nitrohexahydropyrimidine-2,4,6-triyl) tris (N, N-dimethylaniline) (1b)

The reaction mixture of (4.47 g, 0.03 mol) 4-(dimethylamino) benzaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1b). Deep red colour powder yield 3.5 g, 72%; mp 183–184 °C. $C_{28}H_{36}N_6O_2$. FTIR (ATR) (ν, cm^{-1}): 3104 (N—H), 2982 (C—H $_{aromatic}$), 2905 (C—H $_{aliphatic}$), 1529, 1314 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.99 (s, $2H_{1,3}$, N—H), 3.0 (s, 18H, $3(CH_3)_2$), 3.07 (s, $2H_{4,6}$, C—H), 3.32 (s, $2H_{2,5}$, C—H), 6.73–8.03 (dd,dd, J^4=10 Hz, 12H, 3(Ar—H ring)). Mass spectrum m\z: 488 $[C_{28}H_{36}N_6O_2]^+$, 442 $[C_{28}H_{36}N_5]^+$, 368 $[C_{20}H_{26}N_5O_2]^+$, 239 $[C_{16}H_{19}N_2]^+$, 192 $[C_{11}H_{18}N_3]^+$, 134 $[C_9H_{12}N]^+$, 120 $[C_8H_{10}N]^+$, 105 $[C_8H_9]^+$, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$.

2,4,6-tris(2-bromophenyl)-5-nitrohexahydropyrimidine (1c)

The reaction mixture of (5.55 g, 0.03 mol) 2-bromobenzaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1c). White colour powder yield 4.4 g, 74%; mp 188–189 °C. $C_{22}H_{18}Br_3N_3O_2$. FTIR (ATR) (ν, cm^{-1}): 3308 (N—H), 2982 (C—H $_{aromatic}$), 2903 (C—H $_{aliphatic}$), 1545, 1333 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 3.37 (t, $2H_{1,3}$, N—H), 5.15 (t, $2H_{4,6}$, C—H), 5.52 (t, $2H_{2,5}$, C—H), 7.33–8.15 (m,d, 12H, 3(Ar—H ring)). Mass spectrum m\z: 595 $[C_{22}H_{17}Br_3N_3O_2]^+$, 551 $[C_{22}H_{18}Br_3N_2]^+$, 368 $[C_{15}H_{14}Br_2N]^+$, 276 $[C_9H_{15}BrN_3O_2]^+$, 236 $[C_{16}H_{16}N_2]^{++}$, 165 $[C_8H_9N_2O_2]^+$, 140 $[C_7H_{10}NO_2]^+$, 106 $[C_7H_8N]^+$, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$.

2,4,6-tris(2-methoxyphenyl)-5-nitrohexahydropyrimidine (1d)

The reaction mixture of (4.10 g, 0.03 mol) 2-methoxybenzaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1d). White colour powder yield 4.1 g, 80%; mp 169–170 °C. $C_{25}H_{27}N_3O_5$. FTIR (ATR) (ν, cm^{-1}): 3345 (N—H), 2986 (C—H $_{aromatic}$), 2903 (C—H $_{aliphatic}$), 1533, 1363 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 3.38 (t, $2H_{1,3}$, N—H), 3.94 (d, 9H, J^2=15 Hz, $3CH_3$), 4.54 (t, $2H_{4,6}$, C—H), 4.84 (t, $1H_5$, C—H), 5.51 (t, $1H_2$, C—H), 6.88–7.37 (m, 12H, 3(Ar—H ring)). Mass spectrum m\z: 448 $[C_{25}H_{26}N_3O_5]^+$, 403 $[C_{25}H_{27}N_2O_3]^+$, 342 $[C_{18}H_{20}N_3O_4]^+$, 314 $[C_{17}H_{18}N_2O_4]^{++}$, 268 $[C_{17}H_{20}N_2O]^{++}$, 254 $[C_{16}H_{18}N_2O]^{++}$, 179 $[C_9H_9NO_3]^{++}$, 134 $[C_8H_8NO]^+$, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$.

2,4,6-tris(3-methoxyphenyl)-5-nitrohexahydropyrimidine (1e)

The reaction mixture of (4.10 g, 0.03 mol) 3-methoxybenzaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1e). White colour powder yield 3.8 g, 75%; mp 158–159 °C. $C_{25}H_{27}N_3O_5$. FTIR (ATR) (ν, cm^{-1}): 3310 (N—H), 2972 (C—H $_{aromatic}$), 2903 (C—H $_{aliphatic}$), 1542, 1372 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 2.76 (t, $2H_{1,3}$, N—H), 3.75 (d, 9H, J^2=15 Hz, $3CH_3$), 4.47 (t, $2H_{4,6}$, C—H), 4.94 (t, $2H_{2,5}$, C—H), 6.87–7.27 (m,s, 12H, 3(Ar—H ring)). Mass spectrum m\z: 448 $[C_{25}H_{26}N_3O_5]^+$, 403 $[C_{25}H_{27}N_2O_3]^+$, 342 $[C_{18}H_{20}N_3O_4]^+$, 314 $[C_{17}H_{18}N_2O_4]^{++}$, 268 $[C_{17}H_{20}N_2O]^{++}$, 254 $[C_{16}H_{18}N_2O]^{++}$, 179 $[C_9H_9NO_3]^{++}$, 134 $[C_8H_8NO]^+$, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$.

5-nitro-2,4,6-tris(3-nitrophenyl) hexahydropyrimidine (1f)

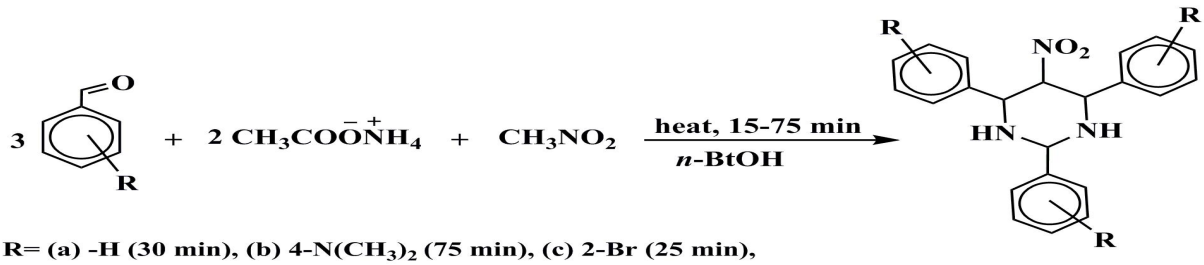
The reaction mixture of (4.53 g, 0.03 mol) 3-nitrobenzaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1f). White colour powder yield 4.5 g, 91%; mp 160–161 °C. $C_{22}H_{18}N_6O_8$. FTIR (ATR) (ν, cm^{-1}): 3090 (N—H), 2964 (C—H $_{aromatic}$), 2904 (C—H $_{aliphatic}$), 1515, 1345 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 1.23 (s, $2H_{1,3}$, N—H), 3.28 (t, $2H_{4,6}$, C—H), 3.36 (s, $1H_5$, C—H), 6.37 (s, $1H_2$, C—H), 7.73–8.89 (m,s, 12H, 3(Ar—H ring)). Mass spectrum m\z: 494 $[C_{22}H_{18}N_6O_8]^+$, 434 $[C_{22}H_{18}N_4O_6]^+$, 315 $[C_{15}H_{15}N_4O_4]^+$, 299 $[C_{15}H_{13}N_3O_4]^+$, 286 $[C_{14}H_{12}N_3O_4]^+$, 237 $[C_{16}H_{17}N_2]^+$, 208 $[C_{10}H_{14}N_3O_2]^+$, 150 $[C_8H_8NO_2]^+$, 136 $[C_7H_6NO_2]^+$, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$.

5-nitro-2,4,6-tri(thiophen-2-yl) hexahydropyrimidine (1g)

The reaction mixture of (3.36 g, 0.03 mol) thiophene-2-carbaldehyde, (1.54 g, 0.02 mol) ammonium acetate and (0.61 g, 0.01 mol) nitromethane gave (1g). White colour powder yield 3.1 g, 82%; mp 164–165 °C. $C_{16}H_{15}N_3O_2S_3$. FTIR (ATR) (ν, cm^{-1}): 3108 (N—H), 2982 (C—H $_{aromatic}$), 2903 (C—H $_{aliphatic}$), 1545, 1335 (–NO_2). ^{1}H NMR (500 MHz, DMSO-d_6), δ, ppm: 3.32 (s, $2H_{1,3}$, N—H), 3.40 (s, $2H_{4,6}$, C—H), 5.29, 5.34 (dd, $1H_5$, J^2=5 Hz, C—H), 5.60 (t, $1H_2$, C—H), 6.96–8.44 (m,d, 9H, *J*=10 Hz, 3(thiophenyl ring)). Mass spectrum m\z: 376 $[C_{16}H_{14}N_3O_2S_3]^+$, 280 $[C_{12}H_{12}N_2O_2S_2]^{++}$, 250 $[C_{12}H_{14}N_2S_2]^+$, 171 $[C_6H_7N_2O_2S]^+$, 140 $[C_7H_{10}NS]^+$, 111 $[C_6H_7S]^+$, 97 $[C_5H_5S]^+$, 69 $[C_5H_9]^+$.

RESULTS AND DISCUSSION

Scheme 1 revealed the route of the synthesis of 5-nitro-2,4,6-triphenylhexahydropyrimidine and some of its derivatives (1a-g), which was easily synthesized in good to excellent yields of seven derivatives by type Mannich reaction in a single pot via condensation reaction of three components: benzaldehyde or some different monosubstituted its derivatives, ammonium acetate, and nitromethane with 3:2:1 respectively, in presence n-BuOH. From the obtained results it is obvious when comparing the effect of substituted groups on phenyl (e.g. –NO_2 or –$N(CH_3)_2$) the compound containing a strong electron-withdrawing group (EWG, –NO_2) was the faster reaction and gave the greater yield than other compounds, this is due to the nitro group has (–I) inductive effect that increases electrophilic center of the carbon ($^{+\delta}$C) carbonyl group. Hence, increase condensation reaction by the attack a nucleophile toward the carbaldehyde group. The products were characterized by FTIR, ^{1}H NMR, and Mass spectroscopy. The molecular structure of 5-nitro-2,4,6-triphenylhexahydropyrimidine was confirmed by X-ray crystallography analysis of single-crystal.



R= (a) -H (30 min), (b) 4-N(CH_3)$_2$ (75 min), (c) 2-Br (25 min), (d) 2-OCH_3 (40 min), (e) 3-OCH_3 (50 min), (f) 3-NO_2 (15 min), (g) C_4H_4S (thiophenyl) (20 min).

Scheme 1. Synthesis of 5-nitro-2,4,6-triphenylhexahydropyrimidine and its derivatives (1a-g)

Characterization of compounds (1a-g)

Some of the selected FTIR, ^{1}H NMR, and Mass spectra of compounds 1a-g are shown in Figs. 1–3. FTIR spectra of the compounds were characteristic of stretching vibration band (weak) at extent 3090–3345 cm^{-1} which attributed to (N—H) group, besides vibration bands at extents 1515–1545 cm^{-1} (asymmetric, strong) and 1314–1372 cm^{-1} (symmetric, medium) assigned to (–NO_2) group. While appeared stretching bands at extents 2964–2986, 2903–2905 cm^{-1} which attributed to (C—$H_{aromatic}$), and (C—$H_{aliphatic}$), respectively. In ^{1}H NMR spectra, it was observed different signals due to the present variety of substituted groups, appearance three distinct pure signals: triplet or singlet; triplet or singlet; and doublet-doublet or triplet or singlet at ranges δ= 1.23–3.38 ppm; 3.07–5.15 ppm; and 3.32–6.37 ppm, which attributed to protons for groups ($H_{1,3}$, 2N–H), ($H_{4,6}$, 2C–H), and ($H_{2,5}$, 2C–H), respectively. whilst the signals for aromatic protons of three rings were observed as multiplet, singlet or multiplet, doublet signals at range δ= 6.73–8.89 ppm. Moreover, the structures of prepared compounds were confirmed by comparing all molecular ions peaks *m/z* [M$^+$] of mass spectra with conformity their theoretical molecular masses (Some selected peaks of compounds fragments which have a high abundance were showed in the paragraph of properties and characterization of synthesized compounds).

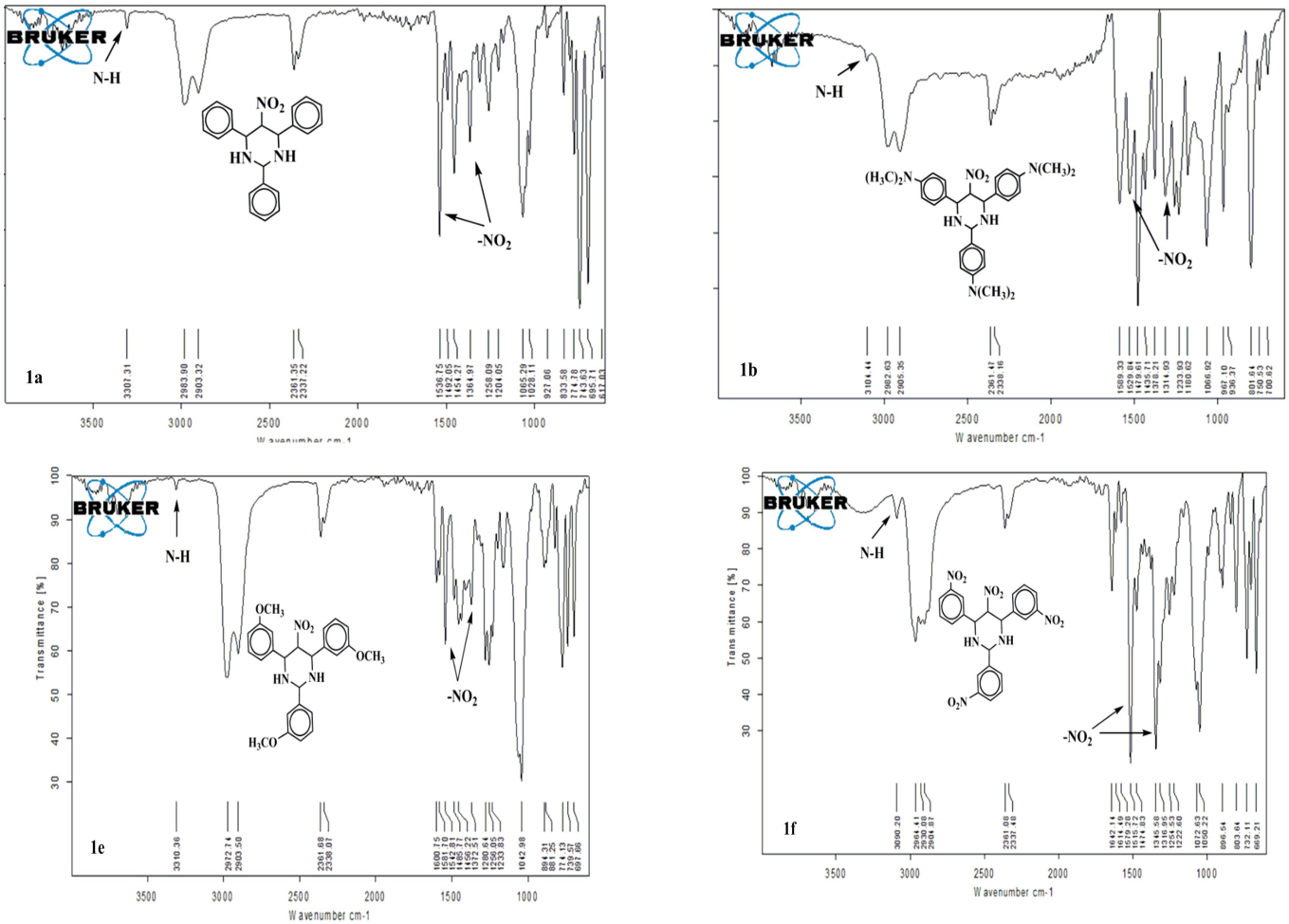


Figure 1. Some of the selected FTIR spectra for the compounds (1a, b,e and f)

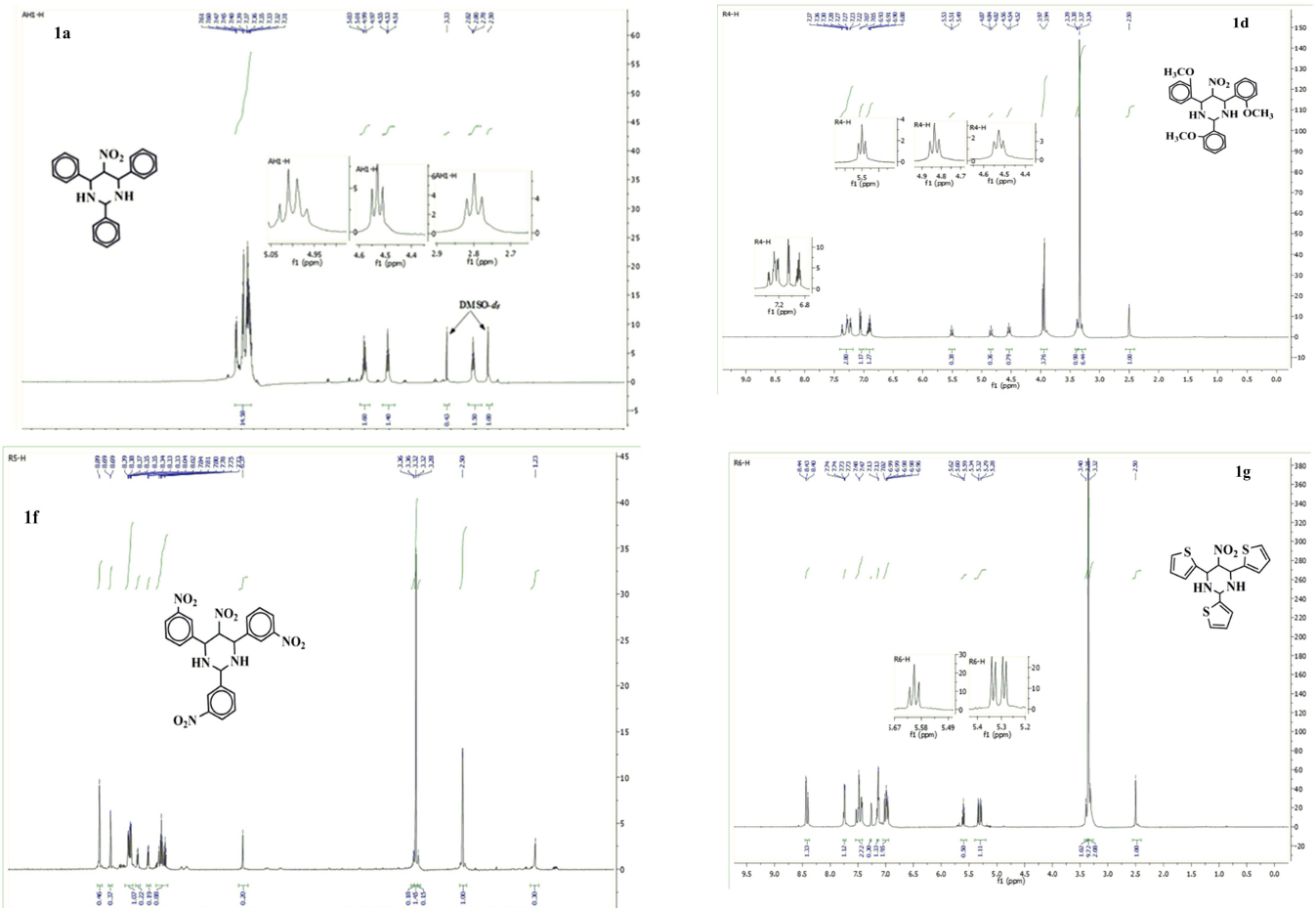
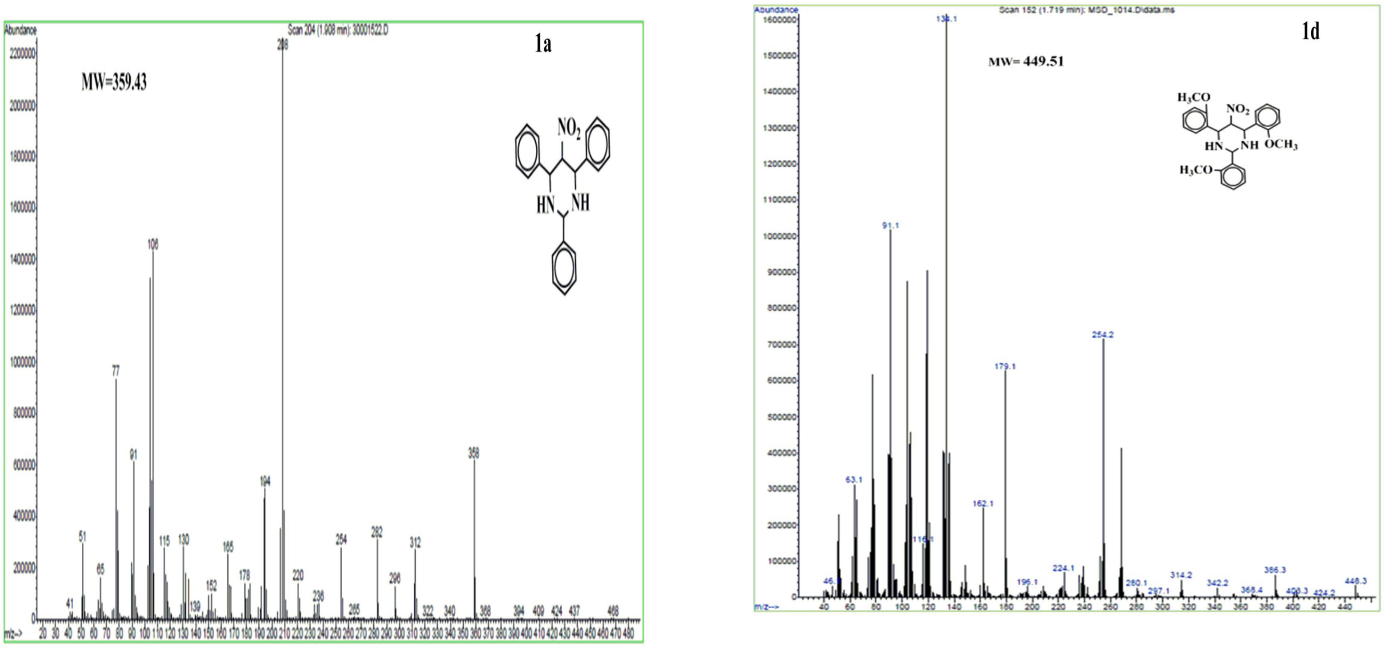


Figure 2. Some of the selected ^{1}H NMR spectra for the compounds (1a, d,f and g)



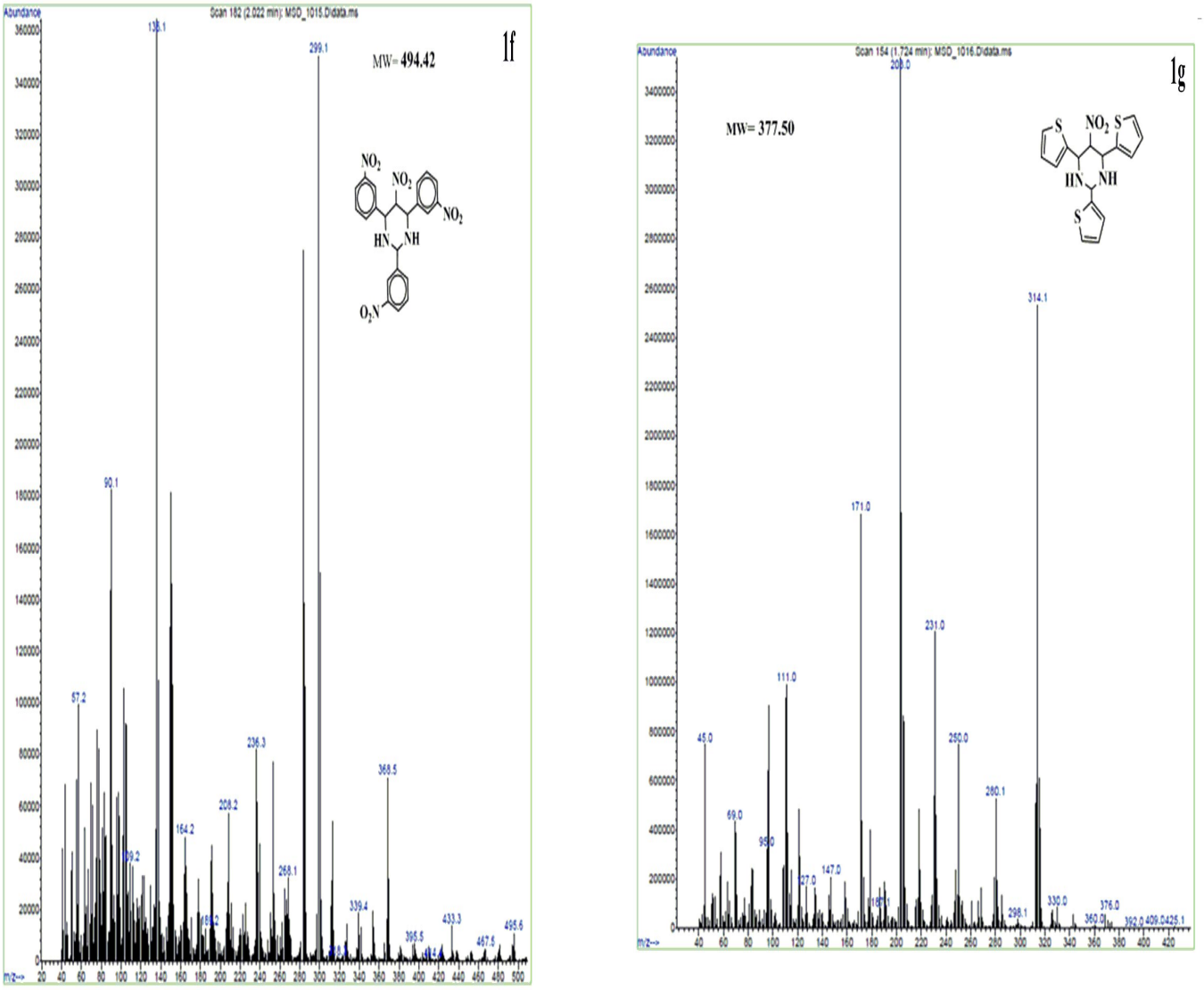


Figure 3. Some of the selected Mass spectra for the compounds (1a, d,f and g)

Crystallographic study

Figs. 4 and 5 showed the molecular structure of the compound (1a). The summary of its crystalline data as follows: $C_{22}H_{21}N_3O_2$, *MW* = 359.42 g.mol^{-1}, crystal system, group of space: Orthorhombic, P n m a, a= 24.6356 (11) A°, b= 13.6001 (6) A°, c= 5.6873 (2) A°, α = 90°, β = 90°, γ = 90°, *V* = 1905.51 (14) A°^{3}, *Z* = 4, size of crystal: (0.1×0.1×0.1 mm), type of radiation: *Cu Kα*, *λ* = 1.5418 A°, D_x = 1.253 Mg m^{-3}.

The intensity of X-ray diffraction of compound 1a was measured by using an *STOE Stadi Vari Pilatus100 K* diffractometer which included (data collection, cell refinement, and data reduction) (22), the type of radiation: *Cu Kα*, *λ* = 1.5418 A° by the ω-scanning technique. All subsequent calculations were carried out by using the *SHELX* program package to solve and refine the structure (23). The crystal structure was resolved by the direct method, thereafter, refined with parameters of anisotropic displacement for all hydrogen and other atoms. The molecular graphics of the structure were designed by using the *MERCURY* program (24). In the crystal of the compound observed supramolecular features, where symmetry-related molecules had been joined into infinite chains along the *C* axis via strong N1—H1••••O1^{ii} hydrogen bonds with symmetry code – (ii) $^{x, y, z + 1}$ Fig. 6. Experimental details of X-ray crystal data of the compound (1a) were provided in Tables 1–5.

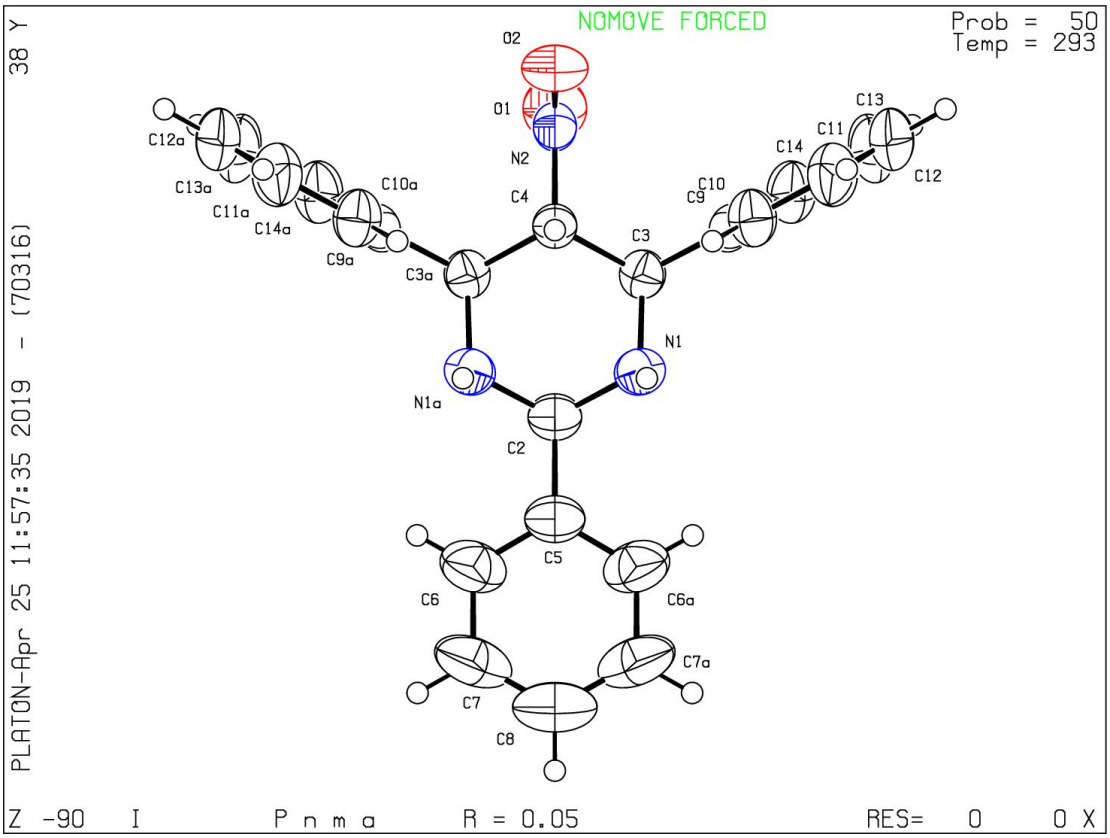


Figure 4. Data block-showing the molecular structure, numbering of atoms and displacement ellipsoids were drawn at 50 % probability level of the compound (1a)

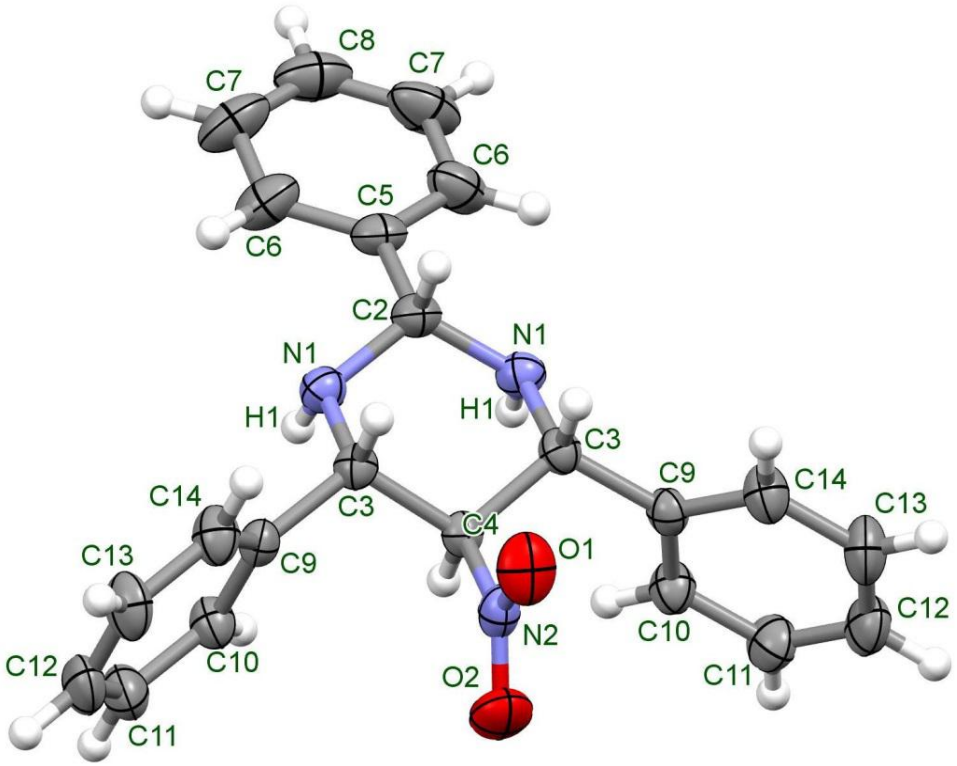


Figure 5. Shows the details of the molecular structure data of the compound (1a) summarized in Tables 1–5

Table 1. Experimental details of X-ray data (Data of crystal, collection of data and structure refinement) of the compound (1a)

Data of crystal	
MF	$C_{22}H_{21}N_3O_2$
MW	359.42
Crystal system, group of space	Orthorhombic, P n m a
Temp.	293 K
a,b,c	24.6356 (11) A°, 13.6001 (6) A°, 5.6873 (2) A°
α,β,γ	*90°, 90°, 90°*
V	1905.51 (14) $A°^3$
Z	4
Type of radiation	*Cu Kα*
μ (mm^{1})	0.66
Size of crystal	0.1×0.1×0.1 mm
Collection of data	
Diffractometer	Stoe Stadi Vari Pilatus 100 K
Absorption correction	Multi-scan
Number of measured, observed and independent [I > 2σ (I)] reflections	13153, 1009, 1900
R_{int}	0.110
$(\sin \theta/\lambda)_{max}$	0.613 $A°^{-1}$
Structure refinement	
R [F2 > 2σ (F2)],wR (F2),S	0.048, 0.116, 0.85
Reflections number	1900
Parameters number	137
Treatment of H—atom	H—atoms were treated by using a mixture of constrained and independent refinements
$\Delta\rho_{max}$, $\Delta\rho_{min}$	0.20, −0.18 (e $A°^{-3}$)
Deposition number in CCDC	1985551

Table 2. Geometric parameters (Bond length, *Aº)* of the compound (1a)

Bond	*d (Aº)*	Bond	*d (Aº)*
O1-N2	1.230 (3)	C6-H6	0.9300
O2-N2	1.208 (3)	C7-C8	1.357 (3)
N1-C2	1.456 (2)	C7-H7	0.9300
N1-C3	1.460 (2)	$C8-C7^{i}$	1.357 (3)
N1-H1	0.85 (2)	C8-H8	0.9300
N2-C4	1.488 (3)	C9-C10	1.383 (3)
$C2-N1^{i}$	1.456 (2)	C9-C14	1.387 (3)
C2-C5	1.522 (4)	C10-C11	1.369 (3)
C2-H2	0.9800	C10-H10	0.9300
C3-C9	1.501 (2)	C11-C12	1.365 (3)
C3-C4	1.541 (2)	C11-H11	0.9300
C3-H3	0.9800	C12-C13	1.367 (3)
$C4-C3^{i}$	1.541 (2)	C12-H12	0.9300
C4-H4	0.9800	C13-C14	1.377 (3)
$C5-C6^{i}$	1.373 (3)	C13-H13	0.9300
C5-C6	1.373 (3)	C14-H14	0.9300
C6-C7	1.399 (3)		

Table 3. Geometric parameters (Bond angle, *ω*°) of the compound (1a)

Angle	*ω*°	Angle	*ω*°
C2-N1-C3	112.05 (16)	C5-C6-C7	120.6 (3)
C2-N1-H1	110.1 (15)	C5-C6-H6	119.7
C3-N1-H1	111.3 (14)	C7-C6-H6	119.7

O2-N2-O1	123.3 (3)	C8-C7-C6	119.7 (3)
O2-N2-C4	118.7 (2)	C8-C7-H7	120.2
O1-N2-C4	118.0 (3)	C6-C7-H7	120.2
N1-C2-N1^{i}	114.9 (2)	C7^{i}-C8-C7	120.7 (4)
N1-C2-C5	110.98 (14)	C7^{i}-C8-H8	119.6
N1^{i}-C2-C5	110.98 (14)	C7-C8-H8	119.6
N1-C2-H2	106.5	C10-C9-C14	118.14 (18)
N1^{i}-C2-H2	106.5	C10-C9-C3	121.69 (17)
C5-C2-H2	106.5	C14-C9-C3	120.16 (17)
N1-C3-C9	111.91 (15)	C11-C10-C9	120.9 (2)
N1-C3-C4	108.64 (15)	C11-C10-H10	119.6
C9-C3-C4	113.01 (13)	C9-C10-H10	119.6
N1-C3-H3	107.7	C12-C11-C10	120.5 (2)
C9-C3-H3	107.7	C12-C11-H11	119.7
C4-C3-H3	107.7	C10-C11-H11	119.7
N2-C4-C3i	109.88 (13)	C11-C12-C13	119.6 (2)
N2-C4-C3	109.88 (13)	C11-C12-H12	120.2
C3^{i}-C4-C3	110.44 (19)	C13-C12-H12	120.2
N2-C4-H4	108.9	C12-C13-C14	120.4 (2)
C3^{i}-C4-H4	108.9	C12-C13-H13	119.8
C3-C4-H4	108.9	C14-C13-H13	119.8
C6^{i}-C5-C6	118.7 (3)	C13-C14-C9	120.4 (2)
C6^{i}-C5-C2	120.62 (14)	C13-C14-H14	119.8
C6-C5-C2	120.62 (14)	C9-C14-H14	119.8

Symmetry code–(i) $^{x, -y+3/2, z}$

Table 4. The coordinates fractional atomic and parameters of displacement isotropic or equivalent isotropic ($A^{\circ 2}$) of the compound (1a)

Atom	*x*	*y*	*z*	*Uiso* / Ueq*
O1	0.15428 (10)	0.7500	−0.9115 (4)	0.0844 (7)
O2	0.22407 (10)	0.7500	−0.6856 (4)	0.0858 (7)
N1	0.06799 (6)	0.65973 (12)	−0.3019 (3)	0.0535 (4)
H1	0.0858 (8)	0.6523 (15)	−0.175 (4)	0.064*
N2	0.17552 (11)	0.7500	−0.7157 (4)	0.0556 (6)
C2	0.03626 (10)	0.7500	−0.2920 (5)	0.0549 (7)
H2	0.0129	0.7500	−0.4316	0.066*
C3	0.10363 (7)	0.65694 (13)	−0.5070 (3)	0.0477 (5)
H3	0.0807	0.6593	−0.6476	0.057*
C4	0.13931 (10)	0.7500	−0.5063 (4)	0.0423 (6)
H4	0.1617	0.7500	−0.3640	0.051*
C5	−0.00143 (11)	0.7500	−0.0801 (5)	0.0607 (8)
C6	−0.01993 (9)	0.8368 (2)	0.0134 (4)	0.0829 (7)
H6	−0.0078	0.8963	−0.0482	0.099*
C7	−0.05680 (11)	0.8367 (3)	0.2005 (5)	0.0987 (9)
H7	−0.0687	0.8958	0.2646	0.118*
C8	−0.07509 (14)	0.7500	0.2879 (7)	0.0982 (13)
H8	−0.1004	0.7500	0.4092	0.118*
C9	0.13608 (7)	0.56358 (12)	−0.5172 (3)	0.0491 (5)
C10	0.17377 (8)	0.54010 (15)	−0.3460 (3)	0.0589 (5)
H10	0.1791	0.5826	−0.2200	0.071*
C11	0.20338 (9)	0.45514 (16)	−0.3596 (4)	0.0720 (6)
H11	0.2282	0.4401	−0.2419	0.086*
C12	0.19670 (10)	0.39229 (16)	−0.5445 (5)	0.0781 (7)
H12	0.2173	0.3352	−0.5542	0.094*
C13	0.15951 (12)	0.41377 (16)	−0.7154 (4)	0.0800 (7)
H13	0.1545	0.3706	−0.8403	0.096*
C14	0.12938 (9)	0.49891 (15)	−0.7037 (4)	0.0657 (6)
H14	0.1044	0.5131	−0.8215	0.079*

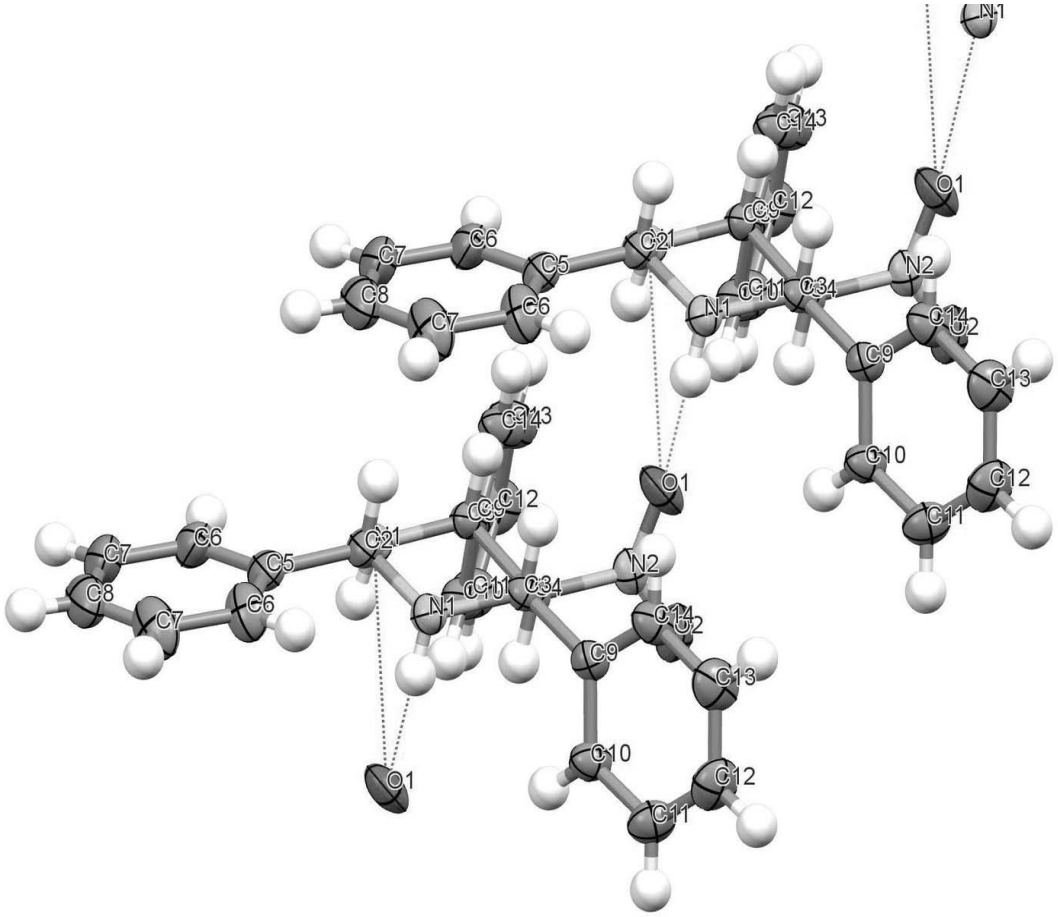


Figure 6. Shows the dashed lines for hydrogen bonds in the packing of the compound (1a)

Table 5. Hydrogen bond geometry (A^{o}) of the compound (1a)

D—H••••*A*	*D*—H	H••••*A*	*D*••••*A*	*D*—H••••*A*
N1—H1••••O1^{ii}	0.85 (2)	2.619 (3)	3.310 (3)	139.3 (2)

Symmetry code–(ii) $^{x, y, z+1}$

CONCLUSION

A novel synthesis of seven derivatives of 5-nitro-2,4,6-triphenylhexahydro-pyrimidine and some of its derivatives, by condensation of three components: benzaldehyde or some various monosubstituted its derivatives, ammonium acetate, and nitromethane via type Mannich reaction in a one pot was achieved successfully. The procedure was occurred simply with short reaction time and a good yield. The products were characterized by FTIR, ^{1}H NMR, and Mass spectroscopy. The molecular structure of 5-nitro-2,4,6-triphenylhexahydropyrimidine was affirmed by X-ray crystallography analysis of single-crystal.

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CONFLICT OF INTEREST

None

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