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Synthesis of some new heterocyclic compounds derived from 4-(4-hydroxy-3-chlorophenyl) azoaceto phenone

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Abstract

p-Aminoacetophenone underwent diazotization and coupling reaction with 2-chlorophenol to give a starting material 4-(4-hydroxy-3-chlorophenyl)azoacetophenone. The prepared starting material was benzylated with benzylbromide producing 4-[4-benzyloxy-3-chloro-phenyl]azoacetophenone. The later has been reacted with different substituted benzaldehydes affording a series of new chalcones. The newly synthesized chalcones were treated with hydrazine hydrate to form the desired molecules azo-benzyloxy-pyrazoline derivatives. The spectrum measurements of FT-IR, ^{1}H-NMR and ^{13}C-NMR, confirmed the expected structures of the newly synthesized compounds.

Introduction

p-Aminoacetophenone is one of the most important compounds that contain two functional (amino and acetyl) groups which can be used as versatile precursor to prepare a large number of organic compounds[1]. The diazotization of the amino group and its coupling reaction forms azo-linkage which combines two aromatic moieties (azobenzene derivatives). In addition the acetyl group undergoes addition reactions through which can be used as a precursor for further synthesis to give new organic molecules such as: azo- chalcone [2], and azo-imine [3].

Chalcones(α,β- unsaturated aromatic ketones) the main product of the condensation reaction of acetophenones and substituted benzaldehydes are a convenient precursors for the formation of a wide variety of heterocyclic compounds such as; oxazine, thiazine and isoxazole [4], pyrazoline [5], pyrimidines [6] and oxazolone [7]. Chalcones and pyrazolines along with corresponding azo-linkages are attracted much considerable attention due to a wide variety of biological effects which include: analgesic [8], antihistaminic [9], antioxidants and inflammatory [10] anti-fungal [11], and other antimicrobial activities [12]. In this work we have described synthesis, and characterization of some new azo-pyrazoline derivatives derived from p-aminoacetophenone.

Experimental

Melting points were determined using an electro thermal melting point apparatus. FT-IR spectra were recorded on IR Affinity-1 Spectrophotometer, using KBr disc. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker (400MHz) with TMS as an internal reference.

A. Synthesis of 4-(4-hydroxy-3-chlorophenyl)azoacetophenone (1):

Compound (1) was prepared by two steps according the reported procedure [13].

The solid azo dye was collected by vacuum filtrations, washed several times with water, dried and recrystallized from xylene to give (yellow-orange) crystals of compound(1): m.p.174-175^{o}C, yield (95%), IR(cm^{-1}): 3201(b), 1670, 1577, 1254. ^{1}H-NMR (ppm): 2.69(s, 3H, CH_3), 8.03(s 1H, OH), 7.21(d, 1H, H_{11}), 7.28(d, 1H, H_{12}), 7.96- 8.03(m 5H Ar-H).

^{13}C-NMR: 26.79(CH_3), 111.87(C_{11}), 117.5(C_{12}), 120.21($C_{3,5}$), 121.03(C_9), 125.36(C_8) 129.12($C_{2,6}$), 138.32(C_1), 147.06(C_7), 154.31(C_{10}), 155.73(C_4), 199.8(C=O).

B. Synthesis of 4-[4`-benzyloxy-3-chlorophenyl]azoacetophenone (2): [14]

A mixture of compound (1) (13.7gm, 50mmol), benzyl bromide (10.26gm, 60mmol) and anhydrous K_2CO_3 (9.562gm, 70mmol), in (60mL) ethanol was refluxed with stirring for 6hrs. The cooled solution poured into water, the solid material was obtained immediately. The precipitate was filtered off, washed several times by cold water dried and recrystallized from a mixture of xylene to give orange crystals of compound (2), m.p.138-140^{o}C, yield (93%), IR(cm^{-1}): 1670(C=O), 1589(C=C), 1249(C-O).

^{1}H-NMR (ppm): 2.68(s, 3H, $COCH_3$); 5.25(s, 2H, OCH_2 -C_{13}); 7.12-8.13(m 12H, Ar-H). ^{13}C-NMR: 26.56(CH_3), 71.16(C_{13}), 113.46(C_{11}), 122.86(C_{12}), 123.79($C_{3,5}$), 124.46(C_9), 125.40(C_8), 127.25($C_{15,19}$), 128.58(C_{17}), 128.96($C_{16,18}$), 129.27($C_{2,6}$), 135.92(C_{14}), 138.54(C_1), 146.90(C_7), 155.03(C_{10}), 157.07(C_4), 197.8(C=O).

C. Synthesis of chalcones: 1-(4-(4-benzyloxy-3 chlorophenyl)diazenyl) phenyl)-3-(substituted phenyl)prop-2-en-1-one [15] (3a-k):

A mixture of compound (2) (0.91gm, 0.003mol), alcoholic sodium hydroxide 4% (3mL), and substituted benzaldehydes (0.003mol) in ethanol (30mL) was stirred for (1-3 minutes) until all starting materials had reacted, the cooled mixture was solidified and filtered off, dried and recrystallized from a mixture of xylene-ethanol(1:10) to give(orange – yellow) colored products. The physical properties are outlined in Table (I).

Table I: Some physical properties and IR data for the prepared Azo-Chalcones (3a-k) Bz: $OCH_2C_6H_4$-

Yield %	M.P./^{o}C	C=C str. cm^{-1}	C=O str. cm^{-1}	Molecular formula	R	Prod.
81	159-161	1604	1656	$C_{28}H_{20}Cl_2N_2O_2$	2-Cl	3 a
98	212-213	1600	1658	$C_{28}H_{20}ClFN_2O_2$	4-F	3 b
78	164-166	1601	1654	$C_{29}H_{23}ClN_2O_3$	4-OCH_3	3 c
89	165-167	1601	1654	$C_{35}H_{27}ClN_2O_3$	3-Bz	3 d
76	162-164	1599	1654	$C_{35}H_{27}ClN_2O_3$	4-Bz	3 e
96	193-194	1602	1656	$C_{29}H_{23}ClN_2O_2$	4-CH_3	3 f
80	192-194	1604	1656	$C_{28}H_{21}ClN_2O_2$	H	3 g
74	176-178	1606	1658	$C_{28}H_{20}ClFN_2O_2$	2-F	3 h
88	232-233	1604	1654	$C_{28}H_{20}Cl_2N_2O_2$	4-Cl	3 i
81	190-192	1604	1656	$C_{35}H_{26}Cl_2N_2O_3$	3-(p-Cl-Bz)	3 j
85	205-207	1600	1654	$C_{35}H_{26}Cl_2N_2O_3$	4-(p-Cl-Bz)	3 k

3a: ^{1}H-NMR: 5.26 (s 2H -O-CH_2-C_{13}), 7.10-8.24 (m 16H Ar-H and 2H of CH-α and CH-β).
^{13}C-NMR: 70.97:O-CH_2-C_{13}, 112.95:C_{11}, 122.85:$C_{9,}$ 123.26:$C_{12,}$ 124.44:$C_{8,}$ 124.74:C_{α}, 125.35:$C_{3,5}$, 127.08:C_{24}, 127.21:$C_{15,19}$, 127.76:C_{17}, 128.13:$C_{25,}$ 128.64:$C_{22,}$ 129.56:$C_{16,18}$, 130.45:$C_{23,}$ 131.23:$C_{2,6}$, 133.22:C_{21}, 135.61:C_{20}, 135.82:C_{14}, 139.24:C_1, 140.93:C_{β}, 146.94:C_7, 156.94:C_{10}, 157.70:C_4, 189.67 C=O.
3b: ^{1}H-NMR: 5.26 (s 2H -O-CH_2-C_{13}), 7.01-8.14 (m 16H Ar-H and 2H of CH-α and CH-β).
^{13}C-NMR: 71.06:O-CH_2-C_{13}, 113.28:C_{11}, 116.31:$C_{22,24}$, 121.70:$C_{12,}$ 122.88:$C_{\alpha,}$ 123.50:$C_{9,}$ 124.52:$C_{3,5}$, 125.32:C_8, 127.08:$C_{15,19}$, 128.26:C_{17}, 128.73:$C_{21,25}$, 129.53:$C_{16,18}$, 130.48:$C_{2,6}$, 130.48:C_{20}, 136.12:C_{14}, 139.92:C_1, 143.90:C_{β}, 146.72:C_7, 154.84:C_{10}, 156.35:C_4, 163.45:C_{23}, 189.55 C=O

3c: ^{1}H-NMR: 3.89 (s 3H –O-CH_3-C_{26}), 5.29 (s 2H -O-CH_2-C_{13}), 6.97-8.09 (m 16H Ar-H and 2H CH-α and CH-β).
^{13}C-NMR: 55.45: OCH3-C26, 71.04:O-CH_2-C_{13}, 113.26:C_{11}, 114.51:$C_{22,24,}$ 119.64:C_{12}, 122.84:$C_{\alpha,}$, 123.48:$C_{9,}$ 124.38:$C_{3,5}$, 125.36:C_8, 127.10:$C_{15,19}$, 127.56:$C_{21,25}$, 128.27:C_{20}, 128.75:C_{17}, 129.47:$C_{16,18}$, 130.38:$C_{2,6}$, 135.86:C_{14}, 139.89:C_1, 145.15:C_{β}, 146.94: C_7, 154.61:C_{10}, 156.85:C_4, 161.87:C_{23}, 189.75 C=O.
3d: ^{1}H-NMR: 5.08 (s 2H -O-CH_2-C_{26}), 5.27 (s 2H -O-CH_2-C_{13}),7.01-8.13 (m 20H Ar-H and 2H of CH-α and CH-β).
^{13}C-NMR: 70.14:O-CH_2-C_{26}, 70.94:O-CH_2-C_{13}, 111.51: $C_{25,}$ 113.42:C_{11}, 115.43:C_{23}, 119.20:C_{21}, 120.41:$C_{\alpha,}$ 121.06:C_{12}, 122.87:$C_{9,}$ 123.62:$C_{3,5}$, 124.92:C_8, 127.08:$C_{28,32}$, 127.39:$C_{15,19}$, 127.95:C_{30}, 128.10:C_{17}, 128.36:$C_{29,31}$, 128.51 :$C_{16,18}$, 129.51:C_{22}, 129.85:$C_{2,6}$, 133.27:C_{20}, 135.12:C_{27}, 135.85:C_{14}, 139.44 :C_1, 145.32:C_{β}, 147.12:C_7, 154.17:C_{10}, 157.25:C_4, 160.34:C_{24}, 189.68 C=O
3f: ^{1}H-NMR: 2.39 (s 3H -Ar-CH_3-C_{26}), 5.26 (s 2H -O-CH_2-C_{13}), 7.10-8.32 (m 16H Ar-H and 2H CH-α and CH-β).
^{13}C-NMR: 21.65:C_{26}, 71.21:O-CH_2-C_{13}, 113.31:C_{11}, 120.95:$C_{12,}$ 122.86:$C_{\alpha,}$ 123.10:$C_{9,}$ 124.28:$C_{3,5}$, 125.26:C_8, 127.14:$C_{21,25}$,128.12:$C_{15,19}$, 128.48:C_{17}, 128.83:$C_{22,24,}$ 129.25:$C_{16,18}$, 129.78:$C_{2,6}$, 131.95:C_{20}, 135.85:C_{14}, 138.96:C_{23}, 139.71:C_1, 144.85:C_{β}, 146.93:C_7, 155.97:C_{10}, 156.86:C_4, 189.88 C=O

D. Synthesis of pyrazolines: 3-(4-(4-benzyloxy-3-chlorophenyldiazenyl) phenyl)-5-(substituted phenyl)- pyrazoline [16] (4a-k):

A mixture of chalcone derivative (1mmole), hydrazine hydrate (2mmoles) and alcoholic sodium hydroxide 4% (1mL) in ethanol (15mL) was refluxed with stirring for appropriate time until completion the reaction which was monitored by either change of the color or the formation of ppt. The ppt. was isolated by suction filtrations, washed with ethanol, dried and purified by recrystallization from xylene as suitable solvent. The physical properties of the prepared azo-pyrazolines (4a-k) are summarized in Table. II

Table II: Some physical properties and IR data for the prepared azo pyrazolines (4a-k)

Prod.	R	Molecular formula	C=N str. cm^{-1}	N-H str. cm^{-1}	Time hrs.	M.P. ^{o}C	Yield %
3 a	2-Cl	$C_{28}H_{22}Cl_2N_4O$	1593	3311	4	149-151	98
3 b	4-F	$C_{28}H_{22}ClFN_4O$	1591	3340	3.5	139-141	96
3 c	4-OCH_3	$C_{29}H_{25}ClN_4O_2$	1591	3336	6	129-131	90
3 d	3-Bz	$C_{35}H_{29}ClN_4O_2$	1591	3340	6	143-144	81
3 e	4-Bz	$C_{35}H_{29}ClN_4O_2$	1591	3327	6	121-123	77
3 f	4-CH_3	$C_{29}H_{25}ClN_4O$	1593	3340	5	137-139	73
3 g	H	$C_{28}H_{23}ClN_4O$	1595	3334	5	125-127	70
3 h	2-F	$C_{28}H_{22}ClFN_4O$	1591	3340	4.5	155-157	88
3 i	4-Cl	$C_{28}H_{22}Cl_2N_4O$	1593	3338	4	143-145	96
3 j	3-(p-Cl-Bz)	$C_{35}H_{28}Cl_2N_4O_2$	1593	3337	5	152-154	72
3 k	4-(p-Cl-Bz)	$C_{35}H_{28}Cl_2N_4O_2$	1593	3334	6	168-170	83

4a: ^{1}H-NMR: 2.93 (dd 1H CH_2-H_a), 3.67 (dd 1H CH_2-H_b), 5.28 (s 2H -O-CH_2-C_{13}), 5.38 (dd 1H CH-H_x), 6.16-8.05 (m 16H Ar-H), 6.16 (s 1H N-H).
^{13}C-NMR: 39.70: CH_2 of pyra., 61.21: CH of pyra., 71.02: O-CH_2-C_{13}, 113.27: C_{11}, 123.11: C_{12}, 123.31: C_9, 124.28: $C_{3,5}$, 124.80: C_8, 126.66: C_{24}, 127.02: $C_{15,19}$, 127.14: C_{17}, 127.30: C_{23}, 128.20: C_{25}, 128.71: C_{22}, 128.82: $C_{16,18}$, 129.67: $C_{2,6}$, 132.75: C_{21}, 135.12: C_1, 135.97: C_{14}, 139.69: C_{20}, 147.05: C_7, 150.25: C=N , 152.32: C_4, 156.35: C_{10}.

4b: ^{1}H-NMR: 3.07 (dd 1H CH_2-H_a), 3.61 (dd 1H CH_2-H_b), 5.46 (s 3H -O-CH_2-C_{13} and CH-H_x), 7.11-8.18 (m 16H Ar-H), 5.67 (s 1H N-H).
^{13}C-NMR: 39.72: CH_2 of pyra., 57.66: CH of pyra., 71.05: O-CH_2-C_{13}, 113.31: C_{11}, 115.38: $C_{22,24}$, 122.80: C_{12}, 123.30: C_9, 124.28: $C_{3,5}$, 124.82: C_8, 126.68: $C_{15,19}$, 127.37: C_{17}, 128.20: $C_{21,25}$, 128.33: $C_{16,18}$, 129.28: $C_{2,6}$, 135.04: C_1, 135.71: C_{20}, 135.80: C_{14}, 146.89: C_7, 150.21: C=N , 152.32: C_4, 156.45: C_{10}, 161.54: C_{23}.

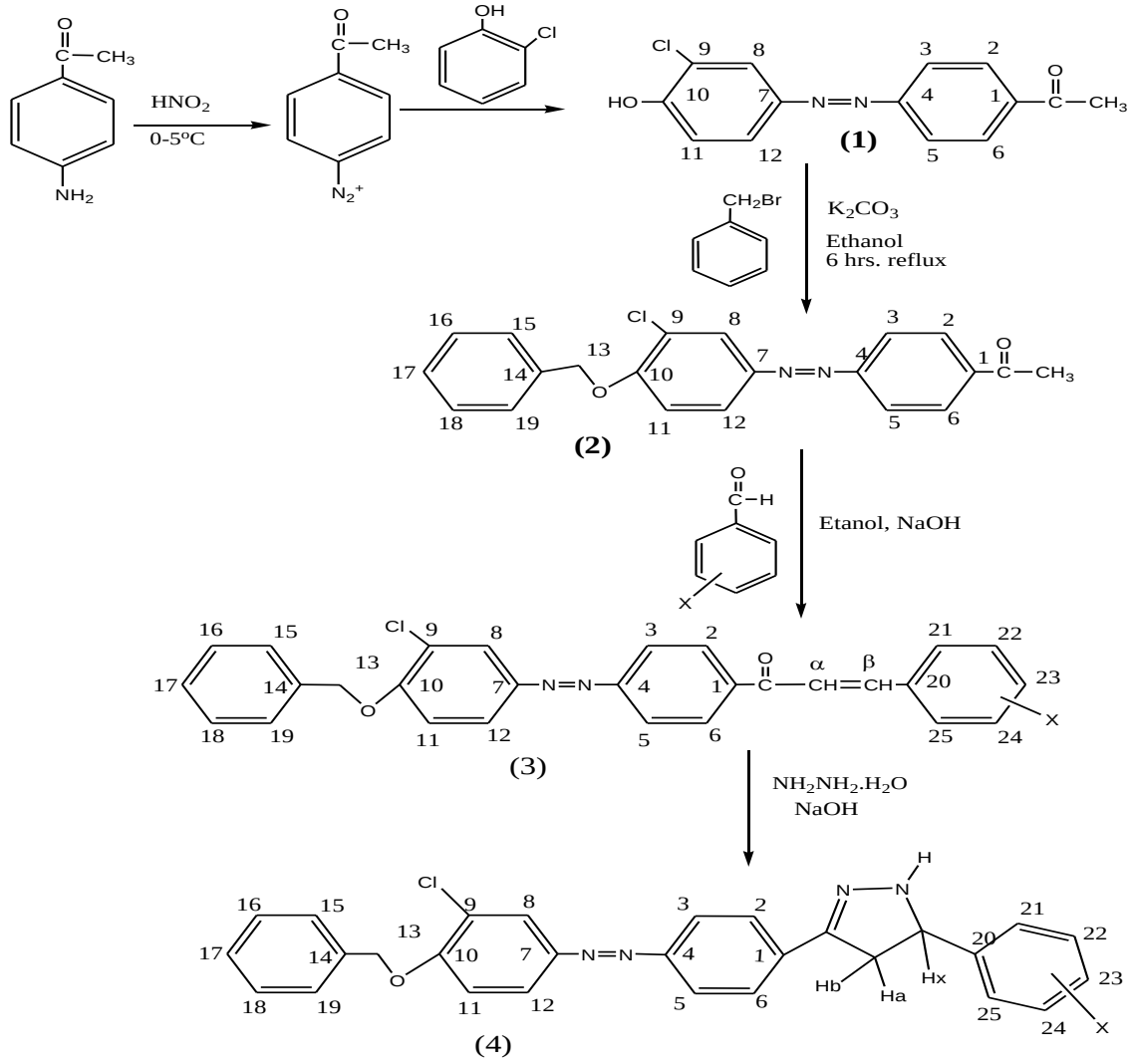
4c: ^{1}H-NMR: 3.10 (dd 1H CH_2-H_a), 3.54 (dd 1H CH_2-H_b), 3.88 (s 3H –OCH_3 C_{26}), 4.94 (dd 1H CH-H_x), 5.28 (s 2H-O-CH_2-C_{13}), 6.87-8.06 (m 16H Ar-H + 1H-N-H).
^{13}C-NMR: 41.13: CH_2 of pyra., 55.35: OCH_3-C_{26}, 64.14: CH of pyra., 71.02: O-CH_2-C_{13}, 113.31: C_{11}, 114.47: $C_{22,24}$, 122.92: C_{12}, 123.29: C_9, 124.28: $C_{3,5}$, 124.82: C_8, 126.64: $C_{15,19}$, 127.10: C_{17}, 127.52: $C_{21,25}$, 128.21: $C_{16,18}$, 128.72: $C_{2,6}$, 134.64: C_{20}, 135.29: C_1, 135.98: C_{14}, 147.07: C_7, 150.28: C=N , 152.26: C_4, 156.35: C_{10}, 159.30: C_{23}.

4d: ^{1}H-NMR: 3.10 (dd 1H CH_2-H_a), 3.55 (dd 1H CH_2-H_b), 5.09 (dd 1H CH-H_x), 5.16 (s 2H -O-CH_2-C_{26}), 5.29 (s 2H -O-CH_2-C_{13}), 6.93-7.89 (m 21H Ar-H + 1H N-H).
^{13}C-NMR: 41.12: CH_2 of pyra., 63.85: CH of pyra., 70.62: O-CH_2-C_{26}, 71.05: O-CH_2-C_{13}, 112.12: C_{25}, 113.21: C_{11}, 116.43: C_{23}, 119.27: C_{21}, 122.89: C_{12}, 123.28: C_9, 124.12: $C_{3,5}$, 124.88: C_8, 127.09: $C_{28,32}$, 127.53: $C_{15,19}$, 128.21: C_{30}, 128.27: C_{17}, 128.60: $C_{29,31}$, 128.67: $C_{16,18}$, 128.75: $C_{2,6}$, 129.44: C_{22} 135.62: C_1, 136.50: C_{14}, 136.25: C_{27}, 144.25: C_{20}, 146.86: C_7, 149.92: C=N, 151.95: C_4, 157.12: C_{10}.
4e: ^{1}H-NMR: 3.11 (dd 1H CH_2-H_a), 3.51 (dd 1H CH_2-H_b), 4.96 (dd 1H CH-H_x), 5.15 (s 2H -O-CH_2-C_{26}), 5.28 (s 2H -O-CH_2-C_{13}), 6.98-8.10 (m 21H Ar-H-C+ 1H N-H).
^{13}C-NMR: 41.06: CH_2 of pyra., 64.08: CH of pyra., 70.05: O-CH_2-C_{26}, 70.97: O-CH_2-C_{13}, 113.26: C_{11}, 115.17: $C_{22,24}$, 122.80: C_{12}, 123.08: C_9, 123.25: $C_{3,5}$, 124.77: C_8, 126.59: $C_{28,32}$, 127.05: $C_{15,19}$, 127.40: C_{30}, 127.50: C_{17}, 127.97: $C_{21,25}$, 128.17: $C_{29,31}$, 128.39: $C_{16,18}$, 128.58: $C_{2,6}$, 134.89: C_{20}, 135.23: C_1, 135.93: C_{27}, 136.86: C_{14}, 147.02: C_7, 150.57: C=N, 152.21: C_4, 156.75: C_{10}, 159.73: C_{23}.

III. Results and Discussion

A series of new heterocyclic compounds azo-pyrazoline derivatives have been synthesized in high yields, and characterized by using different spectral methods like FTIR, ^{1}H-NMR and ^{13}C-NMR. The Schematic diagram of the preparation processes are outlined in the following Scheme (1).
The existence of azo-linkage, carbonyl and hydroxyl groups in compound (1) and the disappearance of the indicated hydroxyl group and shifting the carbonyl group in the compound (2) are characterized and proved according to the obtained spectral data's [17].
The most important evidence for the formation of the synthesized chalcones are the shifting of the absorption band of carbonyl group in the IR spectra and a characteristic deshielding signals for β- protons at aromatic region, in^{1}H-NMR and (140-145ppm) in ^{13}C-NMR spectral data's [18].
The characterization of the synthesized pyrazoline derivatives, are confirmed by the disappearance of carbonyl group band at 1655 cm^{-1} for enone system in the IR spectra Figure (1) and formation of the five membered cyclic imine system [19]. The ^{1}H-NMR spectra Figure (2) of pyrazoline ring show a very distinct signals for the protons attached to the Hx and Ha & Hb carbon atoms in the 2-pyrazoline as an (ABX) spin system, which appeared three doublet to doublet (dd) signals around δ 3 , 4, and 5 ppm for two geminal and

one vicinal protons unequivocally prove a 2-pyrazoline structure. In addition the ^{13}C- NMR spectra Figure (3) show two distinct signals for two carbon atoms around 40 and 60 ppm of the pyrazoline ring and the disappearance of two singlets for C-α and C-β of intermediate chalcones confirm the 2-pyrazoline structure[20].



Scheme (1)

X= H, 2-Cl, 4-CH_3, 3-ClPhOCH_2-, 4-ClPhOCH_2-, 4-F, 4-Cl, 2-F, 4-OCH_3, 4-$N(CH_3)_2$, 4-PhOCH_2-, 3-PhOCH_2-

IV. Conclusions

The starting material p-aminoacetophenone can interact and use both functional groups separately for the preparation purposes depending on the medium and utilized reagents. As a result it can get a huge number of new organic compounds. As in this work we got the azo linkage from the amino group and the chalcone moiety from the other side (acetyl group). Accordingly we were synthesized a high percentage of new pyrazoline derivatives.

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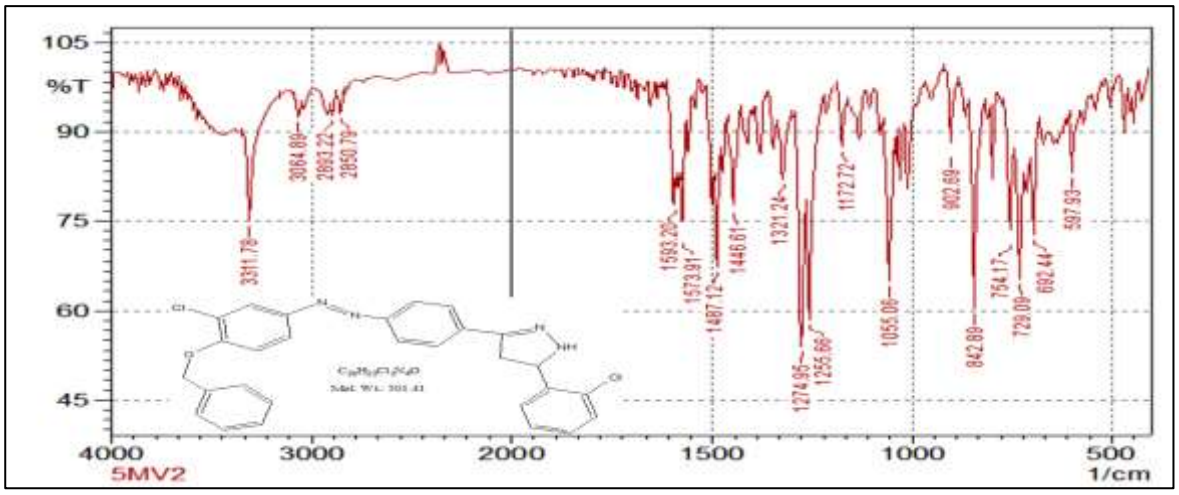

Figure 1: IR spectrum of compound 4a

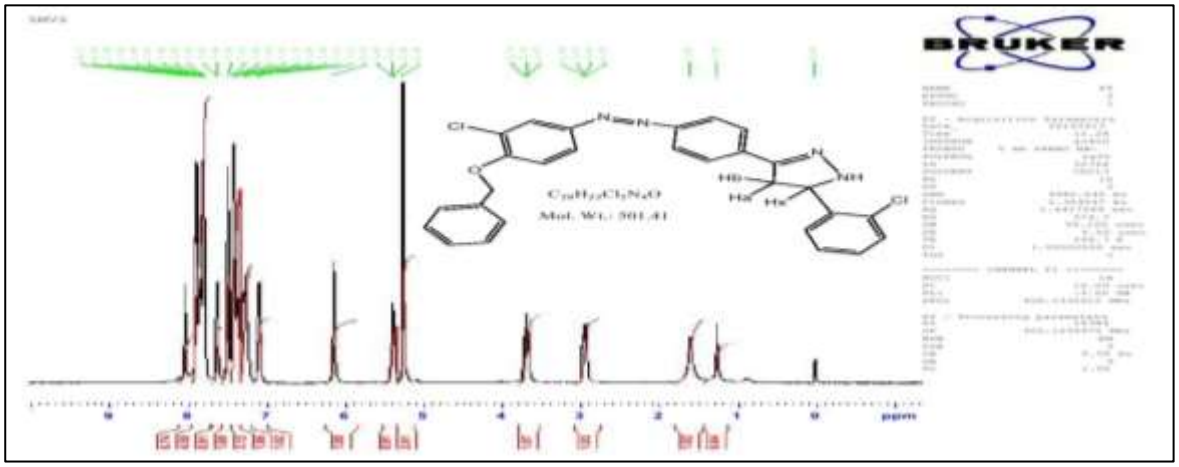

Figure 2: ^{1}H-NMR Spectrum of compound 4a

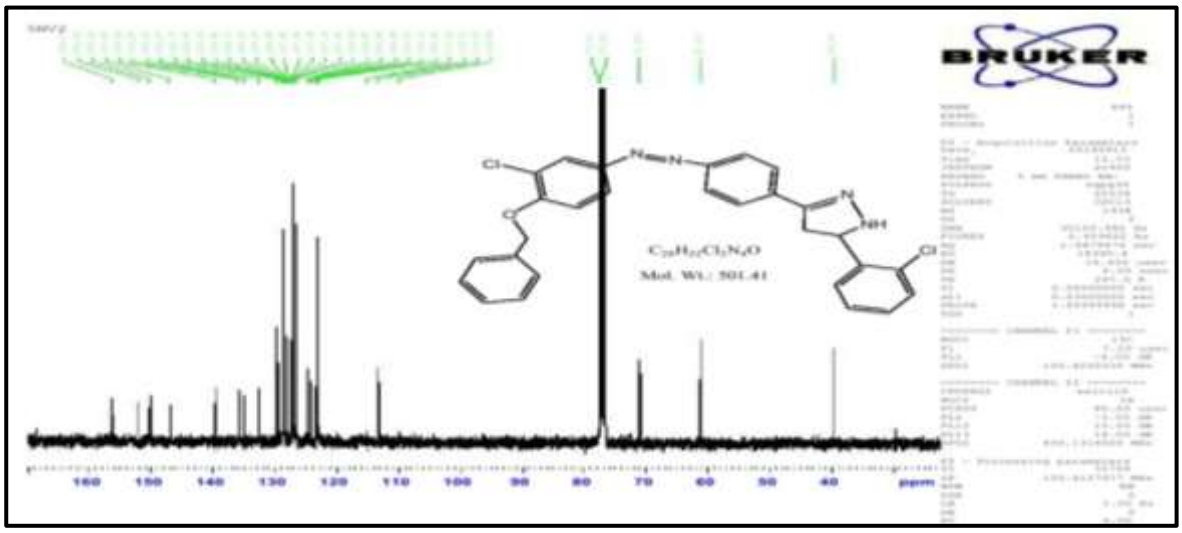

Figure 3: ^{13}C-NMR Spectrum of compound 4a

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