Synthesis and Characterization of New Oxazepine and Oxazepane Derived From Schiff Bases

تحضير وتشخيص اوكسازيبين واوكسازيبان جديده مشتقه من قواعد شيف .

Sawsan Khdeaur Abbas
Chemistry Department,College of Science,University of Kerbala

Abstract

In this work new bis -1,3-oxazepine and bis 1,3- oxazepane - 4,7- dione derivatives have been synthesized by cycloaddition reaction type [2+5→7] of maleic, phthalic and succinic anhydrides to synthesized Schiff bases derivatives . *ortho*-Tolidine was condensed with different aromatic aldehydes (4-hydroxy3-methoxy benzaldehyde, 4- isopropyl benzaldehyde, 2- formyl furan, 2,5-dimethoxybenzaldehyde and 4-chlorobenzaldehyde) in presence of glacial acetic acid as catalyst in absolute ethanol to give schiff bases derivatives A_1-A_5 respectively. The resulting Schiff bases derivatives A_1-A_5 were then introduced in [2+5→7] cycloaddition reaction with each maleic ,phthalic and succinic anhydrides in dry benzene to give new bis-1,3-oxazepine,1,3-oxazepane-4,7-dione derivatives A_6-A_{11} and A_{12}-A_{16} respectively.The synthesized target compounds A_6-A_{11} and A_{12}-A_{16} have been characterized by (C.H.N.)elementary micro analysis and the spectroscopic methods including FT-IR , ^{1}H NMR .

Keywords:- oxazepine , Derivative of schiff bases.

الخلاصه

تم من خلال هذا العمل تحضير مشتقات ثنائية - 3،1 - اوكسازبين-4، 7-دايون و 1 ،3- اوكسازيبان – 4، 7 – دايون وذلك بأستعمال تفاعل الاضافة الحلقية [2+5←7] لانهيدريد الفثالك ، الماليك و السكسنيك الى بعض مشتقات قواعد شيف. تم اولا التكاثف بين اورثو - توليدين ومشتقات بنزالديهايد متنوعة (4- هيدروكسي 3- ميثوكسي بنزالديهايد ،4- ايزو بروبيل بنزالديهايد ، 2- فورميل فيوران ،2 ،5- ثنائي ميثوكسي بنزالديهايد و 4- كلوروبنزالديهايد) على التتالي بوجود حامض الخليك الثلجي كعامل مساعد في الايثانول المطلق. تم إدخال قواعد شيف المحضرةA_1-A_5 لاحقا في تفاعل إضافة حلقية [2+5] مع كل من وانهدريد الماليك ،انهدريدالفثالك و انهدريد السكسنيك في البنزين الجاف فتم الحصول على مشتقات 3،1- اوكسازبين -4،7- دايون و 1 ،3- اوكسازيبان – 4، 7 – دايون ثنائية جديده A_6-A_{11} و A_{12}-A_{16} على التتالي .تم تشخيص مركبات الاوكسازبين والاوكسازيبان الثنائية المحضرة A_6-A_{11} و A_{12}-A_{16} بوساطة التحليل الكمي العنصري الدقيق (C.H.N.) وكذلك بالطرق الطيفية المتضمنة مطيافية الأشعة تحت الحمراء ومطيافية الرنين النووي المغناطيسي للبروتون^{1}H NMR .

Introduction:-

Schiff bases are prepared from an amino and carbonyl compounds [(1)] , which is important class of organic compounds in medical and pharmaceutical field [(2)]. Furthermore, Schiff bases were reported to show a range of interesting biological activities [(3)], as analgesic [(4)], antibacterial [(5)] , antifungal [(6)] , anticancer [(7)] and herbicidal activity [(8)].

Oxazepane are a well- known of a seven-membered heterocyclic organic compound ring with two hetero atoms in position 1,3 [(9)] . The classical methods for synthesis oxazepine ring are limited $5+2 \longrightarrow 7$. Recently, cycloaddition reaction, which is a type from a pericyclic they are prepared by the reaction of Schiff base with maleic, phthalic and anhydride[(10)] which is classified as a , implying five-atom component plus two-atom component leading to seven-membered cyclic ring [(11)]. Oxazepine derivatives have medicinal and biological activities against different types of bacteria [(12)], in addition of their uses as inhibitors of some enzymes action [(13)]. Oxazepine derivatives are known to possess a variety of biological activities like anticancer [(14)], antifungal[(15)] , anticonvulsant[(16)] , antirombotic[(17)] , antiinflammatory[(18)], antipsychotic[(19)], antidepressant[(20)], antiviral[(21)]and telomerase inhibitors[(22)].

Experimental

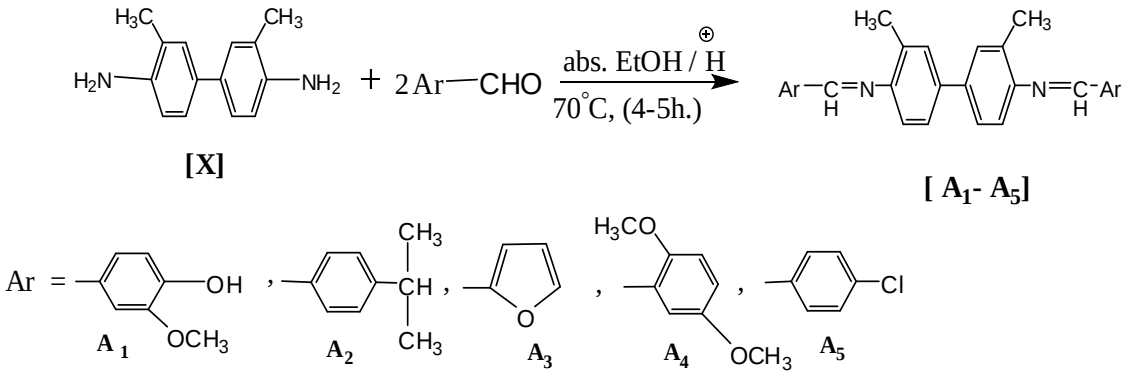
General

All Chemicals were supplied by Merck and BDH Chemicals Co. and used as received.

Melting points were determined by stuart melting point apparatus.Elemental analysis measured on Euro, E.A.300, Single. Instruction manual (V.3.0 single)Babylon University.FT-IR spectra were recorded on FT-IR 8400s, schimadzu - spectrophotometer and using KBr discs-Karbala University.H^1-NMR spectra were recorded on JNM - model \ Joal 400 MHZ usingtetramethyl silane as internal standard and DMSO as solvent. Measurements were made atsapala organics private (India).

Preparation of Schiff bases (A_1-A_5)

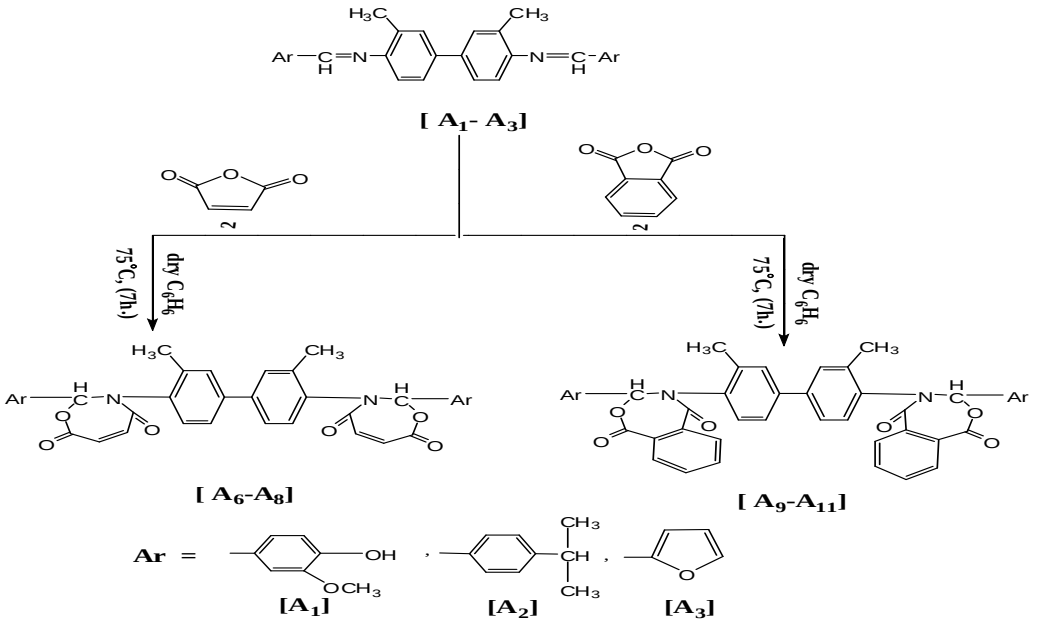
A series of Schiff bases were prepared from the reaction of o-Toludine (X) (1 mole), with different aldehydes (2 moles), in 40ml of ethanol absolute and a few drops of glacial acetic acid. This mixture was refluxed for (4-5) hrs in water bath at (70◦C). The mixture was cooled, the colored precepitate was obtained then rercystallized from ethanol as shown in (scheme 1). The physical properties and other characteristics for the synthesized Schiff bases derivatives (A_1-A_5) were shown in table (1).



Scheme(1)

Preparation of 1,3- Oxazepine derivatives (A_6-A_{11})

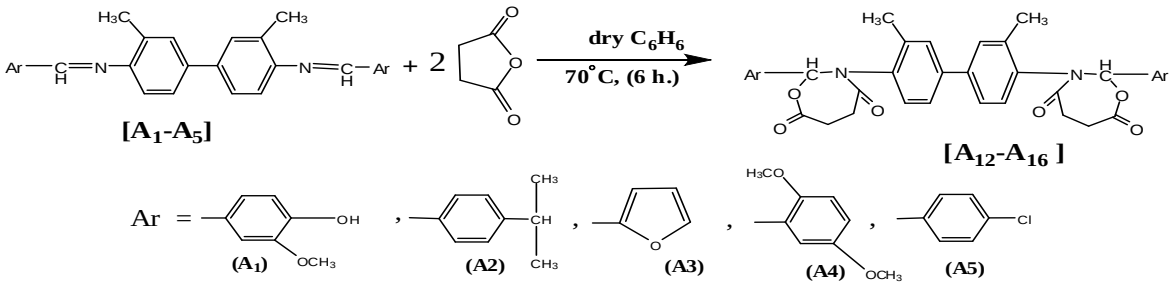
A mixture of Schiff bases (A_1-A_3) (0.0006mole) and maleic or phthalic anhydride (0.0012mole) was dissolved in (20mL) of dry benzene. The mixture was heated for 7hrs in water bath at (75◦C), excess solvent was distilled, the precipitate was filtered and recyrstallized from ethanol to give the products of compounds (A_6-A_{11}) as shown in scheme (2) .The physical properties and other characteristics for the synthesized Oxazepine derivatives (A_6-A_{11}) were shown in table (2) .The (C.H.N) elementary analysis of 1,3-oxazepine derivatives (A_6-A_{11}) was listed in table (4) .



Scheme (2)

Preparation of 1,3- Oxazepane derivatives (A_{12}-A_{16})

A mixture of Schiff bases (A_1-A_5) (0.0006mole) and succinic anhydride (0.0012mole) was dissolved in (20mL) of dry benzene. The mixture was heated for 6 hrs in water bath at (70◦C), excess solvent was distilled, the precipitate was filtered and recyrstallized from dioxane to yield oxazepane compounds (A_{12}-A_{16}) as shown in scheme (3) .The physical properties and other characteristics for the synthesized oxazepane derivatives (A_{12}-A_{16}) were shown in table (3). The (C.H.N) elementary analysis of 1,3-oxazepane derivatives (A_{12}-A_{16}) was listed in table (4) .



Scheme (3)

Table (1):- physical properties and other characteristics for the synthesized Schiff base derivatives (A_1-A_5)

Com. No.	Molecular formula	M.Wt g/ mol	color	Yield%	M.P.ºC
A_1	$C_{30}H_{28}N_2O_4$	480.2	Yellow	79	181-183
A_2	$C_{34}H_{36}N_2$	472.29	Dark yellow	72	142-143
A_3	$C_{24}H_{20}N_2O_2$	368.15	green	86	195-197
A_4	$C_{32}H_{32}N_2O_4$	508.24	Greenish yellow	92	182-185
A_5	$C_{28}H_{22}Cl_2N_2$	459.39	Dark green	70	153-155

Table (2):- physical properties and other characteristics for the synthesized Schiff base derivatives (A_6-A_{11})

Com. No.	Molecular formula	M.Wt g/ mol	color	Yield%	M.P.ºC
A_6	$C_{46}H_{36}N_2O_{10}$	776.24	Dark yellow	63	215-217
A_7	$C_{50}H_{44}N_2O_6$	768.32	yellow	58	231-233dec.
A_8	$C_{40}H_{28}N_2O_8$	664.18	Brown	74	183-185dec.
A_9	$C_{38}H_{32}N_2O_{10}$	676.21	Greenish yellow	72	189-192
A_{10}	$C_{42}H_{40}N_2O_6$	668.29	Grey	66	198-200
A_{11}	$C_{32}H_{24}N_2O_8$	564.15	Dark red	67	191-192

Table (3):- physical properties and other characteristics for the synthesized Schiff base derivatives (A_{12}-A_{16})

Com. No.	Molecular	M.Wt g/ mol	color	Yield%	M.P.ºC
A_{12}	$C_{38}H_{36}N_2O_{10}$	680.24	Light brown	56	199-201
A_{13}	$C_{42}H_{44}N_2O_6$	672.32	brown	52	167-169
A_{14}	$C_{32}H_{28}N_2O_8$	568.18	Light green	61	174-176
A_{15}	$C_{38}H_{36}N_2O_{10}$	680.24	Light yellow	75	159-161
A_{16}	$C_{36}H_{30}Cl_2N_2O_6$	656.15	Yellow	64	189-192

Table (4):- (C.H.N.) elementary micro analysis of the synthesized 1,3-oxazepine derivatives (A_6-A_{11}) and (A_{12}-A_{16})

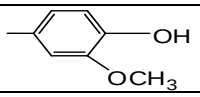
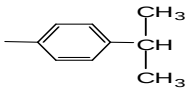
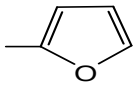
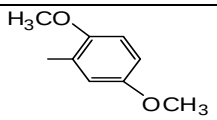
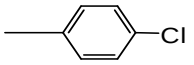
Comp.No.	C%		H%		N%	
	Calculated	Found	Calculated	Found	Calculated	Found
A6	67.45	67.23	4.77	4.95	4.14	4.41
A7	75.43	75.56	6.03	6.41	4.19	4.63
A8	68.08	68.27	4.28	4.36	4.96	4.72
A9	71.13	71.06	4.67	4.49	3.61	3.87
A10	78.10	78.18	5.77	5.91	3.64	3.90
A11	72.28	72.59	4.25	4.35	4.21	4.13
A12	67.05	67.54	5.33	5.59	4.12	4.42
A13	74.98	79.33	6.59	6.48	4.16	4.43
A14	67.60	67.48	4.96	4.08	4.93	4.70
A15	67.78	67.51	5.69	5.55	3.95	3.06
A16	65.76	65.51	4.60	4.03	4.26	4.16

Results and discussion

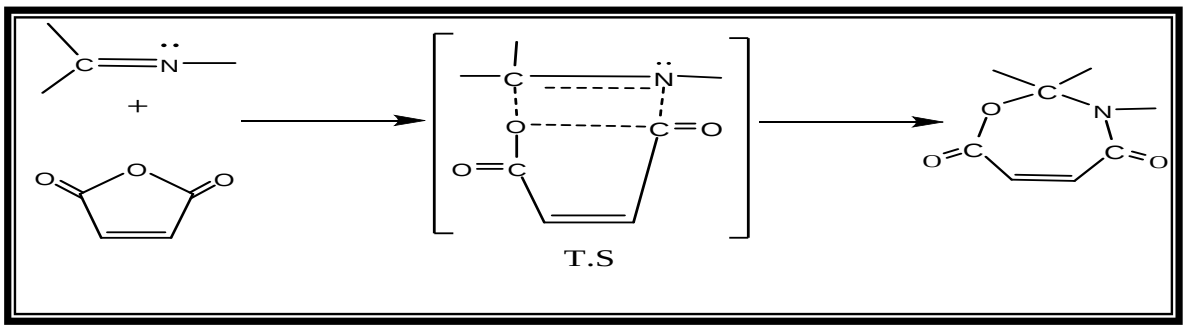
New five Schiff bases were synthesized from the reaction of ***o***-Tolidine (X) with substituted aromatic aldehydes , in presence of glacial acetic acid as catalyst shown in scheme (1) . The FTIR spectra of these compounds (A_1-A_5) , fig. [2-6] table (5) , showed good evidence that the condensation reactions happened successfully by disappearing

the sharp bands at (3468,3410) cm^{-1} and (3373,3338) cm^{-1} which attributed to the asymmetric and symmetric stretching vibrations of the two amino (-NH_2) groups in *o*-tolidine, also and appearance of strong band at (1618-1628) cm-1 which due to the υ (C=N). The other absorption bands for compounds [A_1-A_5] were listed in table (5).

Table (5):- F.T.I.R Characteristic bands and their location of the Schiff bases A_1-A_5 compounds

Comp. No.	Ar	υ (C-H) aromatic	υ (C-H) alphatic	υ(C=N)	υ(C=C) aromatic	υ(C-N)	Others
A_1		3021	2885	1628	1558	1276	υ(O-H) 3421
A_2		3050	2956	1622	1562	1290	–
A_3		3115	2883	1626	1471	1384	υ(C-O-C) ether 1022
A_4		3009	2949	1624	1494	1267	υ(C-O-C) 1217
A_5		3024	2968	1618	1560	1282	υ(C-Cl) 1085

The reaction of the prepared Schiff bases [A_1-A_5] with each Phthalic, Maleic and succinic anhydride in dry benzene as solvent afforded 1,3-oxazepine and 1,3-oxazepane derivatives [A_6-A_{16}]. Reaction of Schiff bases with these cyclic anhydrides was classified as [2+5] cycloaddition reaction[23-24]. which proceeds from a single transition state involves the two atoms of imine group and five atoms from cyclic anhydride to give seven-membered heterocycle as in the following scheme (4) [23,25]

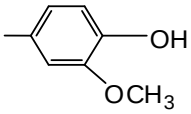
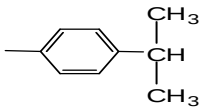
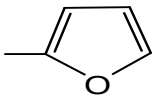
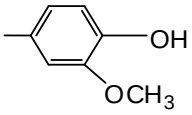
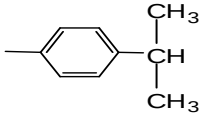
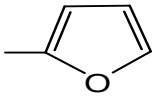


Scheme(4)

The elementary analysis (C.H.N.) of the prepared 1,3-oxazepine and 1,3-oxazepane derivatives [A_6-A_{16}] table (4) showed nearness between the calculated and found values.

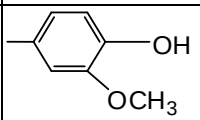
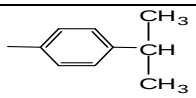
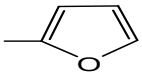
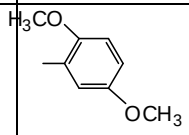
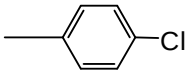
The FT-IR data of the synthesized 1,3-oxazepine derivatives (A_6-A_{11}), figs. (7-12) provide good evidence that the cycloaddition reactions proceeded successfully and produced the desired products by disappearing the strong medium band at the range (1618-1628) cm-1 which attributed to the stretching vibration of imine group (C=N) and appearing band at the range (1708-1712) cm-1attributed to the stretching vibration of (C=O) group lactam and lactone inside oxazepine ring due to the vibration coupling [26], moreover the spectra appeared band at the range (1620, 1627, and 1629) cm-1 due to υ (C=C) inside oxazepine ring. The other data of functional groups which are characteristic of these compounds are given in Table (6).

Table(6):-The FT-IR data of the synthesized 1,3-oxazepine derivatives A_6-A_{11}

Comp. No.	Ar	υ(C-H) aromatic	υ (C-H) alphatic	υ(C=C) oxazepine ring	υ(C= O) Lacton, Lactam	υ(C-N)	υ(C=C) aromatic ring	others
A_6		3047	2934	1627	1710 vib. coupling, oxazepine	1300	1529	υ (O-H) 3406 υ (C-OC) ether 1130
A_7		3062	2966	1629	1708 vib. coupling, oxazepine	1220	1531	
A_8		3053	2883	1620	1712 vib. coupling, oxazepine	1398	1527	υ (C-OC) ether 1033
A_9		3022	2976	–	1697 1654	1288	1587	υ (O-H) 3412
A_{10}		3024	2960	–	1703 1658	1298	1589	–
A_{11}		3022	2954	–	1699 1659	1290	1587	υ (C-O-C) ether 1070

The FT-IR spectra of 1,3- oxazepane derivatives [A_{12}-A_{16}], figures (13-17) respectively, showed disappearance of the sharp strong absorption band at (1618-1628) cm-1 which belong to the υ(C=N) and appearance of the following characteristic absorption bands as shown in table (7).

Table(7):-The FT-IR data of the synthesized 1,3-oxazepane derivatives A_{12}-A_{16}

Comp. No.	Ar	υ(C-H) aromatic	υ (C-H) aliphatic	υ(C-C) oxazepine ring	υ(C=o) Lacton,Lactam	υ(C-N)	others
A_{12}		3021	2935	1591	1997 1653	1276	υ(O-H) 3410
A_{13}		3050	2960	1697	1699 1656	1197	–
A_{14}		3034	2928	1628	1695 1659	1384	υ(C-O-C) 1132
A_{15}		3036	2929	1649	1732 1703	1267	υ(C-O-C) 1211
A_{16}		3037	2945	1593	1693 1653	1195	υ(C-Cl) 1089

^{1}H NMR spectra of bis-1,3-oxazepine and 1,3- oxazepane derivatives

^{1}H NMR spectra, figure (18,18a), (μH_z, DMSO-d_6) of compound [A_6] showed the following signals at δ (ppm):- The singlet signals at 2.302 ppm attributed to methyl groups protons(a), (6H, 2×CH_3) . The singlet signal at 3.855 ppm attributed to methoxy groups protons (b), (6H , 2×H_3C-O). The signal at 2.423 ppm belong to DMSO solvent . The singlet signal at 3.345 ppm due to H_2O in DMSO solvent[26]. The spectrum illustrates good evidence that the cycloaddition reaction happened successfully and formed the desired oxazepine derivative by appearing signals of olefinic (C-H) protons at (6.304- 6.334) ppm for olefinic (C-H) protons (c), (2H, 2×Hb) and (6.595 -6.626) ppm for olefinic (C-H) protons (d), (2H, 2×Hc) which is considered.The signals at the range (7.478-9.956) ppm attributed to aromatic protons and (C-H) protons of oxazepine rings. The signal at 13.192 due to (O-H) proton.

^{1}H NMR spectra, figure (19,19a), (μH_z, DMSO-d_6) of compound [A_7] showed the following signals at δ (ppm):- The singlet signal at 2.302 ppm attributed to methyl groups protons, (6H, 2×CH_3) . The singlet signals at 1.123-1.240 ppm belong to methyl protons in isopropyl group (12H, 4×CH_3) . The signals at 2.353 ppm belong to (C-H) proton in isopropyl group . The signal at 2.504 ppm belong to DMSO solvent .The broad signal at 3.389 ppm due to H_2O in DMSO solvent.The spectrum showed appearance of signals at (6.305-6.336) ppm for olefinic (C-H) protons (a),(2H, 2×Ha) and (6.598 -6.629) ppm for olefinic (C-H) protons (b), (2H, 2×Hb) which is considered good evidence that the cycloaddition reaction happened successfully and formed the desired oxazepine derivative [A7].The signals at the range (7.366-9.947) ppm attributed to aromatic protons and (C-H) protons of oxazepine rings.

^{1}H NMR spectra, figure (20,20a), (μH_z, DMSO-d_6) of compound [A_8] showed the following signals at δ (ppm) : The signals at (2.302 - 2.357) ppm attributed to methyl groups protons(a), (6H, 2×CH_3) . The signal at 2.506 ppm belong to DMSO solvent. The broad signal of H_2O in DMSO solvent appeared at 3.385 ppm. The spectrum provides good evidence that the cycloaddition reaction proceeded successfully and gave the desired oxazepine derivative [8] by appearing signals of olefinic (C-H) protons at (6.212-6.336) ppm for protons (b), (2H, 2×Hb) and (6.598 -6.730) ppm for protons (c), (2H, 2×Hc). .The signals of aromatic protons and (C-H) protons inside oxazepine rings appeared at the range (7.366 -9.948) ppm.

^{1}H NMR spectra, figure (21,21a), (μH_z, DMSO-d_6) of compound [A_{10}] showed the following signals at δ (ppm): The singlet signal at 2.302 ppm attributed to methyl groups protons(a), (6H, 2×CH_3) . The singlet signal at 2.127 ppm belong to (C-H) proton in isopropyl group. The singlet signal at 2.353 ppm belong to the methyl groups protons in isopropyl group (12H, 4×CH_3) .The singlet signal at 2.505 ppm belong to DMSO solvent. The singlet signal at 3.387 ppm due to H_2O in DMSO solvent.The signals at the range (6.672 -9.815) ppm attributed to aromatic protons and (C-H) protons of oxazepine rings.

^{1}H NMR spectra, figures (22,22a), (μH_z, DMSO-d_6) of compound [A_{11}] showed the following signals at δ (ppm): The singlet signal at 2.353 ppm attributed to methyl groups protons, (6H, 2×CH_3) .The singlet signal at 2.564 ppm belong to DMSO solvent. The singlet signal at 3.359 ppm due to H_2O in DMSO solvent.The signals at the range (7.507 -9.818) ppm attributed to aromatic protons and (C-H) protons of oxazepine rings.

^{1}H NMR spectra, figures (23,23a), (μH_z, DMSO-d_6) of compound [A_{12}] showed the following signals at δ (ppm) : The signals at (2.264 -2.415) ppm assigned to methyl groups protons(a) , (6H , 2×CH_3) . The singlet signal at 3.858 ppm attributed to methoxy groups protons (b), (6H , 2×H_3C-O) . The broad signal at 3.465 ppm due to H_2O in DMSO solvent. The spectrum illustrates good evidence that the cycloaddition reaction happened successfully and produced the desired oxazepane derivative [A_{12}] by appearing signals of cyclic (CH_2-CH_2) protons at 2.545 ppm for proton (c) ,(2H, 2×Hc) and 2.612 ppm for protons (d) , (2H, 2×Hd). The signals of aromatic protons and (C-H) protons inside oxazepane rings appeared at the range (6.904 -9.774) ppm.The signals at 12.171 ppm due to (2H، 2×O-H phenolic proton).

^{1}H NMR spectra, figures (24,24a), (μH_z, DMSO-d_6) of compound [A_{13}] showed the following signals at δ (ppm): The signals at (2.273 -2.363) ppm attributed to methyl groups protons(a), (6H, 2×CH_3) . The singlet signal at 3.483 ppm belong to H_2O in DMSO solvent. The signals at 1.123-1.240 ppm belong to methyl protons(b) (12H,4×CH_3) in isopropyl group. The signals at(2.956-2.937) ppm belong to (C-H) protons in isopropyl group . The signals of cyclic (CH_2-CH_2) protons at (2.415 -2.542) ppm for protons (c) ,(2H, 2×Hc) and 2.617 ppm for protons (d) , (2H, 2×Hd). The signals of aromatic protons and protons of (C-H) inside oxazepane rings appeared at the range (7.107 -9.343) ppm.

^{1}H NMR spectra, figures (25,25a), (μH_z, DMSO-d_6) of compound [A_{14}] showed the following signals at δ (ppm) : The signals at (2.270 - 2.353) ppm attributed to methyl groups protons, (6H, 2×CH_3) . The signal of H_2O in DMSO solvent appeared at 3.397 ppm. The spectrum illustrates good evidence that the cycloaddition reaction happened successfully and formed the desired oxazepane derivative [14] by appearing signals at (2.418-2.542) ppm ppm assigned to cyclic (CH_2-CH_2) protons (a), (2H , 2×Ha) and (2.612) ppm belong to protons (b) (2H , 2×Hb), respectively inside oxazepine rings .The signals of aromatic protons and (C-H) protons inside oxazepane rings appeared at the range (6.725-9.343) ppm.

^{1}H NMR spectra, figures (26,26a), (μH_z, DMSO-d_6) of compound [A_{15}] showed the following signals at δ (ppm) : The signals at (2.263-2.276) ppm attributed to methyl groups protons, (6H, 2×CH_3) . The signal at 2.541 ppm belong to DMSO solvent. The signal of H_2O in DMSO solvent appeared at 3.392 ppm.The signals at (3.754 -3.853) ppm assigned to methoxy groups protons, (12H, 4×O-CH_3). The spectrum illustrates good evidence that the cycloaddition reaction happened successfully and formed the desired oxazepane derivative [15] by appearing two signals at (2.366-2.414) ppm and (2.547-2.620) ppm assigned to cyclic (CH_2-CH_2) protons (a), (2H , 2×Ha) and protons (b),(2H, 2×Hb) respectivly inside oxazepane rings.The signals of aromatic protons and (C-H) protons inside oxazepane rings appeared at the range (7.049 -9.351) ppm.

^{1}H NMR spectra, figures (27,27a) , (μH_z, DMSO-d_6) of compound [A_{16}] showed the following signals at δ (ppm) : The signals at 2.279 ppm attributed to methyl groups protons, (6H, 2×CH_3) .The singlet signal at 2.415 ppm belong to DMSO solvent. The singlet signal at 3.369 ppm due to H_2O in DMSO solvent. The spectrum showed appearance of signals at (2.382 -2.415) ppm for cyclic (CH_2-CH_2) protons(a) ,(2H, 2×Ha) and (2.542-2.614) for protons (b), (2H, 2×Hb) respectivly inside oxazepane rings which is considered good evidence that the cycloaddition reaction took place successfully and yielded the desired oxazapane derivative [16]. The signals of aromatic protons and (C-H) protons inside oxazapane rings appeared at the range (7.163-9.351) ppm.

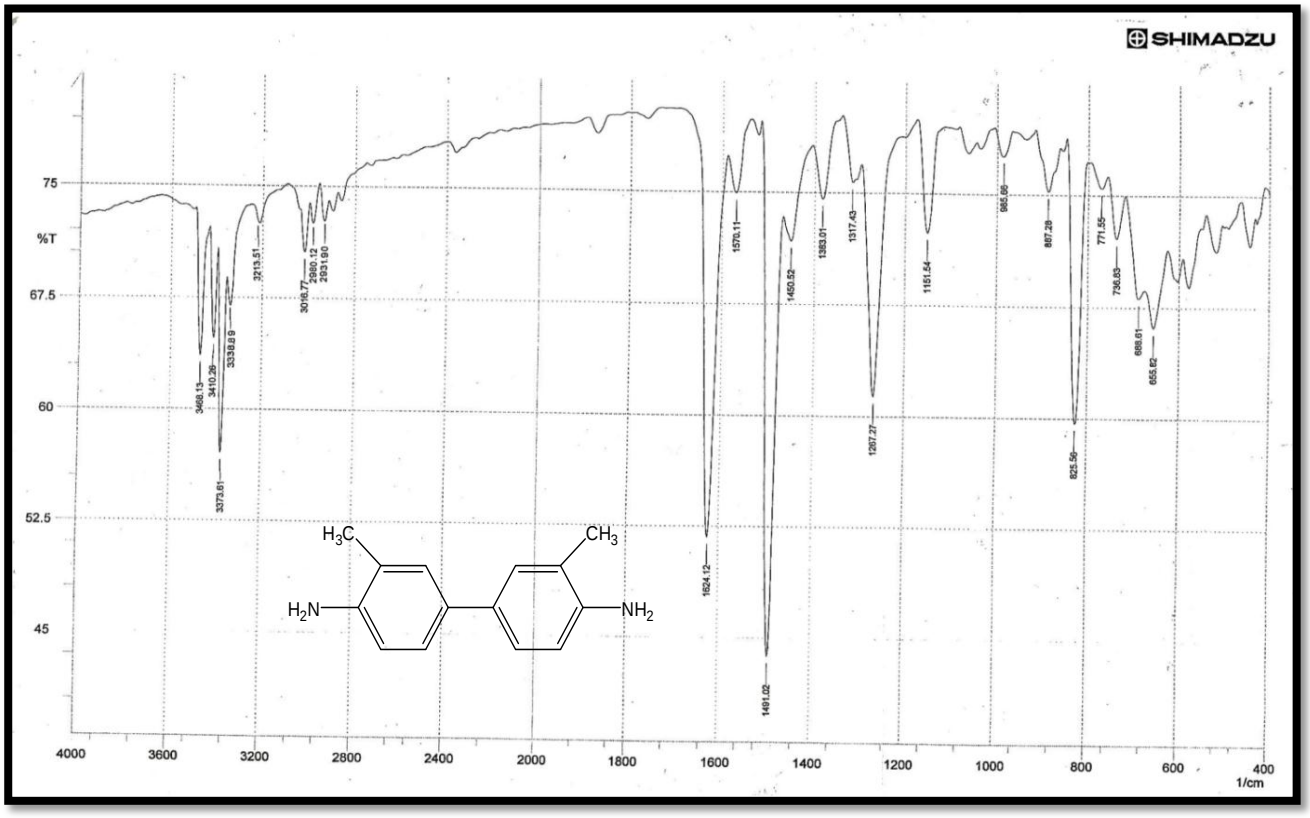


Fig. (1) :- F.T.I.R spectrum of compound [X]

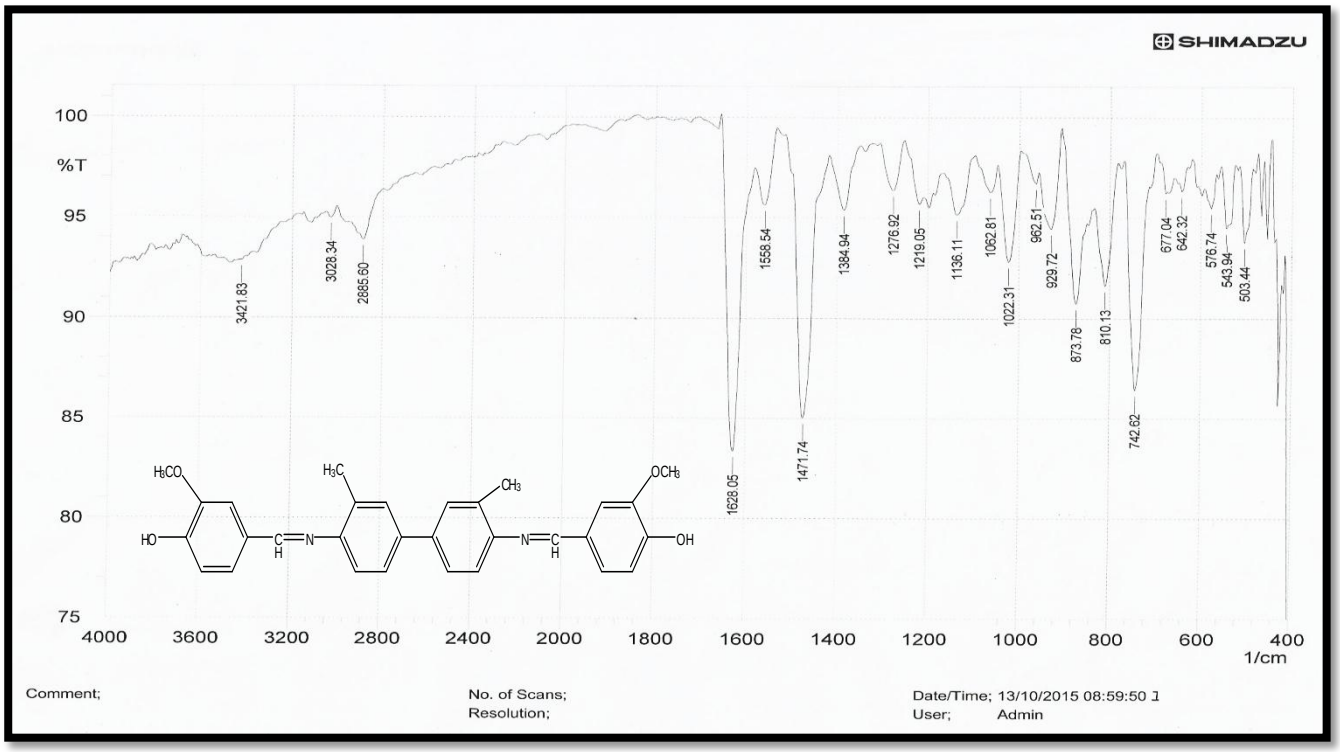


Fig. (2) :- F.T.I.R spectrum of compound [A1]

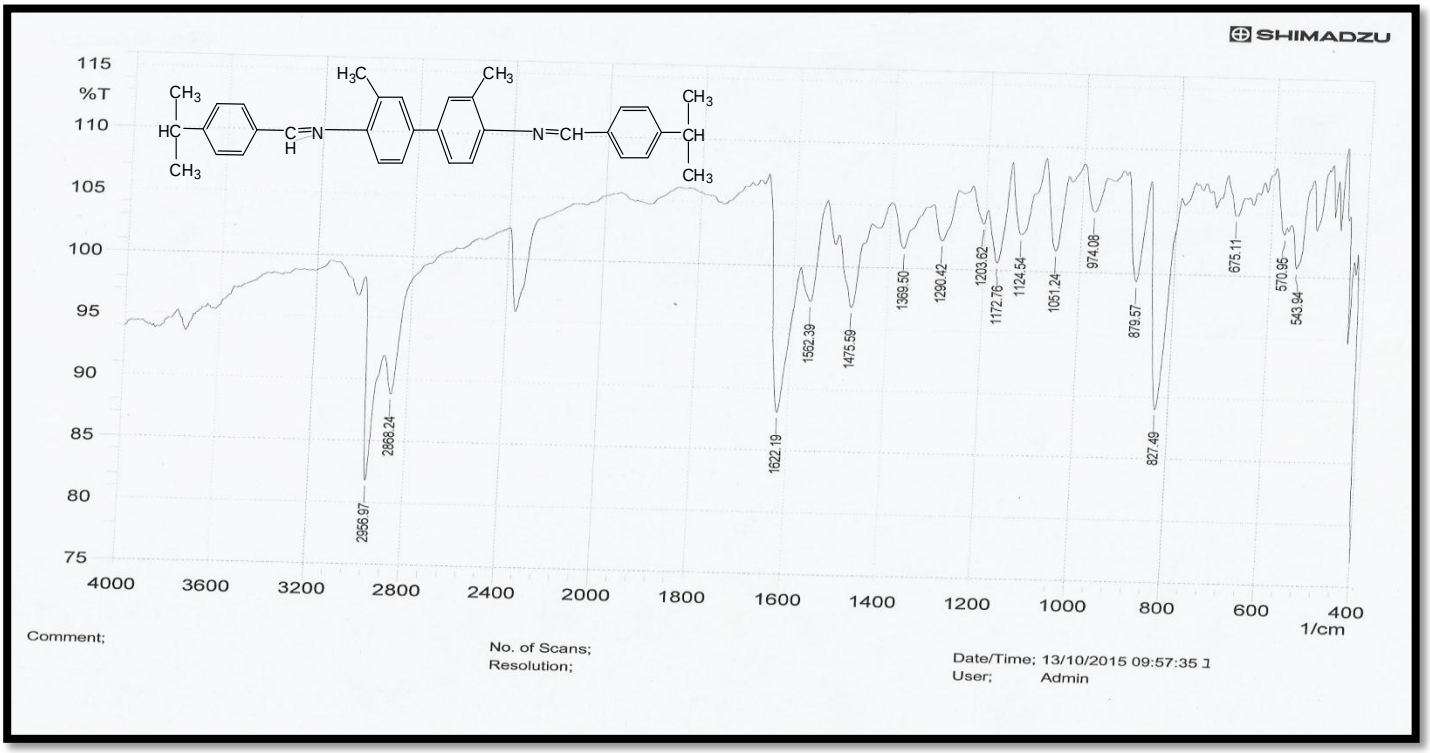


Fig. (3) :- F.T.I.R spectrum of compound [A2]

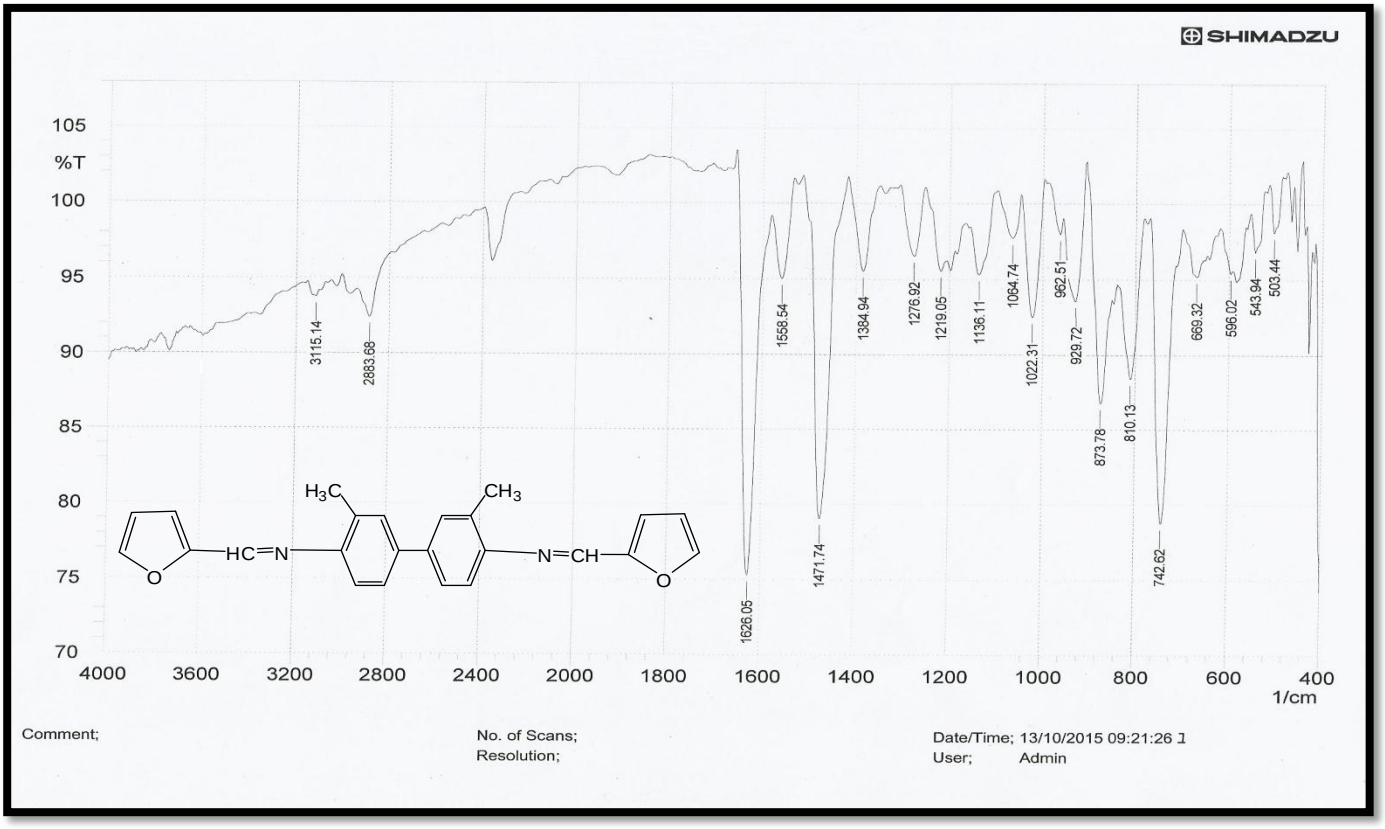


Fig. (4) :- F.T.I.R spectrum of compound [A3]

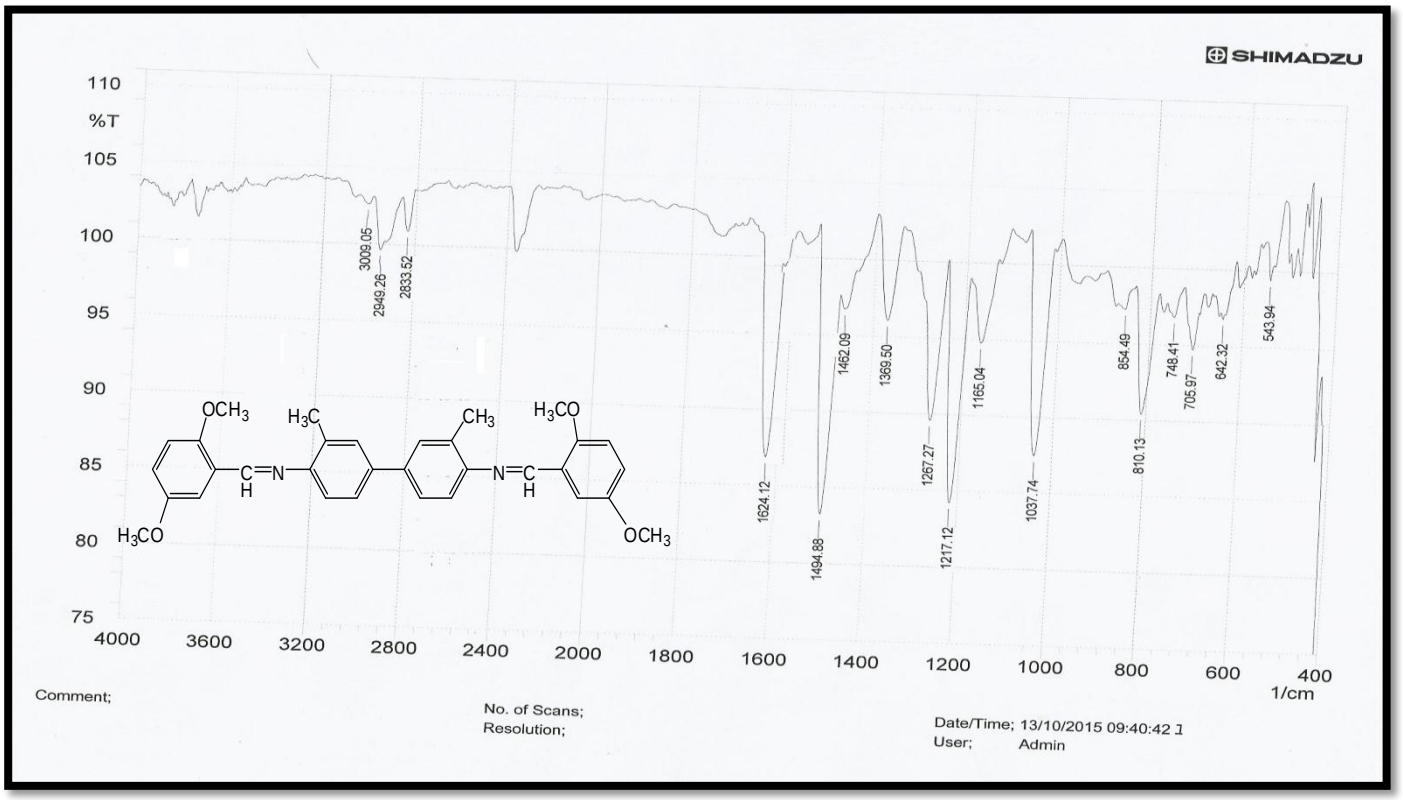


Fig. (5) :- F.T.I.R spectrum of compound[A4]

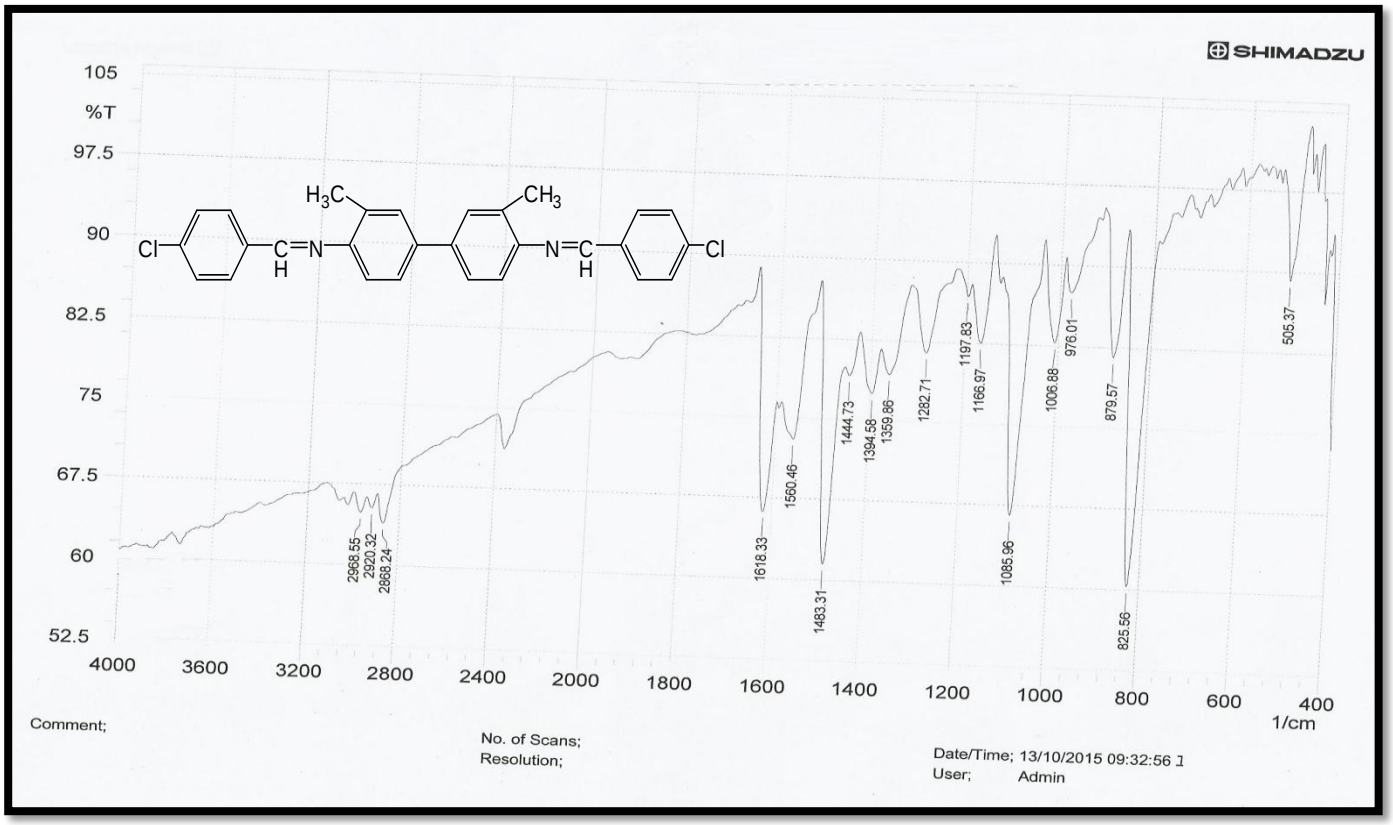


Fig. (6) :- F.T.I.R spectrum of compound[A5]

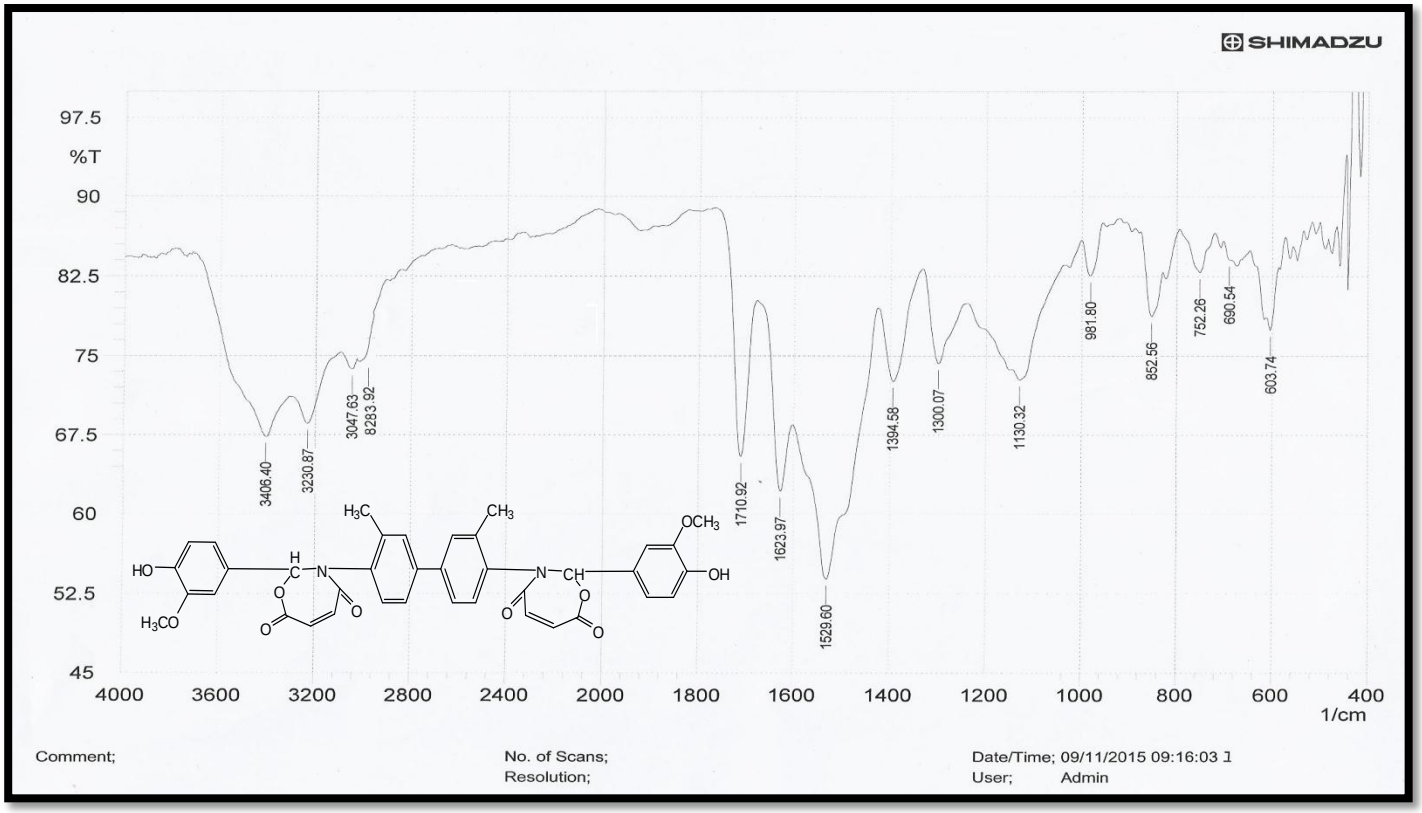


Fig. (7) :-F.T.I.R spectrum of compound[A6]

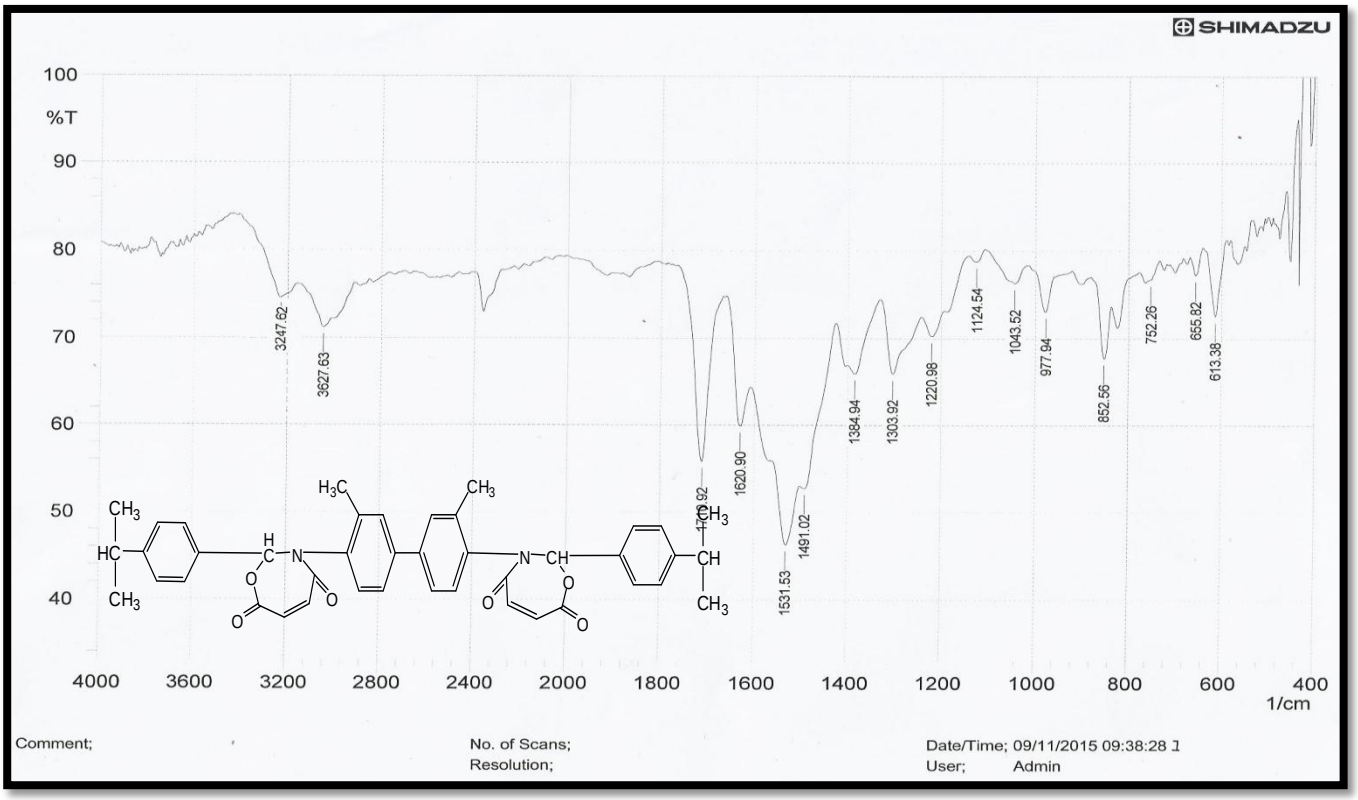


Fig. (8) :-F.T.I.R spectrum of compound[A7]

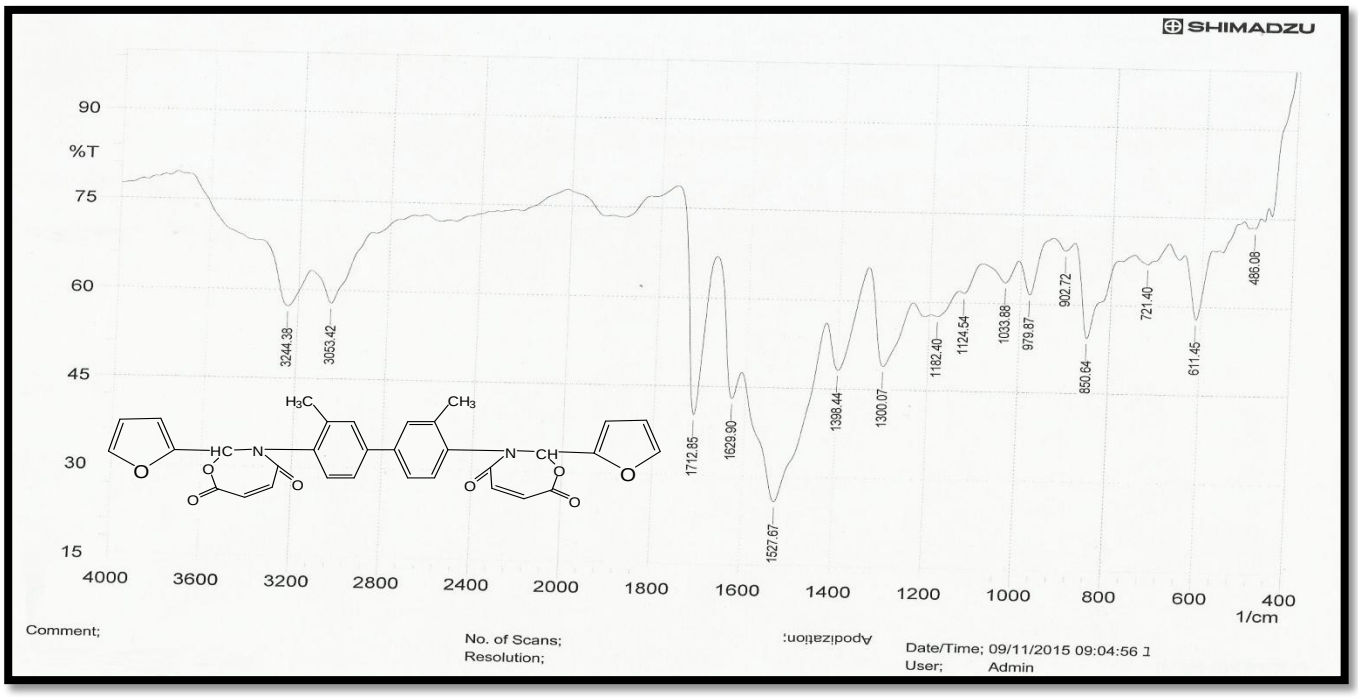


Fig. (9) :-F.T.I.R spectrum of compound[A8]

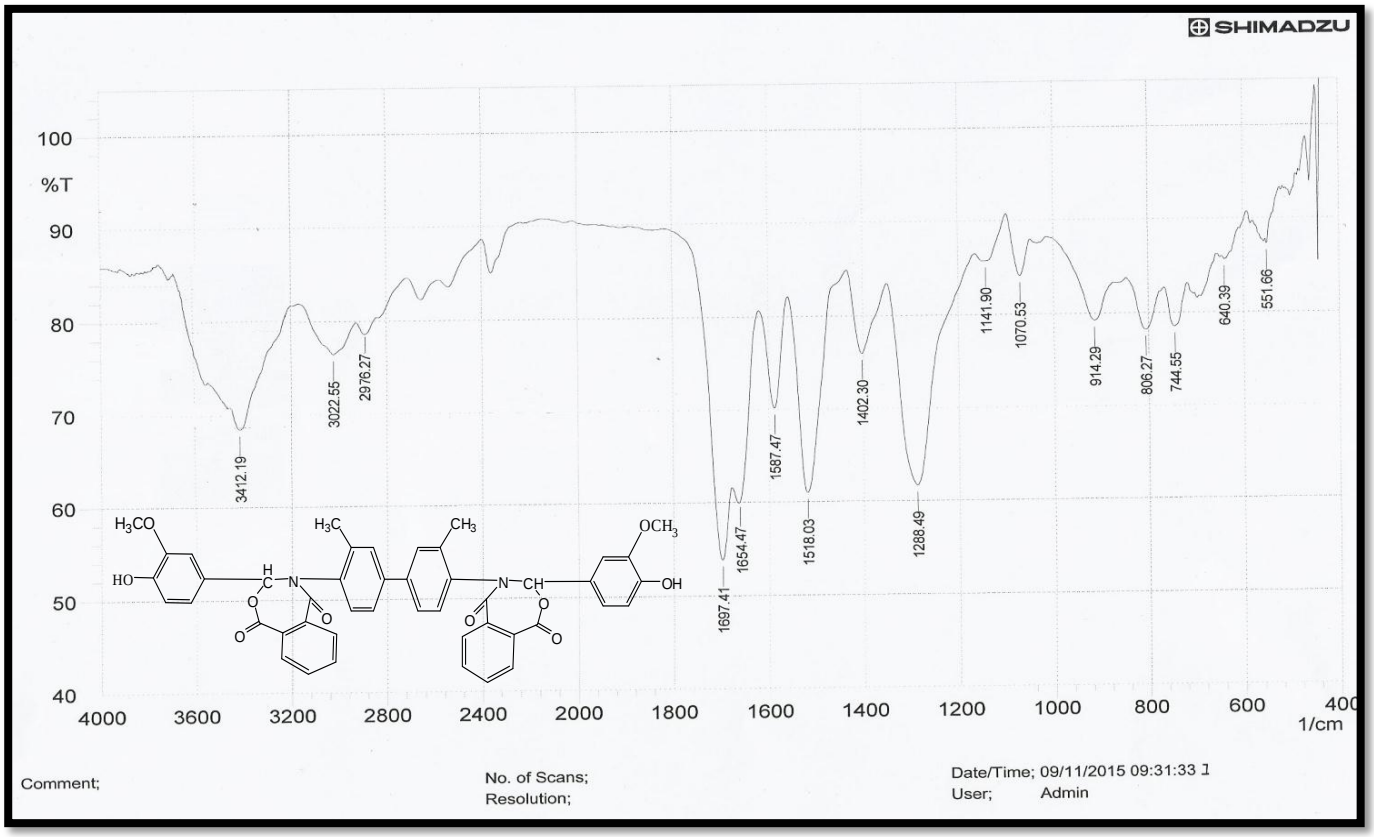


Fig. (10) :-F.T.I.R spectrum of compound[A9]

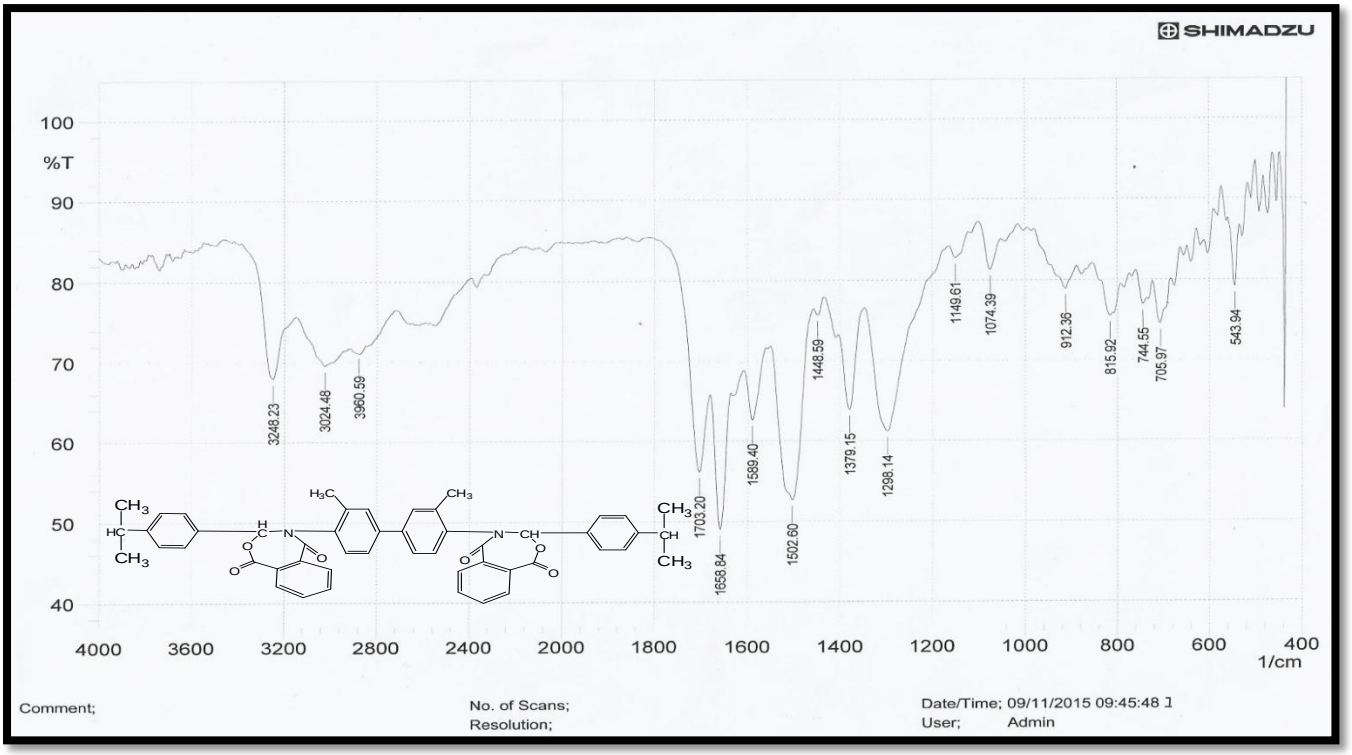


Fig. (11) :-F.T.I.R spectrum of compound[A10]

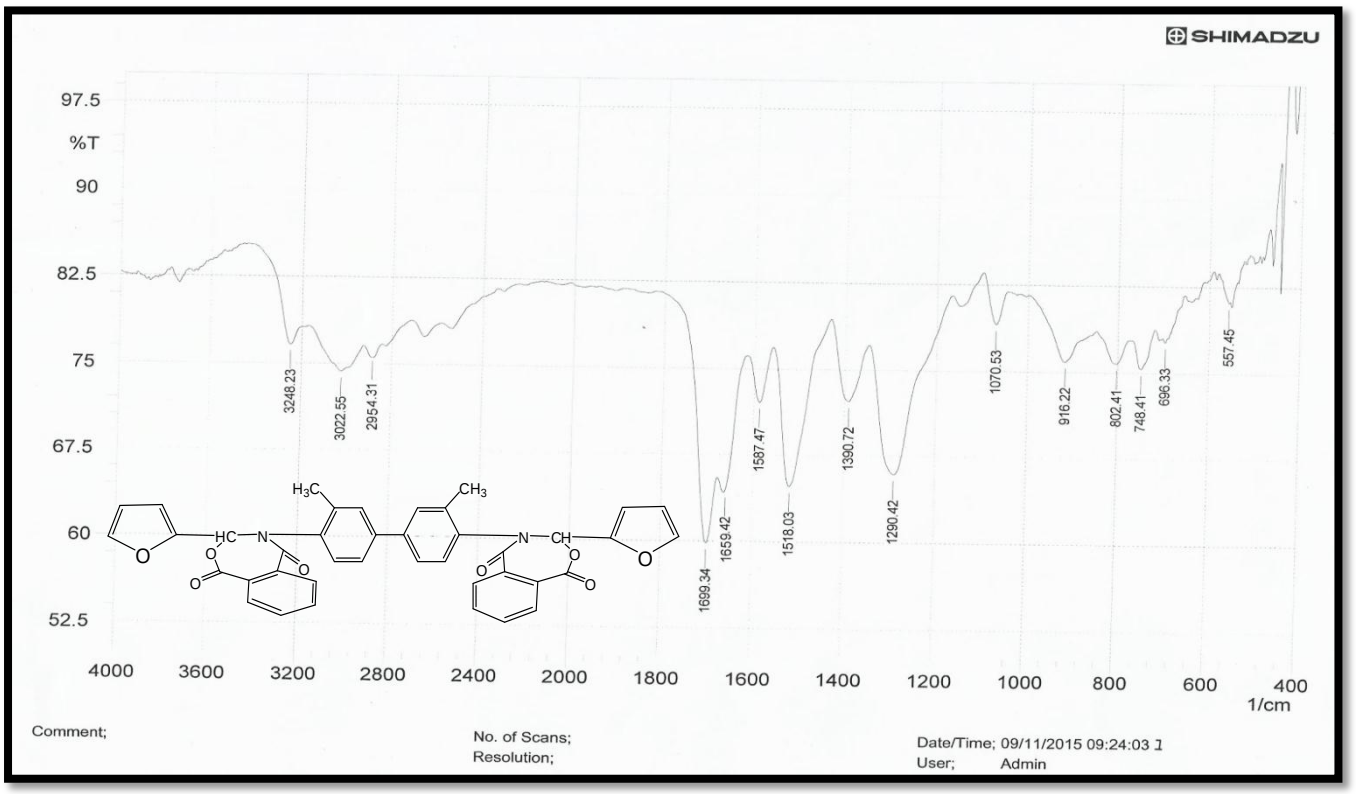


Fig. (12) :-F.T.I.R spectrum of compound[A11]

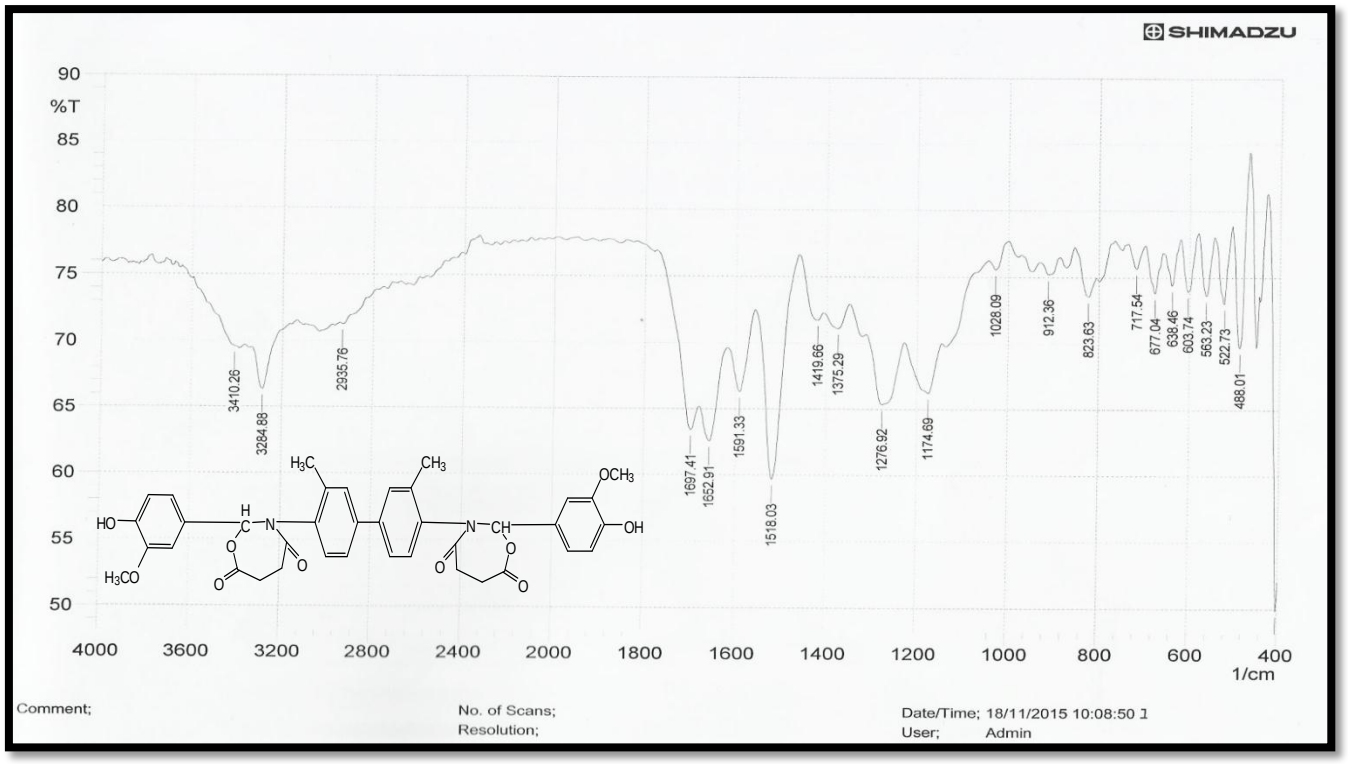


Fig. (13) :-F.T.I.R spectrum of compound[A12]

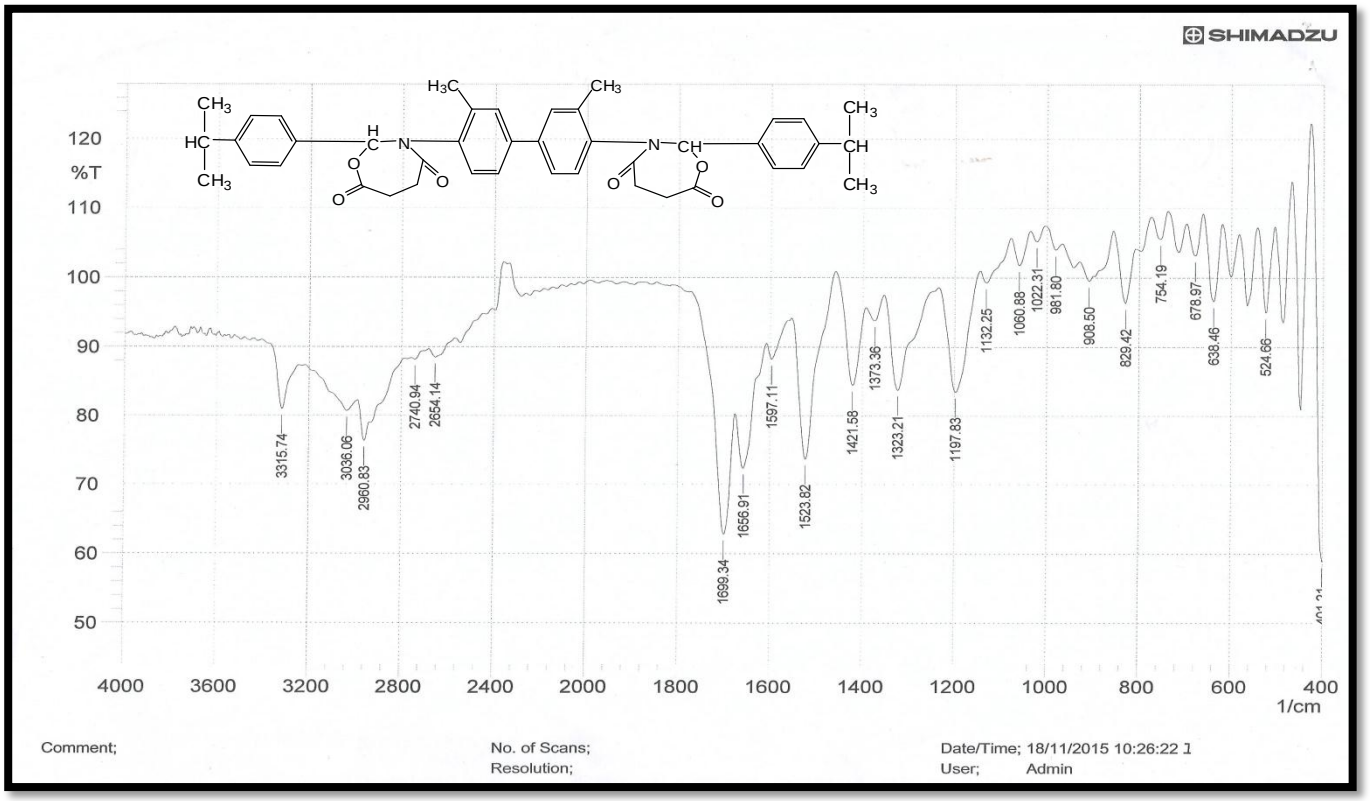


Fig. (14) :-F.T.I.R spectrum of compound[A13]

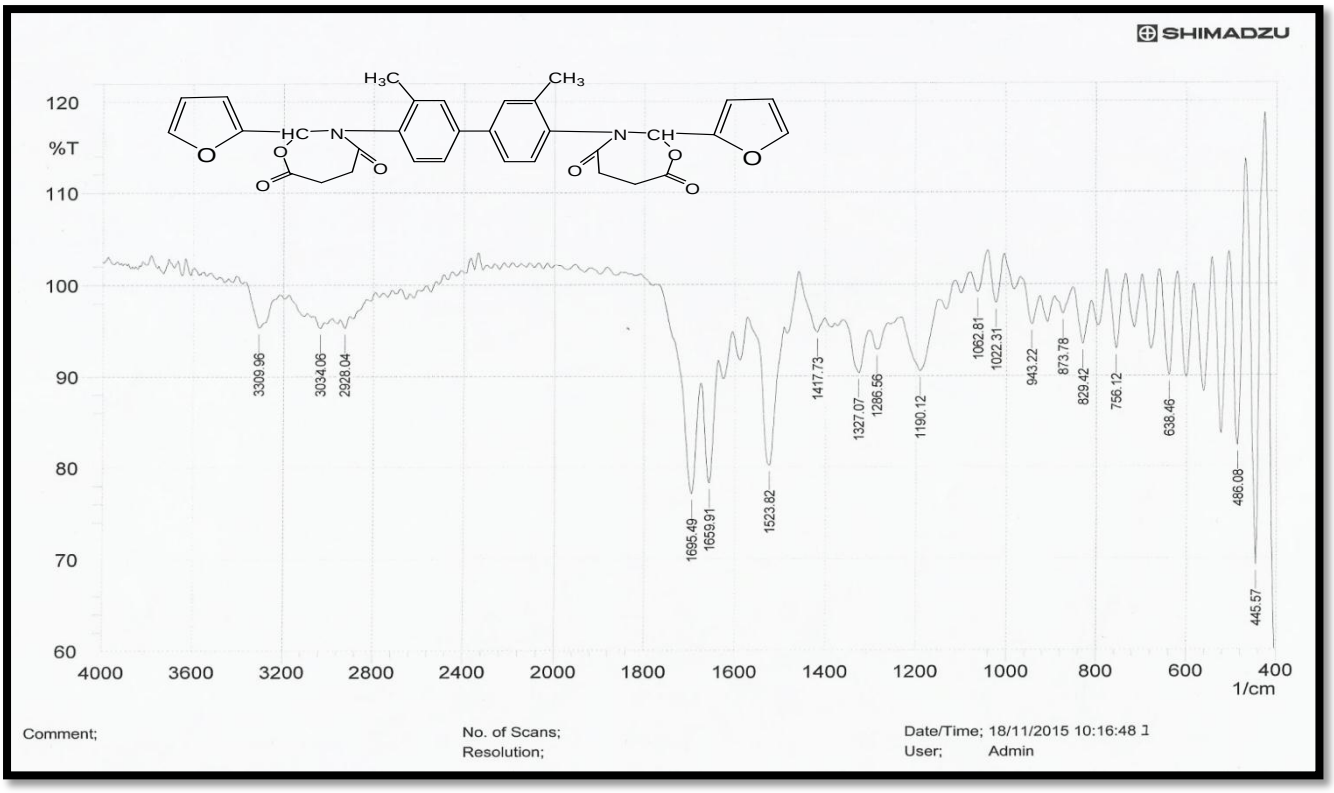


Fig. (15) :-F.T.I.R spectrum of compound[A14]

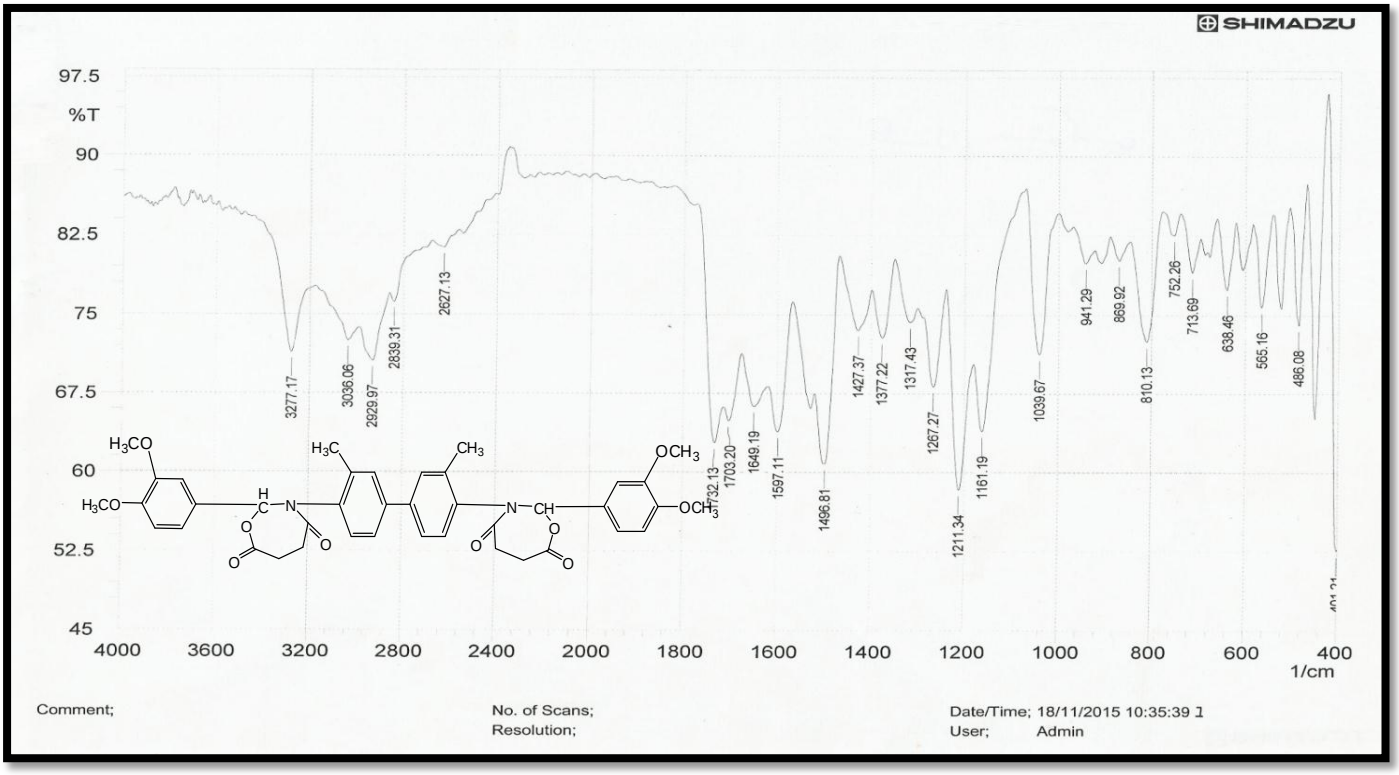


Fig. (16) :-F.T.I.R spectrum of compound[A15]

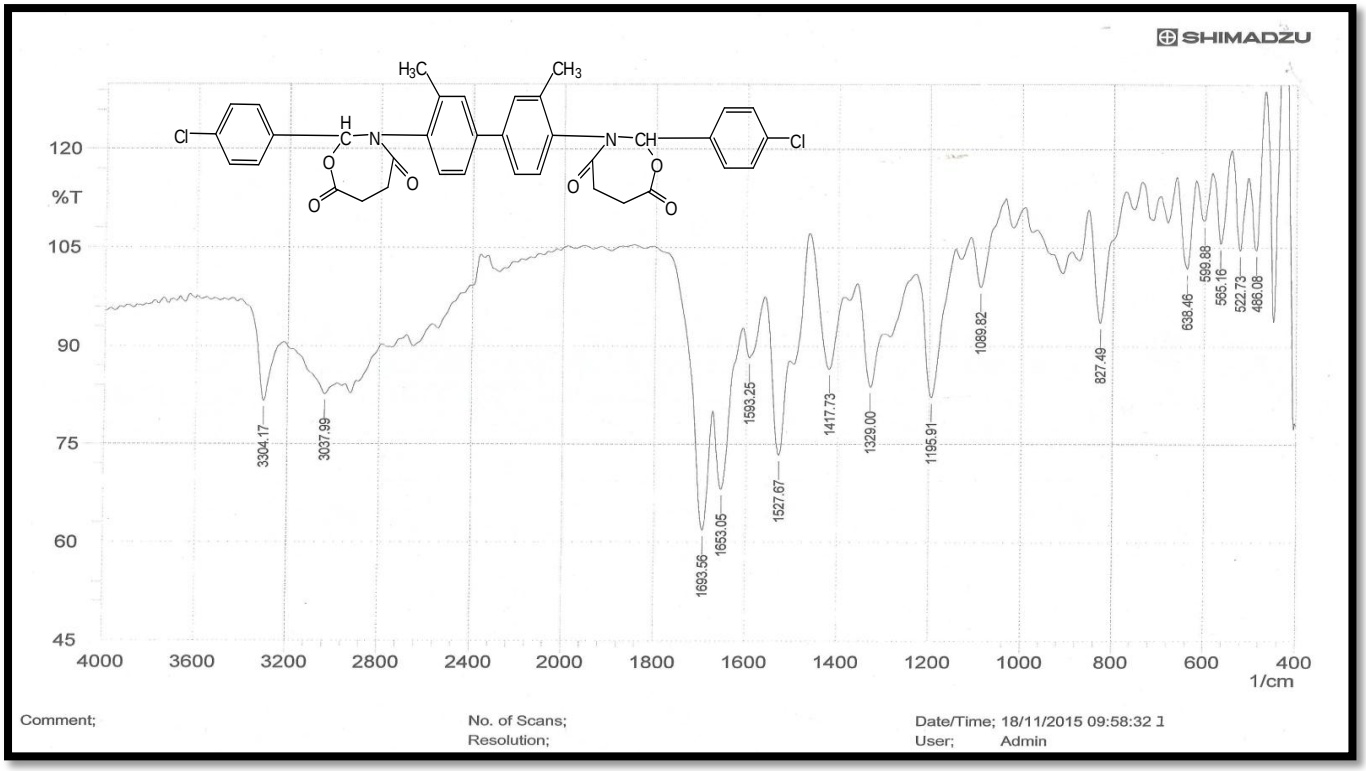


Fig. (17) :-F.T.I.R spectrum of compound[A16]

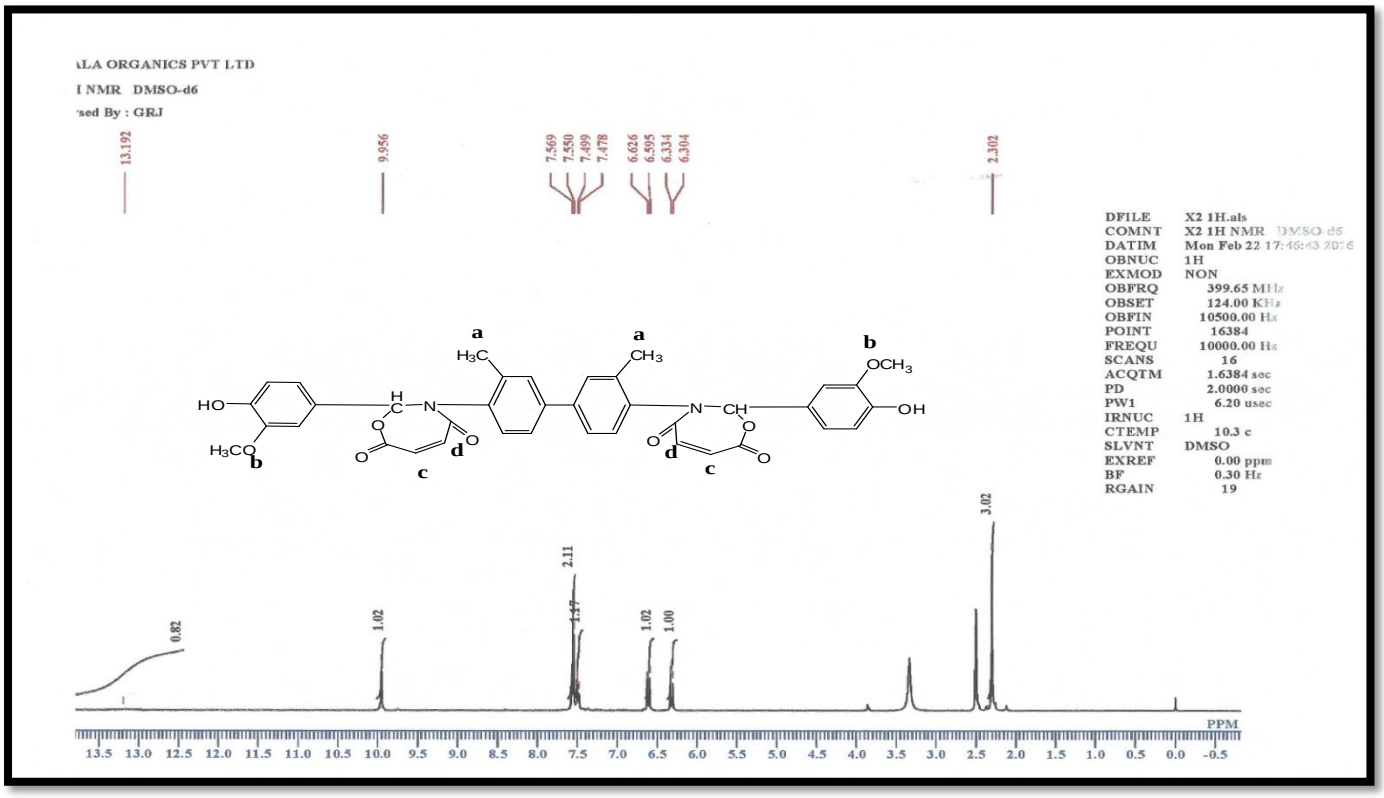


Fig.(18) : ^{1}H NMR spectrum of compound (A6)

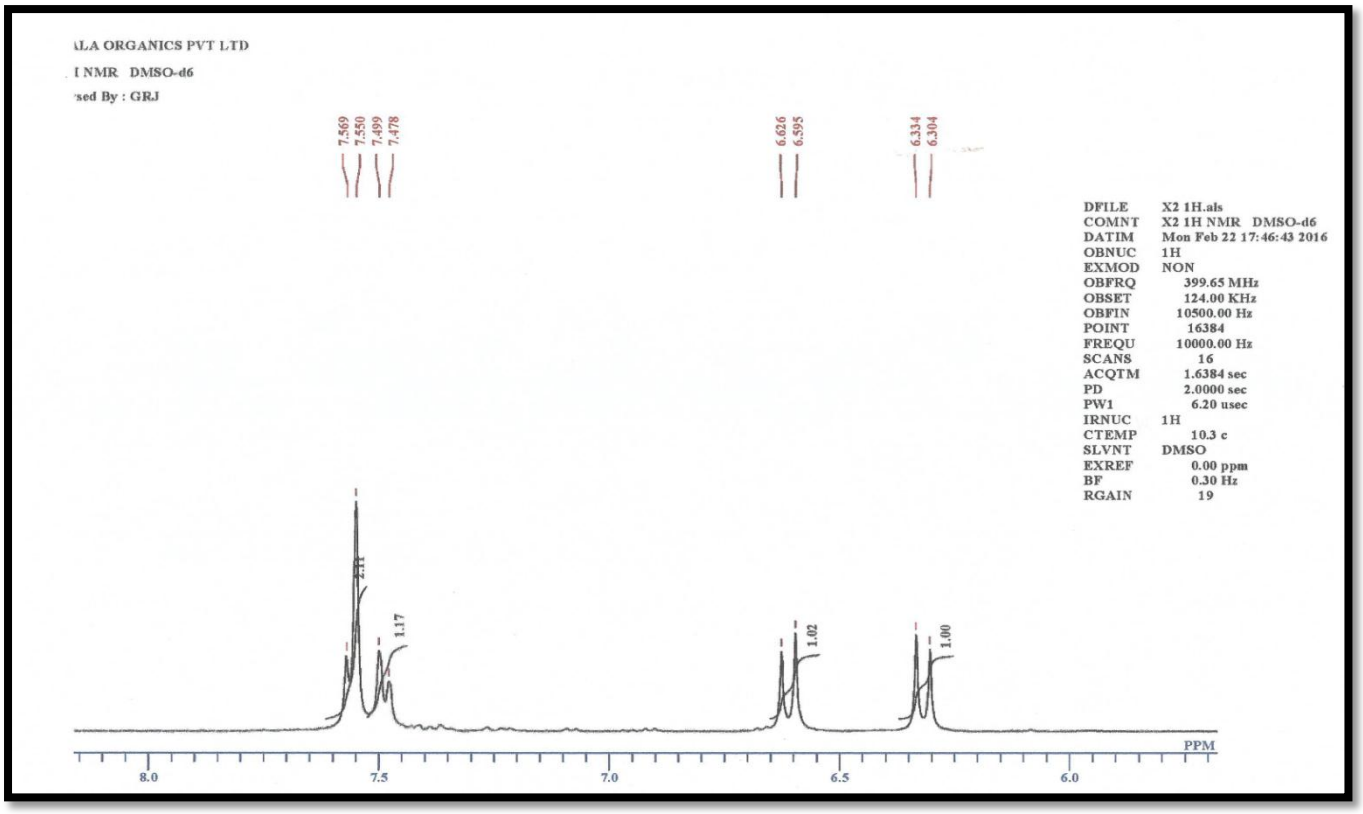


Fig.(18a) : expanded ^{1}H NMR spectrum of compound (A6)

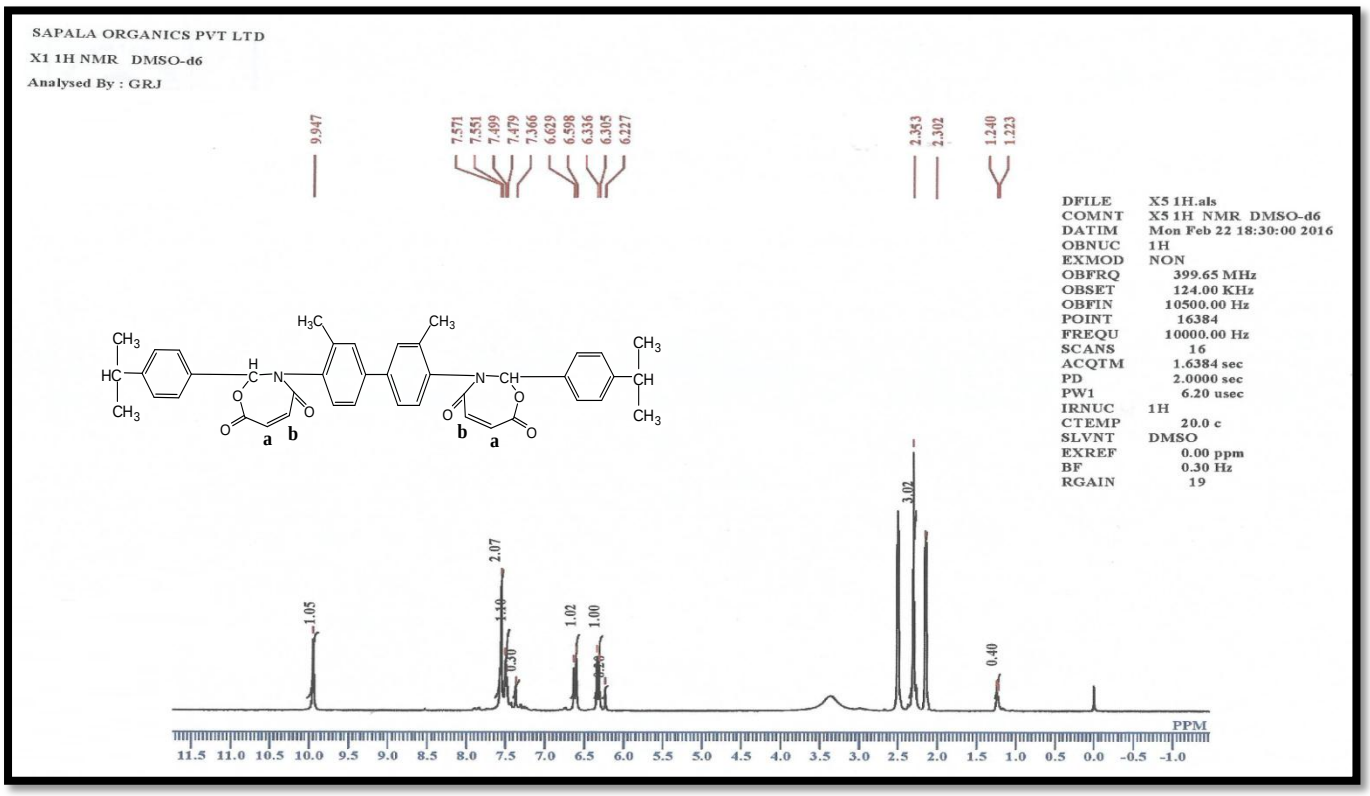


Fig.(19) : ^{1}H NMR spectrum of compound (A7)

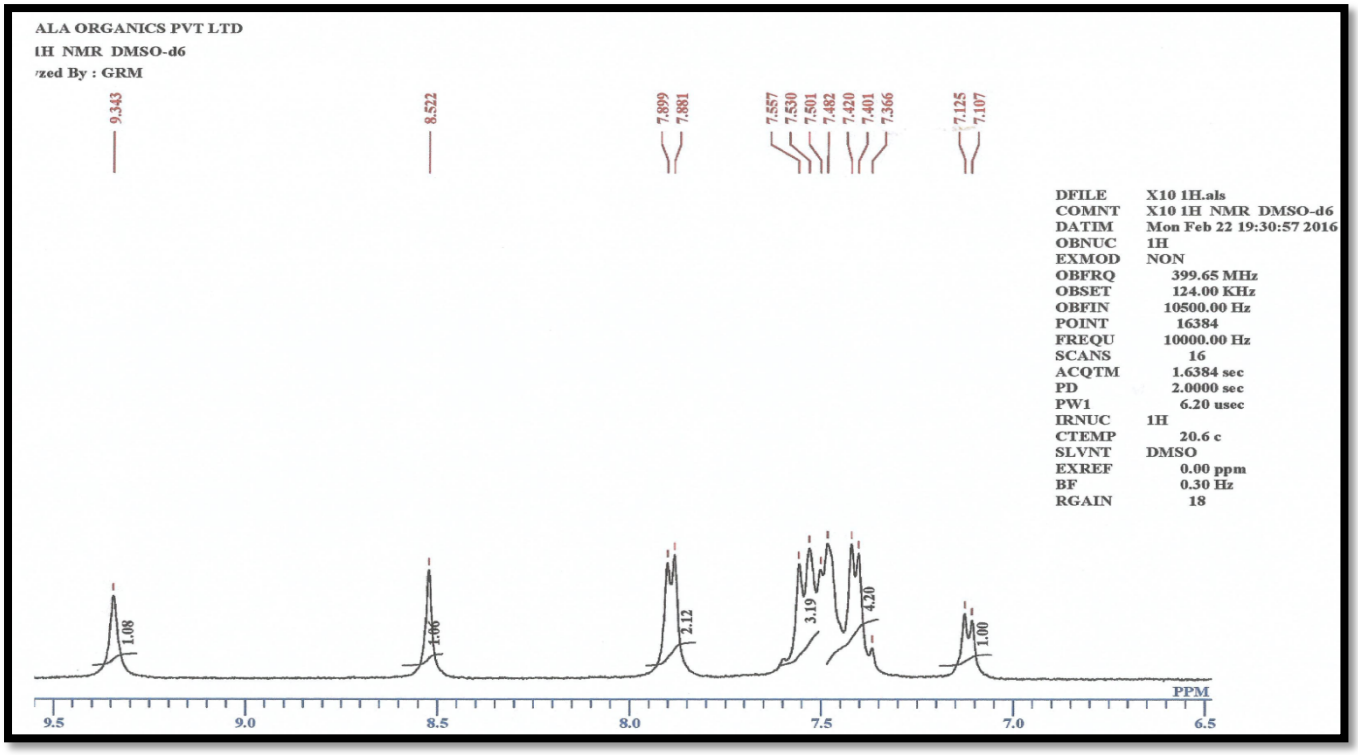


Fig.(19a) : expanded ^{1}H NMR spectrum of compound (A7)

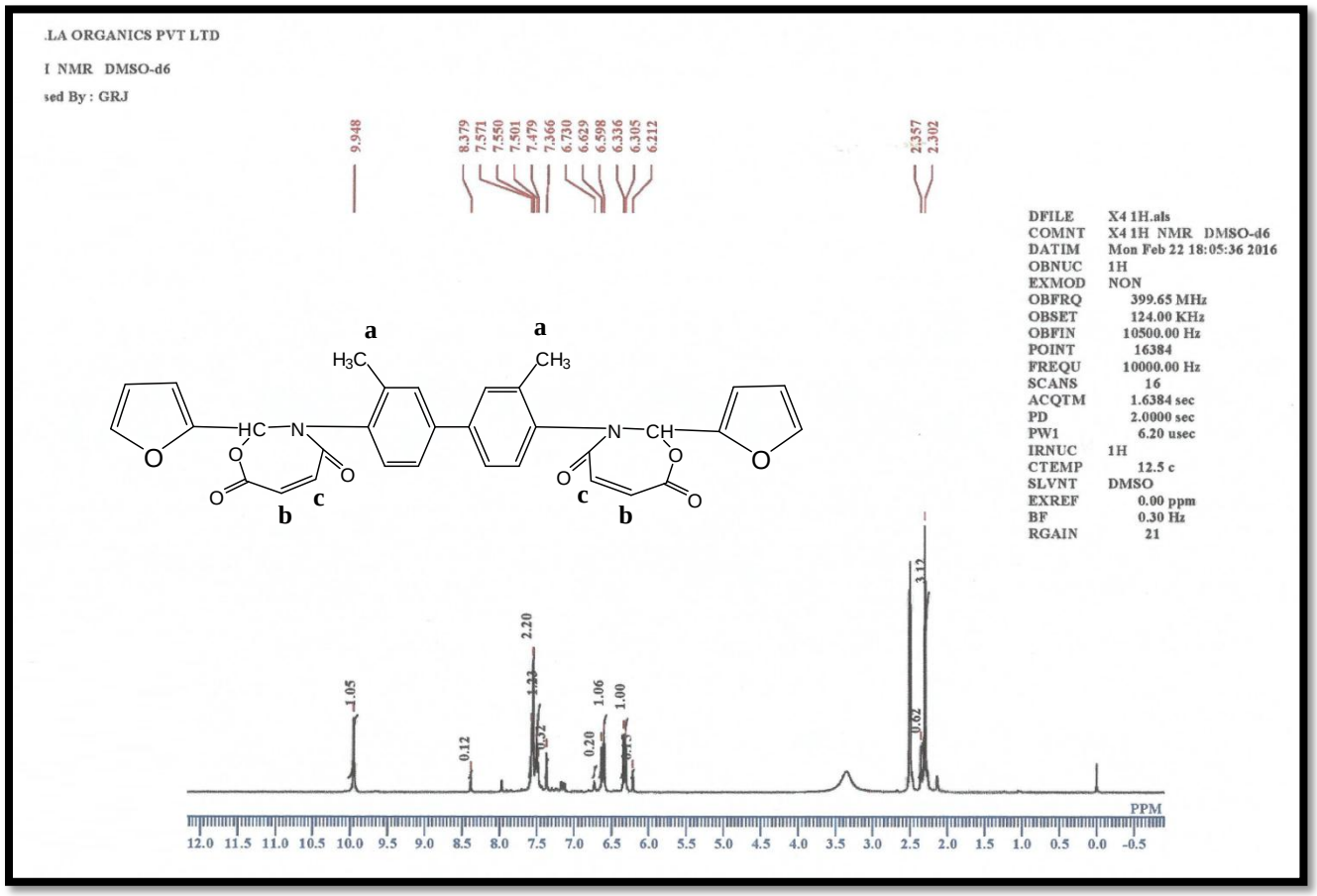


Fig.(20) :- ^{1}H NMR spectrum of compound (A8)

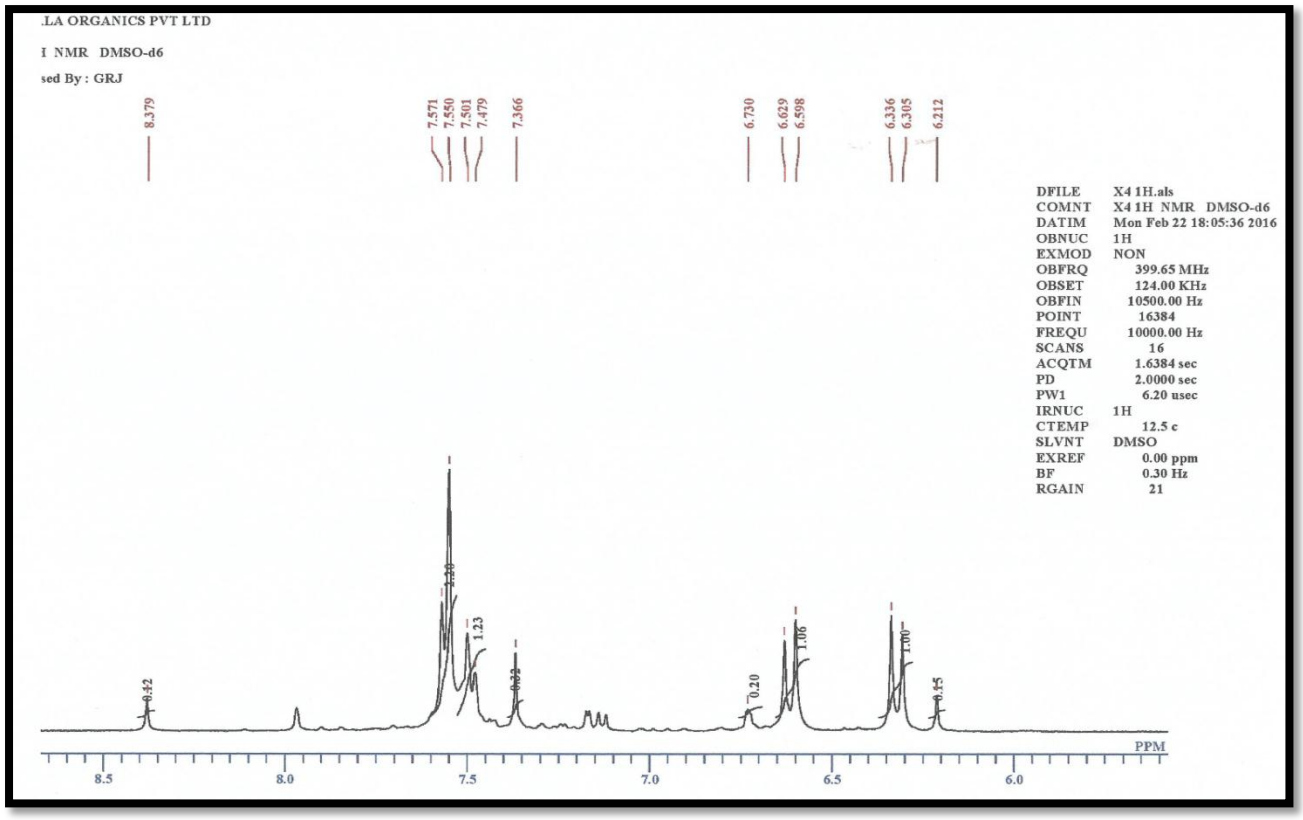


Fig.(20a) :- expanded ^{1}H NMR spectrum of compound (A8)

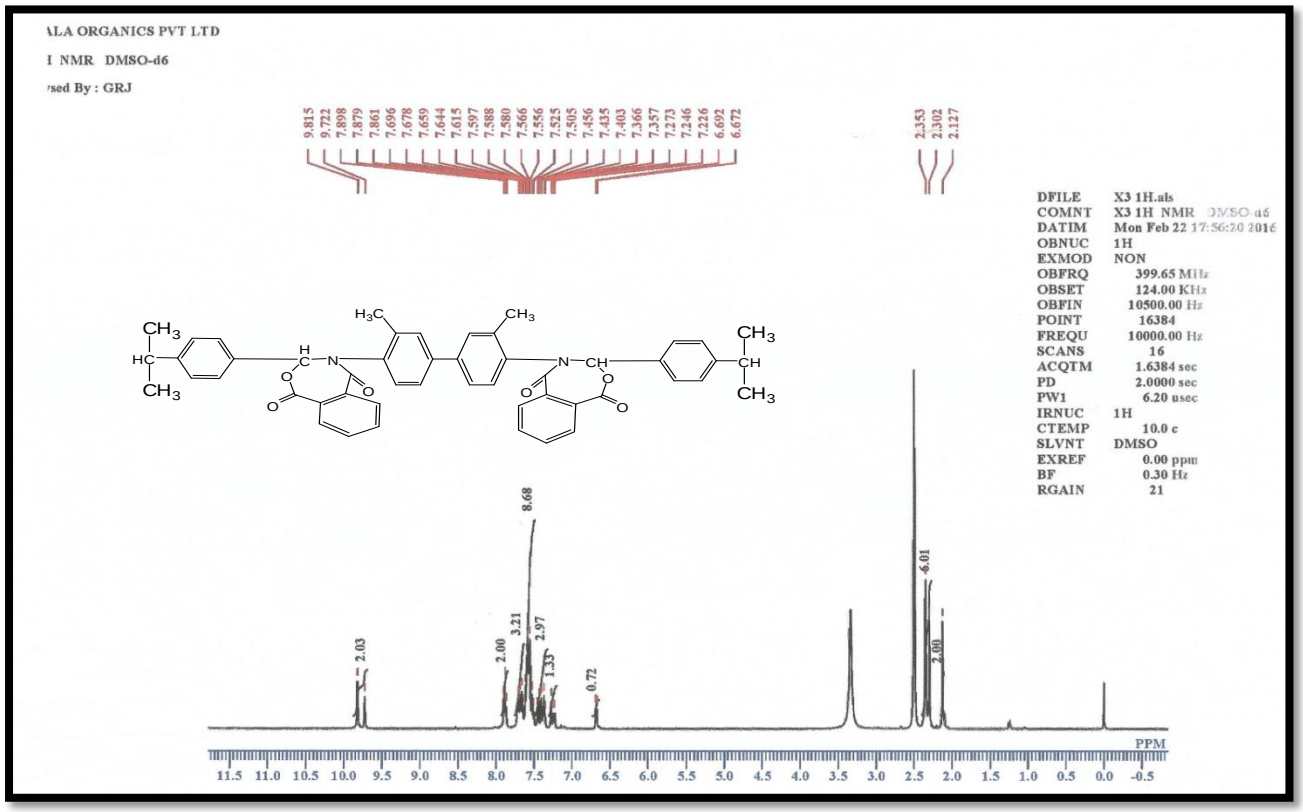


Fig.(21) :- ^{1}H NMR spectrum of compound (A10)

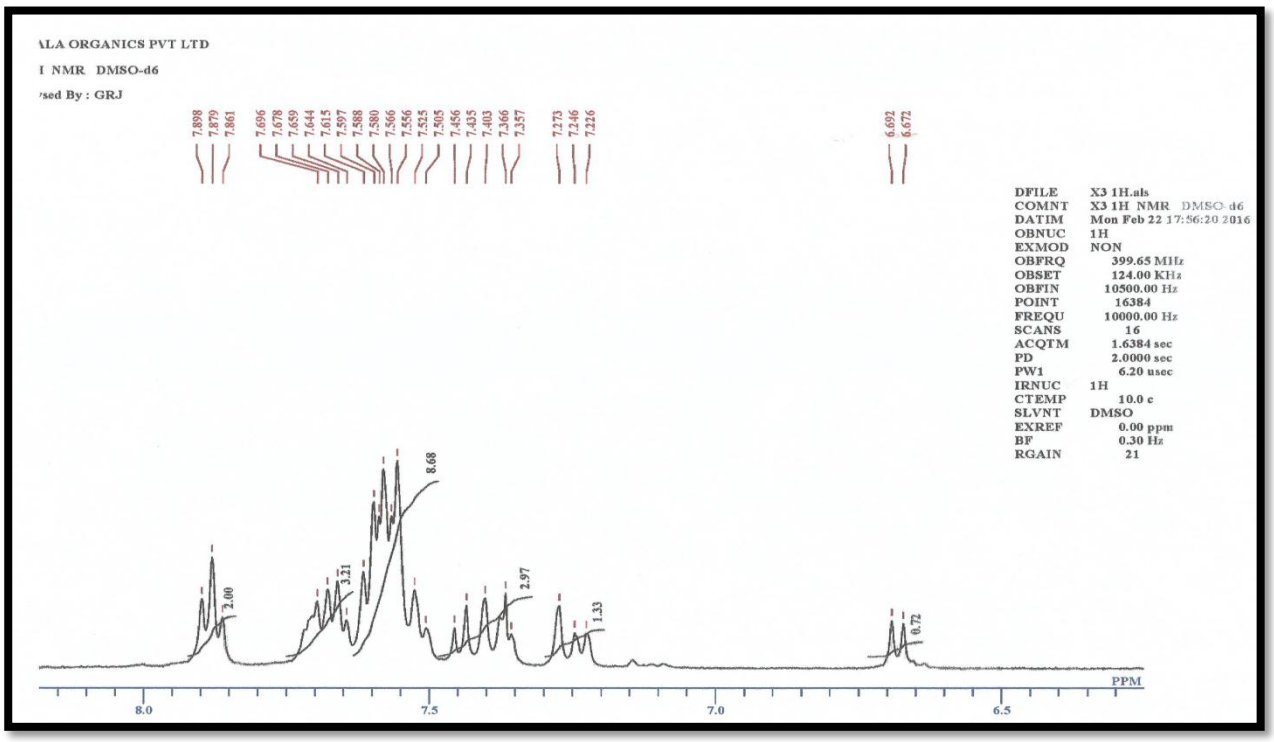


Fig.(21a) :- expanded ^{1}H NMR spectrum of compound (A10)

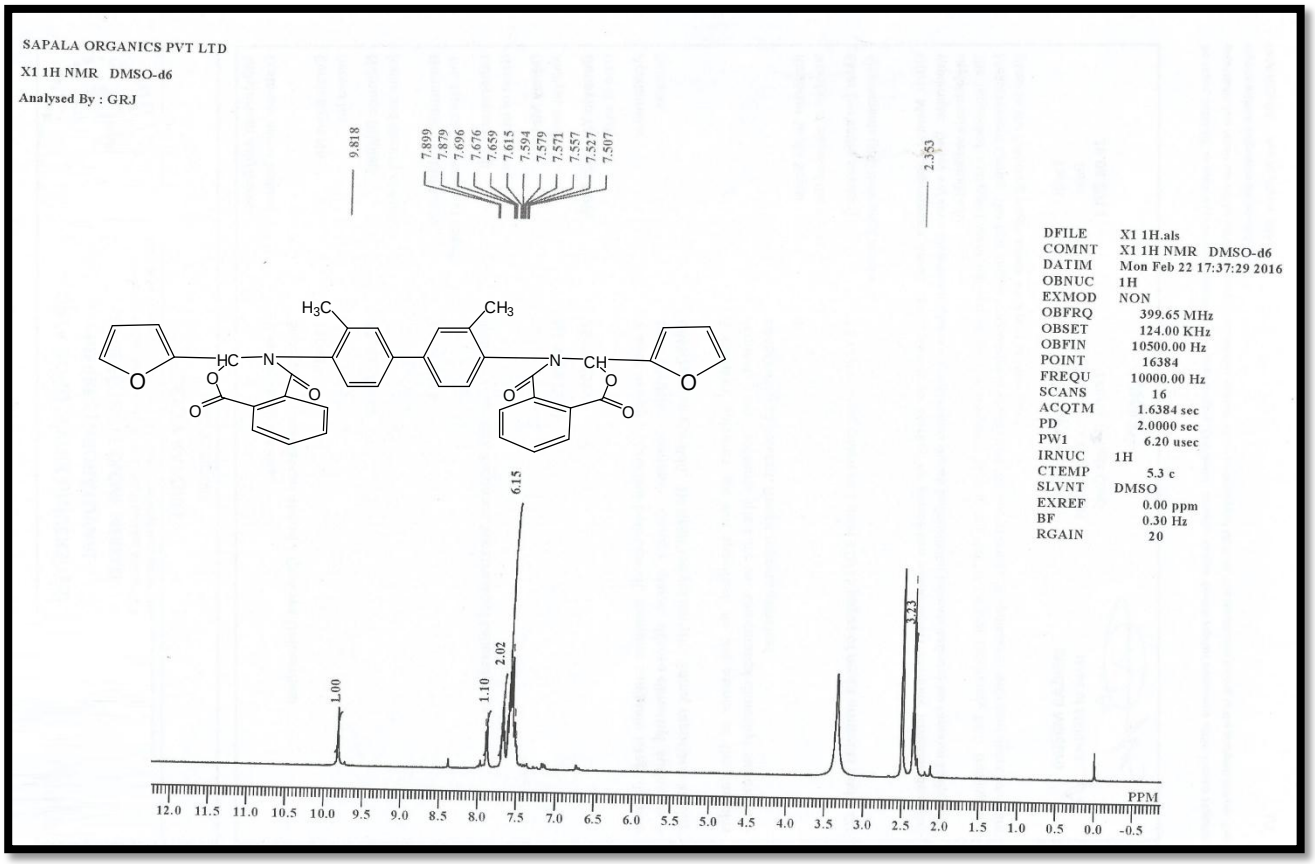


Fig.(22) :- ^{1}H NMR spectrum of compound (A11)

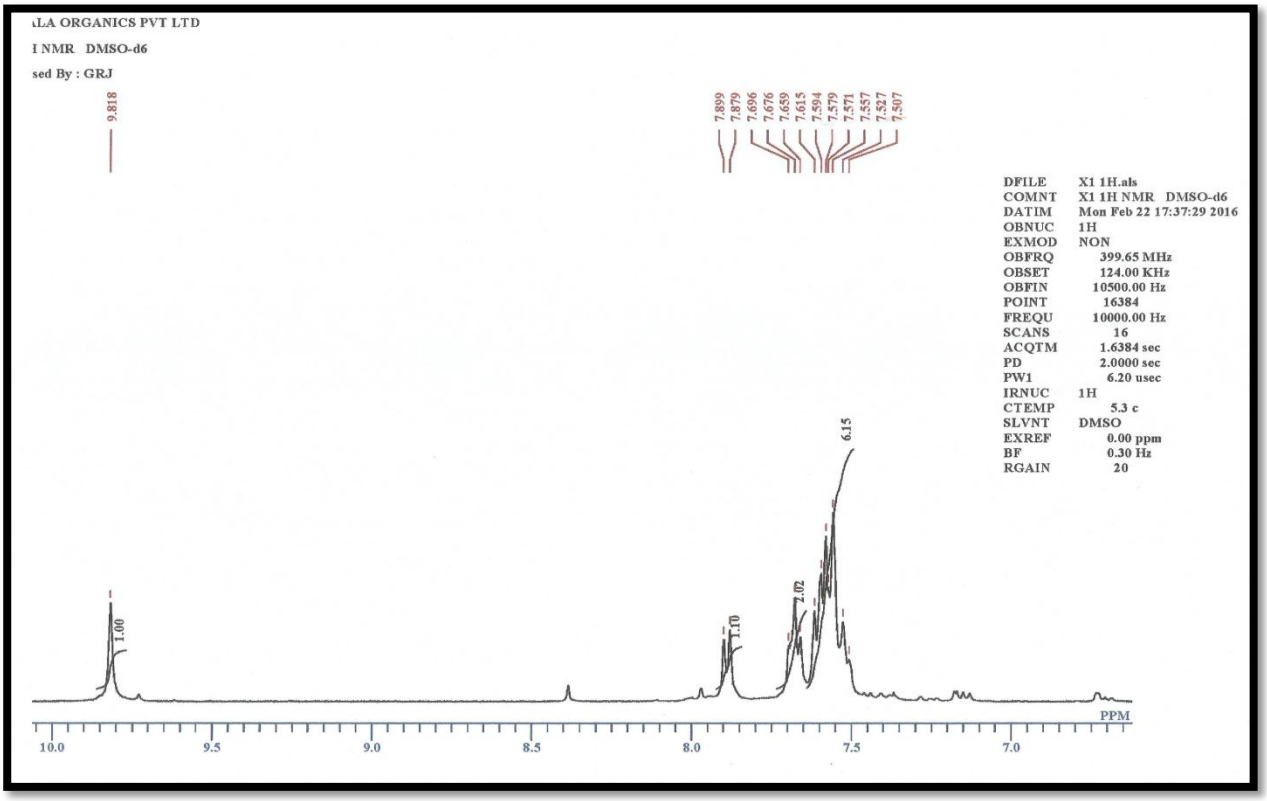


Fig.(22a) :- expanded ^{1}H NMR spectrum of compound (A11)

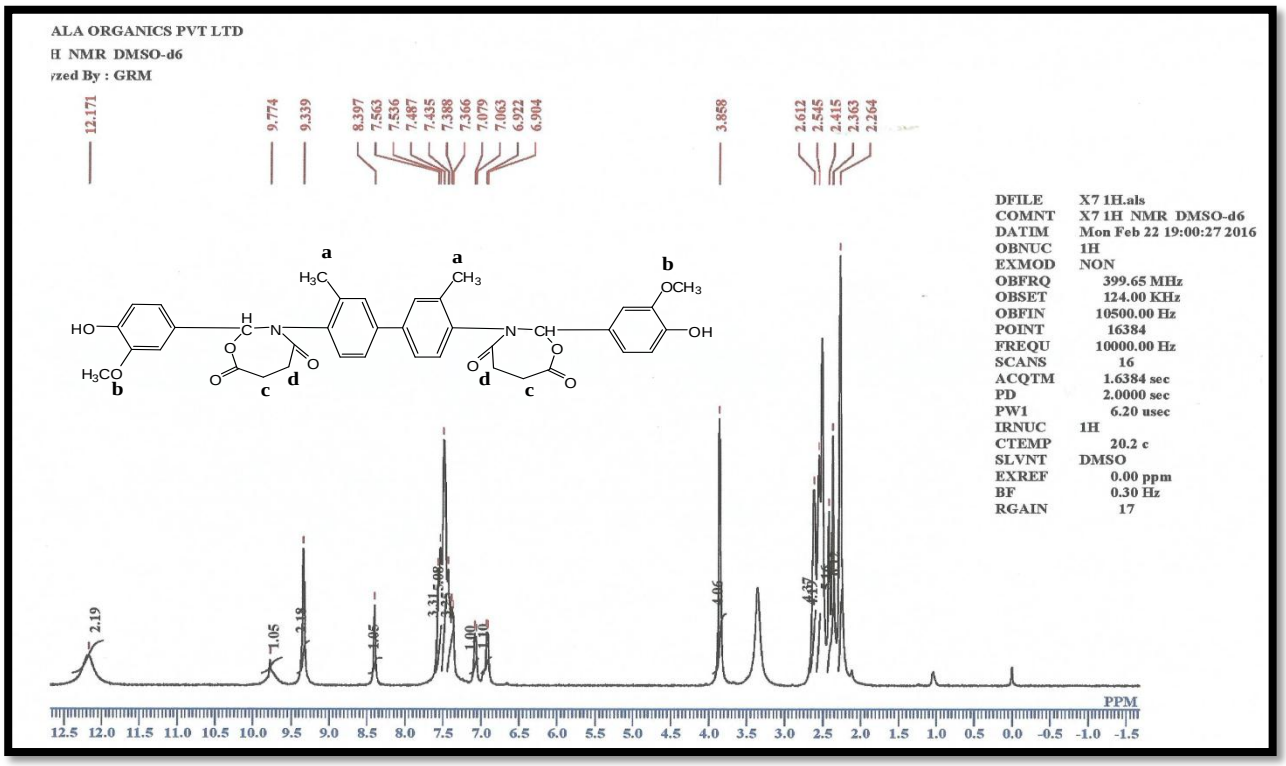


Fig.(23) :- ^{1}H NMR spectrum of compound(A12)

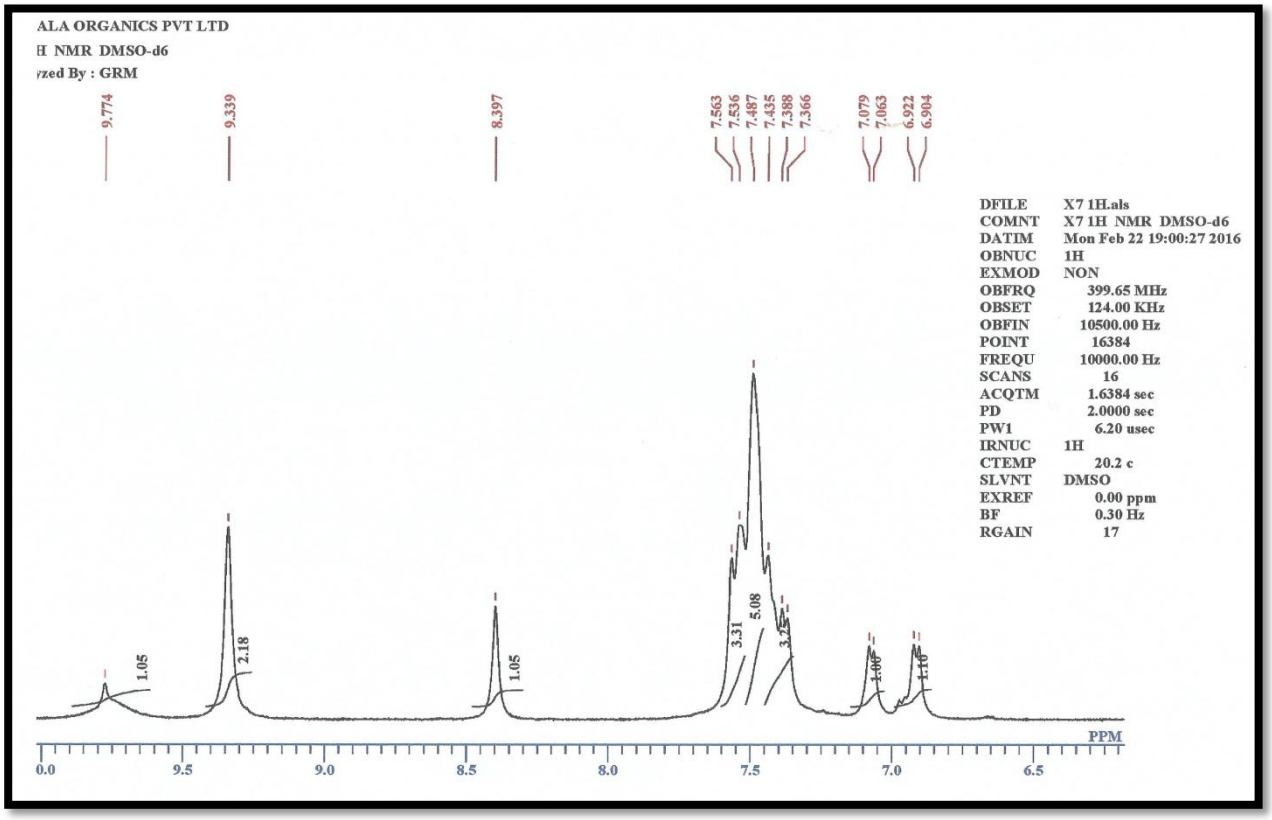


Fig.(23a) :- expanded ^{1}H NMR spectrum of compound (A12)

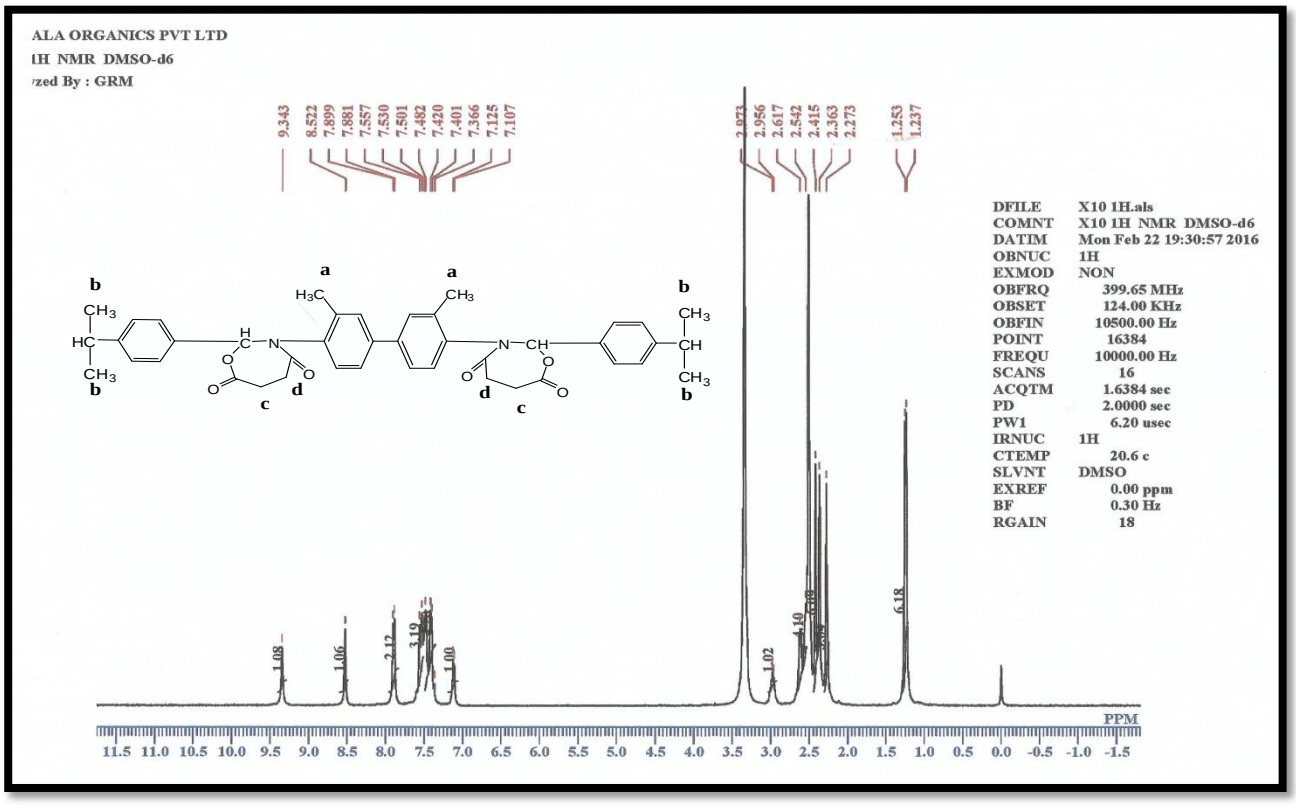


Fig.(24) :- ^{1}H NMR spectrum of compound(A13)

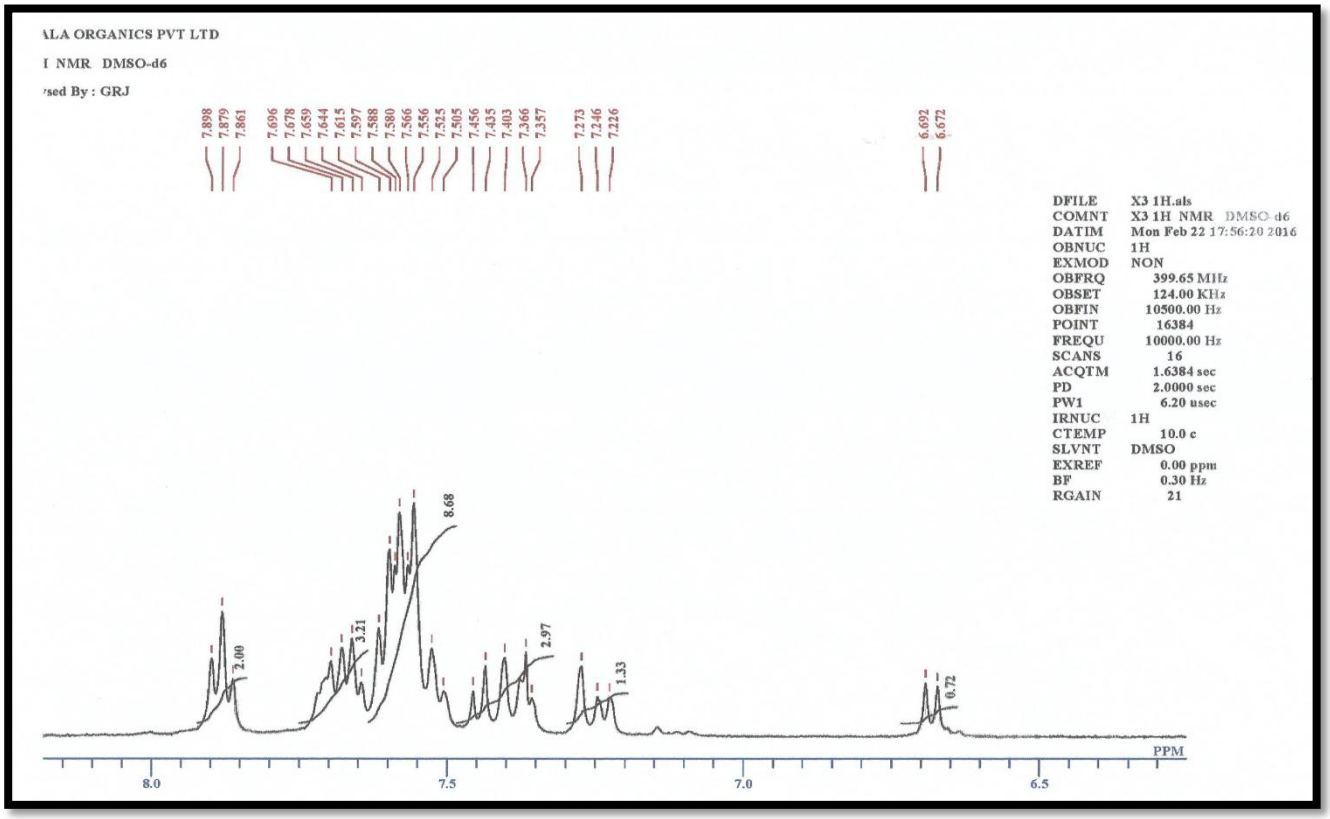


Fig.(24a) :- expanded ^{1}H NMR spectrum of compound (A13)

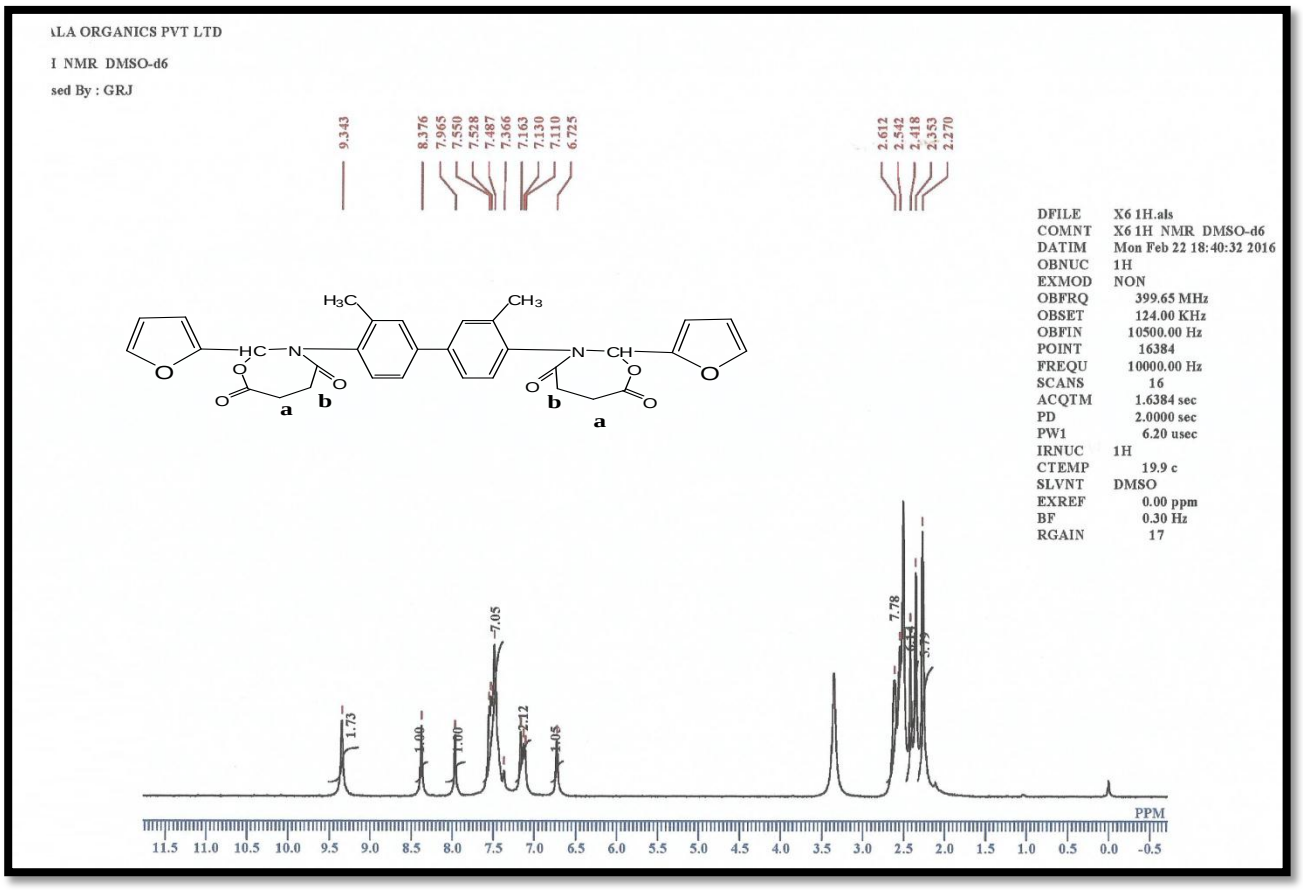


Fig.(25) :- ^{1}H NMR spectrum of compound(A14)

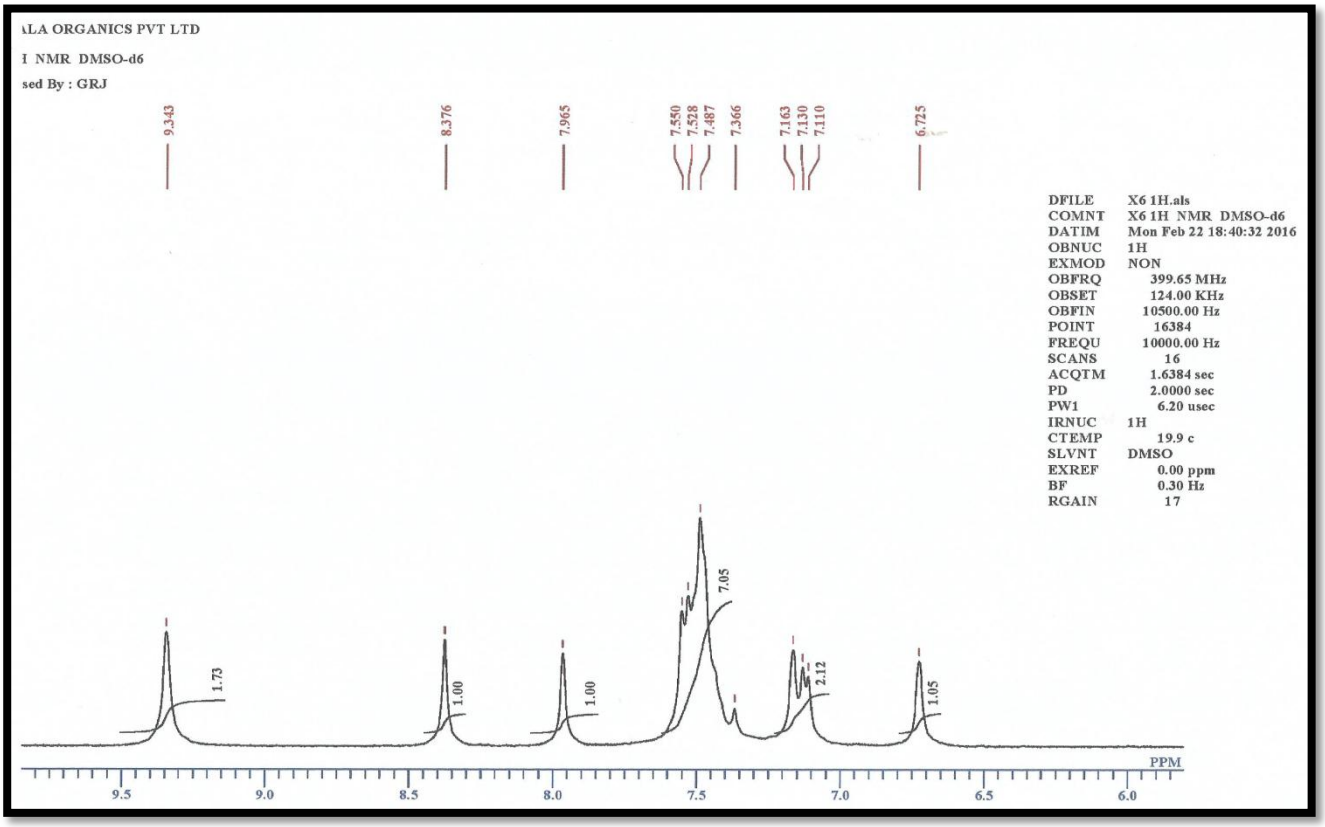


Fig.(25a) :- expanded ^{1}H NMR spectrum of compound (A14)

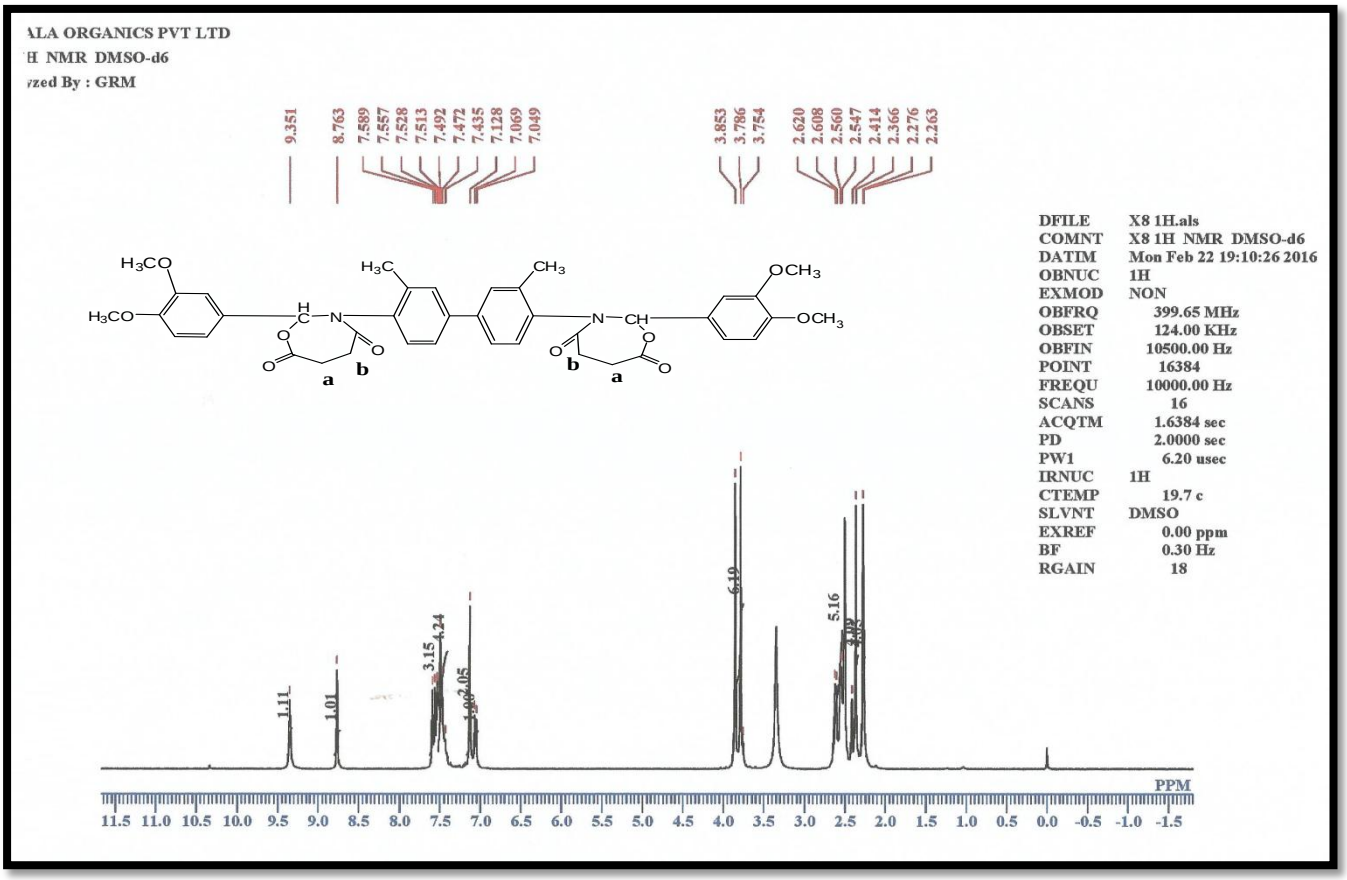


Fig.(26) :- ^{1}H NMR spectrum of compound(A15)

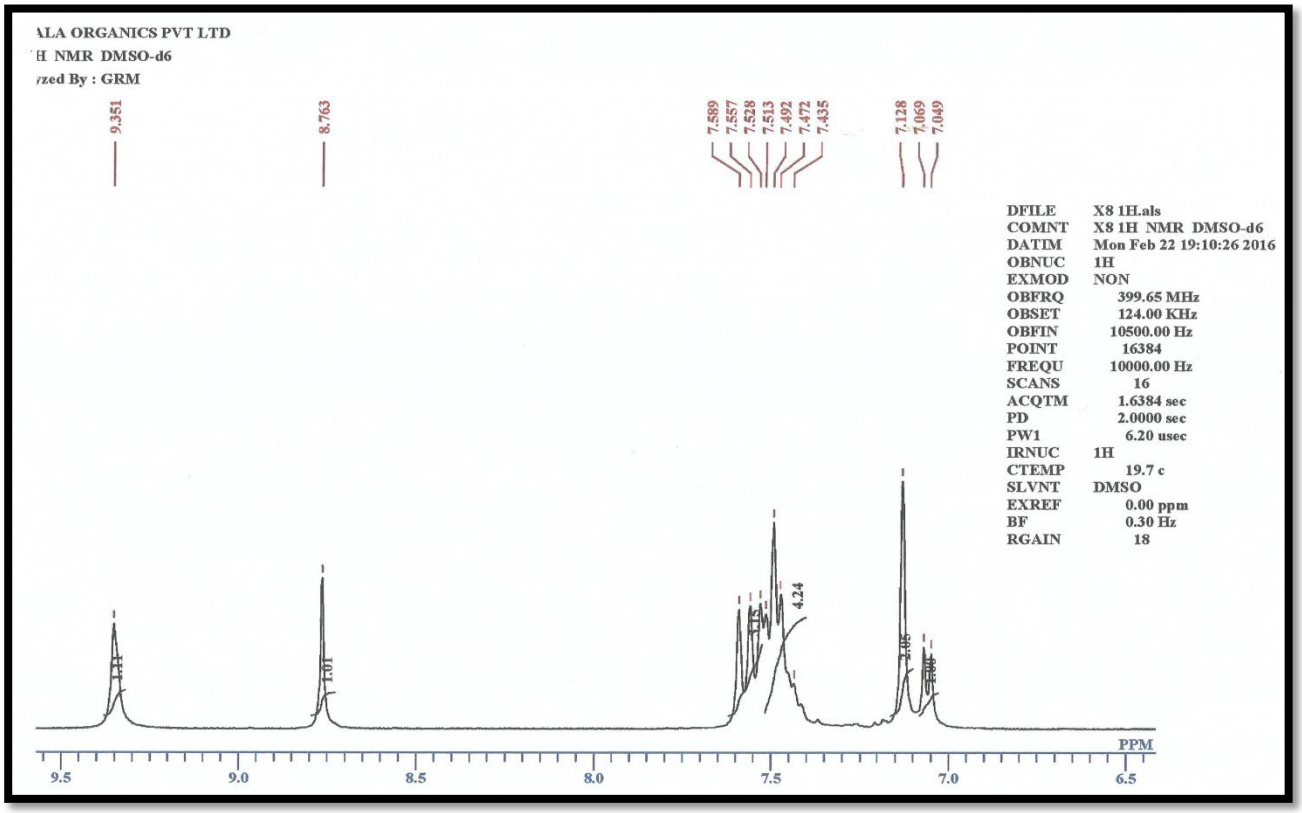


Fig.(26a) :- expanded ^{1}H NMR spectrum of compound (A15)

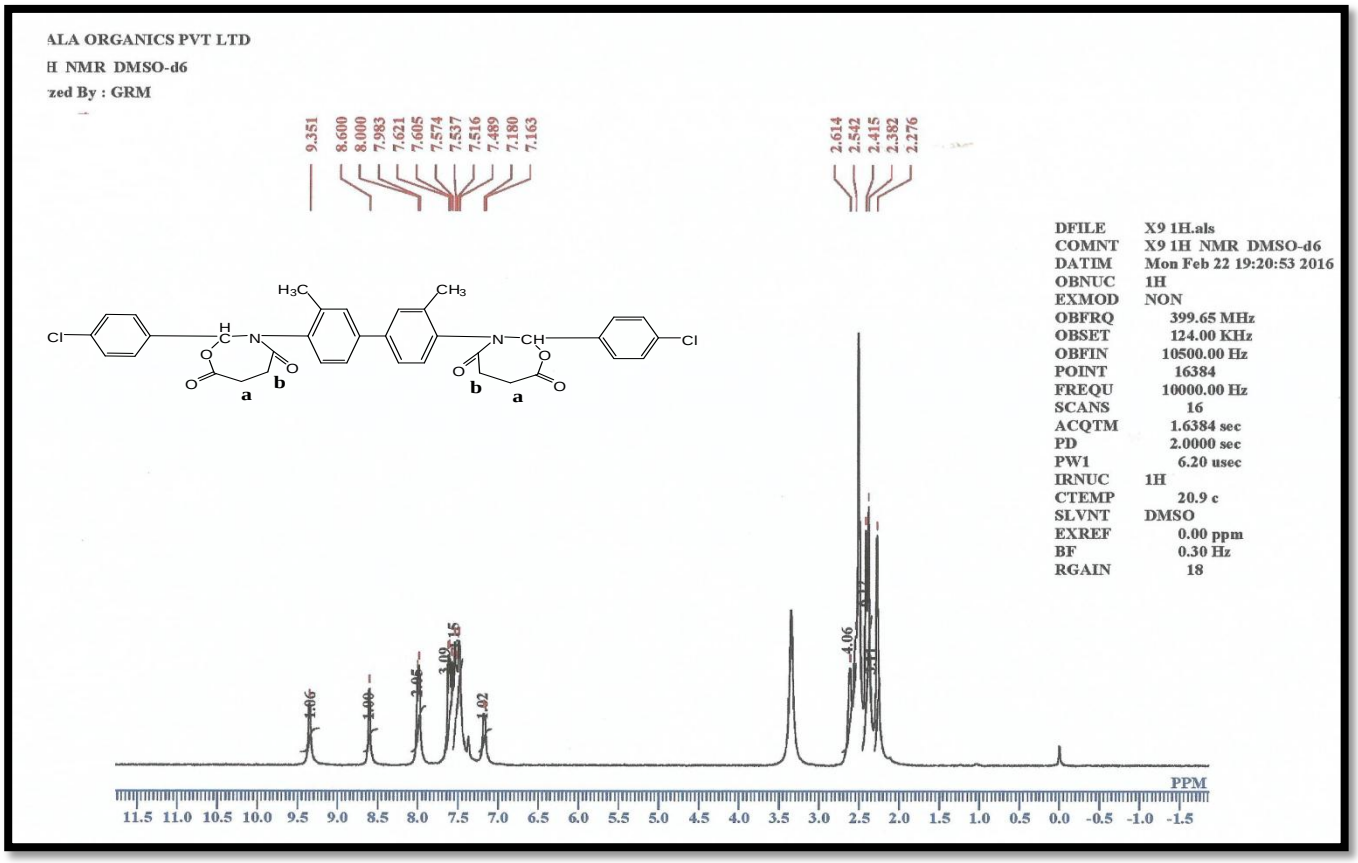


Fig.(27) :- ^{1}H NMR spectrum of compound (A16)

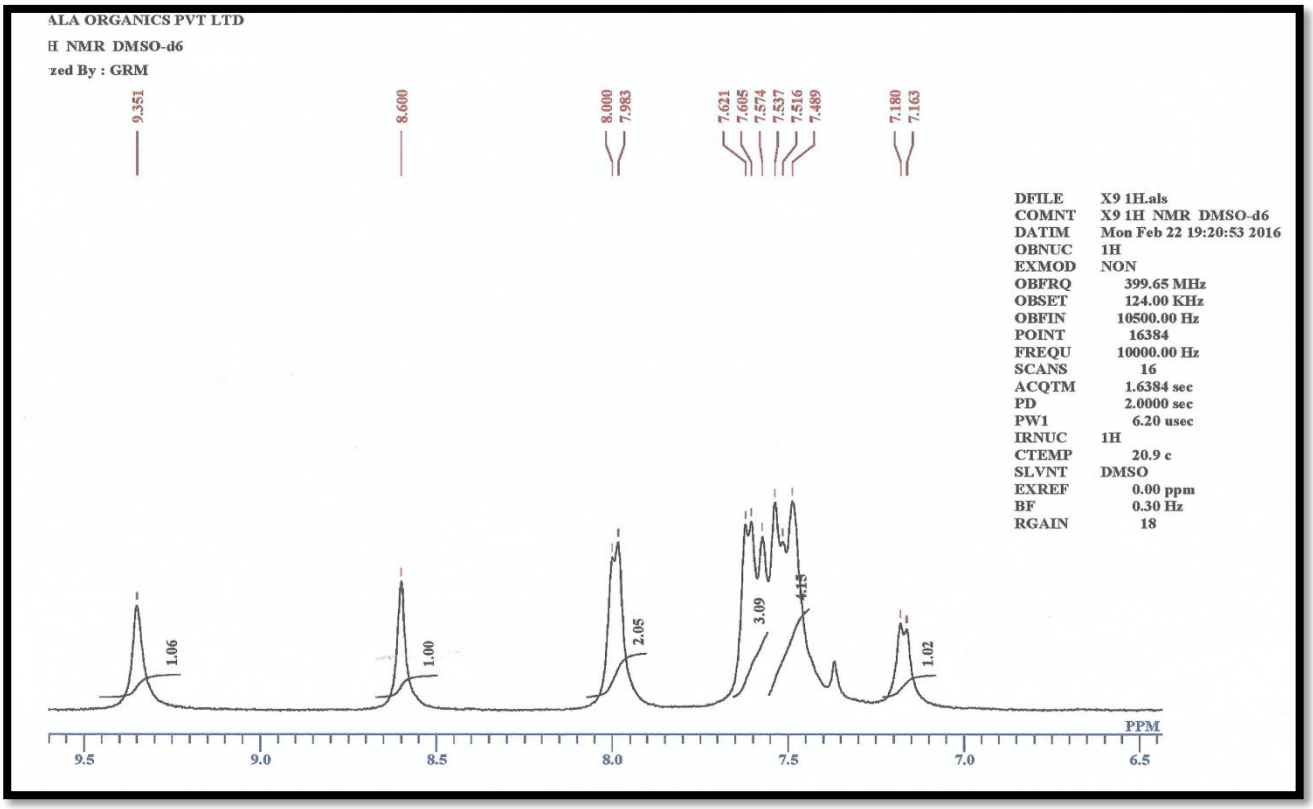


Fig.(27a) :- expanded ^{1}H NMR spectrum of compound (A16)

References :-

1- A. Noureen, S. Saleem, T. Fatima, H. M. Siddiqi and B. Mirza , ***Pak. J. Pharm. Sci.,*** 26, 1, 113,2013.

2- S. J. khammas , ***Tikrit Journal of Pure Science,*** 17 ,4, 2012.

3- A. A. Jarrahpour , M. Motamedifar , K. Pakshir, N. Hadi and M. Zarei **, Molecules,** 9, 815,2004.

4- K. F. Hamak and H. H. Eissa, ***International Journal of ChemTech Research,***5, ,6, 2924, 2013.

5- R. M. Al-Juburi ,**Journal of Al-Nahrain University,** 15 ,4, 60,2012.

6- Y.K. Gupta, S.C. Agarwal, S. P. Madnawat and R. Narain***, Research Journal of Chemical Sciences***, 2,4, 68, 2012.

7- A. W. Naser, H. T. Ghanem and A. M. Ali***, J. of university of anbar for pure science*** ,4,3 , 2010.

8- M. Zarei and A. Jarrahpour **,** ***Iranian Journal of Science & Technology***,3, 235 ,2011.

9- A.T. Mohammad, H, Osman and G.Y. Yeap ***, Australian Journal of Basic and Applied Sciences***, 5,3,192, 2011.

10- D.Sunil, C. Ranjitha, M. Rama and K. Pai***, International Journal of Innovative Research in Science Engineering and Technology,*** 3, 8, 2014.

11- Z. H. Abood and M. M. Hussein ***,Journal of Karbala University*** , 12 ,1 , 2014.

12- A. W.Naser and A.F. Abdullah **,** ***Journal of Chemical and Pharmaceutical Research***, 6,5,872,2014.

13- F. H. abanha and A. Shaabani **,** ***RSC Adv.*** , 4, 46844,2014.

14- H. Matsuzaki, I.Takeuchi, Y.Hamada and K. Hatano***, Chem. Pharm. Bull.*** 48,5,755 ,2000.

15- A.Hikmet, , K. Berat, , and B. Fatma**,** ***Medicinal Chemistry Research***,20, 1170, 2011.
16- X. Deng , C. Wei , F. Li , Z. Sun and Z. Quan **,** ***European, Journal of Medicinal Chemistry,*** 45 , 3080,2010.
17- J. K Mishra, K. Samanta, M. Jain, M. Dikshit, and G. Panda**,** ***Bioorg Med Chem Lett***., 20, 244, 2010.
18- Y. Ai , F. Song ,S.Wang ,Q. Sun and P.Sun ***,Molecules,*** 15,9364,2010.
19- F. F. Liegeois , F. A. Rogister, J. Bruhwyler, J. Damas‹T. P. Nguyen, M. O. Inarejos, E. M. G. Chleide‹ G. A. Mercier and J. E. Delarge**,** ***J. Med. Chem.,*** 37‹519, 1994.
20- R. Raja , M. Suresh , R. R. Nathan and A. S. Pandi **,** ***Acta Cryst. ,***70, 316,2014.
21- A. J .Khalaf , ***Molecules,*** 14, 2431, 2009.
22- X.Liu , Y. Jia , B.Song , Z.Pang and S. Yang ***,Bioorganic & Medicinal Chemistry Letters,*** 23 ,720, 2013.
23. K.F. Ali , ***Ph. D. Thesis,*** Baghdad University , 2005.
24- Z. H. Abood, ***J. Kerbala University,*** 7, 1, 297,2009.
25- R. T. Haiwal**,** ***J. Kerbala University,*** 6, 4, 216,2008.
26- R.M.Silverstein,G.C.Bassler and D.J.Kiemle"**Spectroscopic identification of Organic Compounds**"**2005**,7thEd., John Wiley and Sons, Inc,NJ,USA.