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Formation of 1,5-Disubstituted 2-hydroxypyrroles via Reaction of 2-(Triphenylphosphoranylidene) succinic Anhydride with Schiff's Bases

Obaid H. Abid* & Omar G. Mahdy**

*University of Falluja,
**University of Anbar College of Education for Pure Science

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1, 5-Disubstituted 2-hydroxypyrroles.

ABSTRACT

Schiff's Bases were synthesized from the reaction of furfuraldehyde and substituted benzaldehyde with 4-aminoantipyrine and 2,4-dinitrophenylhydrazine and reacted with 2-(triphenylphosphoranylidene) succinic anhydride in an anhydrous 1,4- dioxane at reflux conditions. The resulting products were found to be 1,5-disubstituted 2-hydroxypyrroles. The structures of the products were confirmed by their melting points, FT-IR, ^{1}HNMR spectra and C.H.N. elemental analyses.

Introduction

Pyrrole is a five – membered heterocyclic aromatic ring with nitrogen as heteroatom located at position-1, which appears to be both an amine and conjugated diene, but it's chemistry is not consistent with either of these structural feature. The loan- pair electrons at nitrogen atom is in a 2p orbital and are a part of the aromatic sextet, by which pyrrole being similar to cyclopentadienyl anion. Pyrrole chemistry is therefore quite similar to that of activated benzene rings, and the loss of aromaticity by addition of proton to the loan- pair electrons is energetically unfavoured and severely limited, hence pyrrole is less electron-rich, less basic and less nucleophilic reagent than an aliphatic amine. It is relatively acidic because of the chang in hybridization from sp^3 to sp^2 and the delocalization of the negative charge in 1-N- 2,4-cyclopentadienyl anion.

* Corresponding author at: University of Anbar College of Education for Pure Science Anbar, Iraq.
E-mail address:

Pyrroles are prone to both electrophilic addition and substation reactions and resistant to both nucleophilic addition and substation reactions. Electrophilic substation specifically occurs at C2 rather than C3 due to the greater electron- releasing ability of neutral trivalent nitrogen and the high stability of the resulting intermediate carbocation. Whereas electrophilic addition occurs at nitrogen and carbon, the reversible proton addition for example, occurs by for the fastest at nitrogen and about twice as fast at C2 as at C3 in the heterocyclic ring. In addition pyrroles react readily with oxidizing, reducing, radicals, dienophilic, carbenes reagents and to some extent with Nucleophilic reagent via proton transfer.(1-7)

Pyrrole was first isolated from coal tar in 1834 and then in 1857 from bone oil. It is the backbone of the structure of many life- important natural products such as porphyrins (tetracyclopyrroles), bilirubins and biliveridines (liner tetrapyrroles), porphobilogen, octaethylporphyrine, metacycloprodigiosin, tetraazapophyrins, phthalocyanin, porphyrinaids and chlorophylls.(8) Polymers and copolymers of pyrrole are used as organic sensitizers in voltaic cells and pigments. (9)

Pyrroles are widely known as bio-active compounds which possess a variety of bio-activities.[10-12] Of these compounds are, anti-flammatory and analgesic, Zomepirac, antibiotic Pyrrolnitrin [8], cholesterol reducer, Atorvastatin (Lipitor).[1], anti –HIV-1, Lamivudine, Zalcitabine, Didanosine ,anti-herpes simplex virus (HSV), Acyclovir, anti-varicella zooster virus (VZV), Ganciclovir.[13]

Synthesis of pyrroles or N- substituted pyrroles was achieved by several methods and different starting materials. Paal-Knorr synthesis is one of them in which 1,4-dicarbonyl compounds react with ammonia or a primary amines to give Pyrroles. Tetrasubstituted pyrroles are prepared by Hantzsch synthesis through the reaction of α-halocarbonyl compounds with β-keto esters or β-diketones and ammonia or primary amines. Cyclocondensation of α- amino ketones with β-keto esters or β-diketones gives 3-alkoxycarbonyl- or 3-acyl- substituted pyrroles via Knorr synthesis. Kenner synthesis is the method for synthesis of 3- substituted-pyrrole-2- carboxylic esters, by the reaction of N-tolylsulphonyl glycerin esters with vinyl ketones. Nitro alkenes undergo cyclocondensation with C-H acidic isocyanides according to Barton- Zard synthesis to give trisubstituted-pyrroles. According to vanLeusen synthesis a stabilized amino group of tolylmethyl isocyanide (TosMIC) undergoes Micheal addition to unsaturated ketones and/ or esters followed by subsequent closure onto the isocyanide carbon to give the pyrrole ring. An N-acyl- pyrroles carrying identical groups R at 3-and 4-position were synthesized by Piloty- Robison synthesis from the reaction of aldehyde's azines with an acid chloride. The 2,3-disubstituted pyrroles are synthesized by heating ketoxims and acetylene in the presence of NaOH in DMSO according to Trofimor synthesis. [14- 17]

2. Experimental

2.1. Instrumentation

Melting points were determined in open capillary tubes and are uncorrected. The FT-IR spectra were recorded, (4000- 600 cm^{-1}) range on an Infrared spectrophotometer Model Tensor 27 Bruker Co., Germany. The ^{1}HNMR spectra were recorded on a Bruker Ultershield *300MHz* NMR spectrometer, Co., Germany, using DMSO- d_6 as a solvent, and the chemical shifts are reported as δ values in part per million (ppm) relative to TMS δ=0, as internal standard. The C.H.N. elemental analyses were performed by Euro Ea Elemental Analyser.

2.2. General procedure for synthesis of Schiff's Bases 3(a- j).

An equimolar mixture of (0.004 mole) of the aromatic aldehydes (1) and the primary amine (2) in absolute ethanol (40 ml) with few drops of glacial acetic acid was refluxed for (60 -180 min.) with continuous stirring by magnetic bar. The reaction mixture was allowed to cool down in an ice bath, whereupon a crystalline solid product was separating out during cooling. The solid product was filtered off, washed with distilled water, dried and recrystallized from absolute ethanol. The structural formula, IR characteristic absorption, yield%, melting point, colors, and the reaction time are given in table (1).

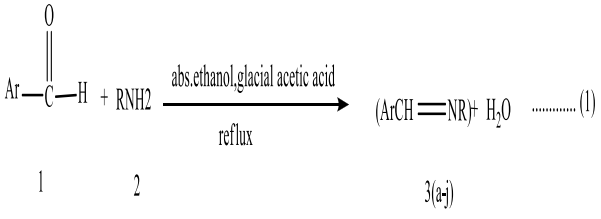
2.3. General procedure for synthesis of 1,5-Disubstituted 2-hydroxypyrroles 4(a-j).

In a well dried 100-ml round- bottom flask equipped with condenser and anhydrous calcium chloride guard tube, a mixture of equimolar amount (0.001mole) of Schiff's bases 3(a-j) and 2-(triphenylphosphoranylidene) succinic anhydride in anhydrous 1,4-dioxan (40 ml) was refluxed for (30- 90 min.). The reaction mixture was allowed to cool down in an ice bath, whereupon a crystalline solid product separated out during cooling. The product was filtered off, washed with distilled water, dried and recrystallized from 1,4- dioxan. The chemical formula, molecular weights, C.H.N %, yield %, melting points, colors, and the reaction time are given in table (2).

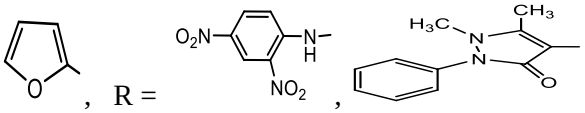
3. Results and Discussion

In this work, the reaction of 2-(triphenylphosphoranylidene) succinic anhydride as electrophilic reagent with imines (Schiff's bases) as mild nucleophilic reagents in reported.

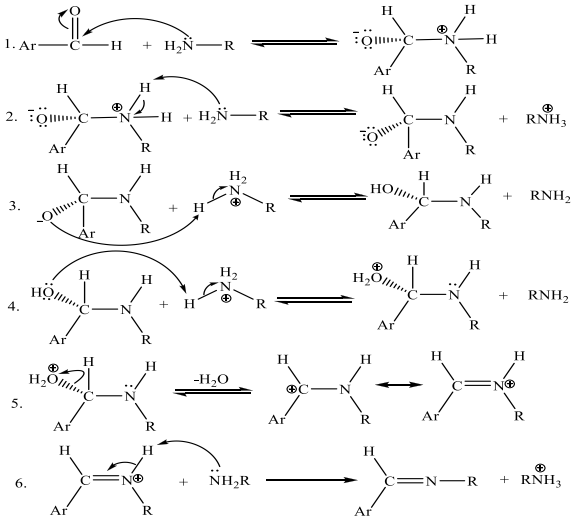
Schiff's bases were synthesized from furfuraldehyde and substituted benzaldehyde and 4-aminoantipyrine and phenylhydrazine via acid catalyzed thermal condensation reaction according to a well- known procedure. [18]



Where Ar =2-Br-C_6H_4-, 4-Br-C_6H_4-, 4-Cl-C_6H_4-, 2-NO_2-C_6H_4-, 4-$(CH_3)_2$N-C_6H_4-, 4-OH-C_6H_4- and



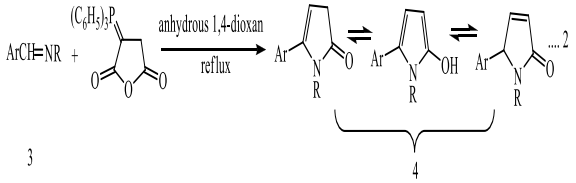
The mechanism of imine formation is thoroughly elucidated in the literatures.[2-7]



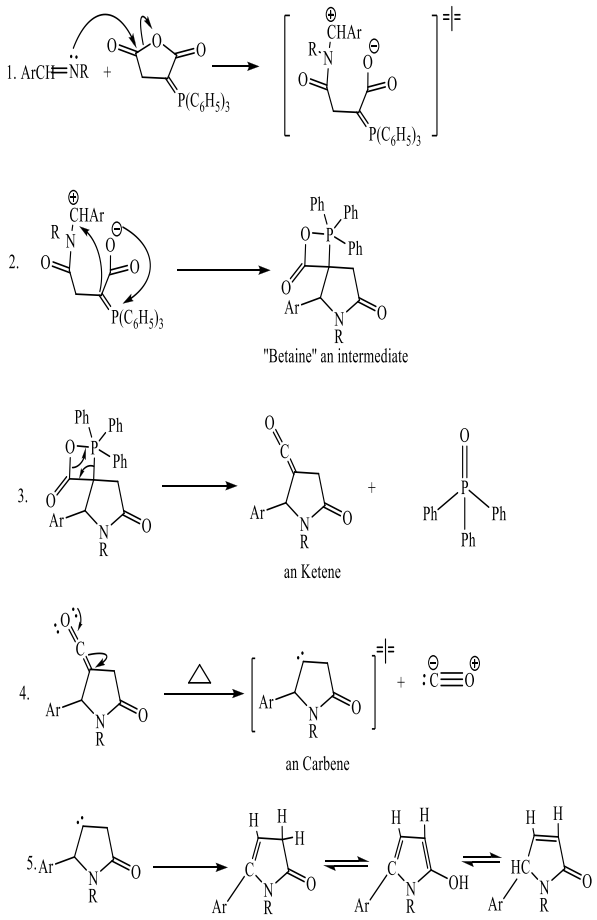
Scheme-1: Mechanism of formation of an imine from an aldehyde or a ketone

The structures of the synthesized Schiff's bases were confirmed by their melting points and the FT-IR spectra which showed the disappearance and appearance of the characteristic absorption frequencies (bands) of the principal functional groups. The FT-IR spectra showed the disappearance of the characteristic absorption frequencies of both (C=O) at (1720-1740) cm^{-1} and (-NH_2) at (3300-3500) cm^{-1} of the aldehyde and the primary amine respectively, and the appearance of the stretching absorption bands of azomethine group (C=N) at (1590-1623) cm^{-1}, in addition to the appearance of stretching absorption of the other groups in the structure of each individual compounds table (1).

Schiff's bases were reacted with 2-(triphenylphosphoranylidene) succinic anhydride in an anhydrous 1,4- dioxane under reflux conditions.



Of particular importance is to understand how this transformation occurs, therefor a plausible mechanism can be suggested as follows:



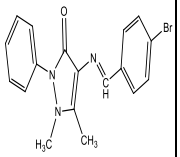
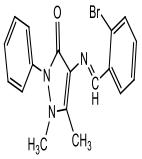
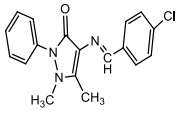
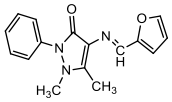
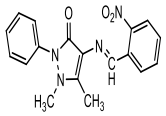
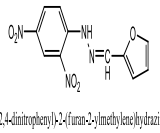
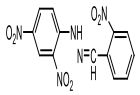
Scheme-2: Suggested mechanism for the formation of 1,5-Disubstituted 2-hydroxypyrroles

In the first step the nucleophilic azomethine group attack the electrophilic carbon atom of the carbonyl group to give a very reactive dipolar intermediate which in turn undergo internal rearrangement reaction with phosphoranylidene to an intermediate "betaine" while in the second step the betaine callapsis to give an intermediate "ketene" which in turn regect carbon monoxide to give an intermediate "carbene" in the third step. In the fourth step, carbine undergoes insertion reaction to give the tautomeric target molecule. The resulting 1,5-disubstituted 2-hydroxypyrroles are existing as tautomers of the two isomeric structure, 4- pyrrolin -2-one and 3- pyrroline -2- one. This assumption is fairly consistent with the resulting product from pyrrole oxidation by hydrogen peroxide. [7]

The structures of the synthesized 1,5-disubstituted 2-hydroxypyrroles were confirmed by their melting points and both FT-IR and ^{1}HNMR spectra and the C.H.N.% of the products, table 2 and table 3. The FT-IR spectra showed the disappearance of the characteristic absorption frequencies (bands) of both (C=O) group at (1760- 1810) cm^{-1} of 2-(triphenylphosphoranylidene) succinic anhydride and

that of azomethine group (C=N) at (1590-1623) cm^{-1}. And the appearance of the characteristic absorption frequencies of both ($C=O_{Keto}$) at (1590-1615) cm^{-1} and (O-H $_{enolic}$) at (2900-3200) cm^{-1} in addition to the appearance of stretching absorption of the other groups in the structure of each individual compounds. The resulting signals at the chemical shifts in the ^{1}HNMR spectra of each individual molecular structure of the product are in fair consistency with the expected signals of each proton in the different environment. Additional evidence was obtained from C.H.N. %, since the founded percentage of these elements is in high agreement with the calculated figures. The obtained analytical data are in high consistency with the letterature.[19-21]

Table (1): The structural formula, IR characteristic absorption, yield, melting point, colors, and the reaction time of Schiff's Bases 3(a- j).

Code	3a	3b	3c	3d	3e	3f	3g
Molecular structure and IUPAC Name	 (*E*)-4-(4-bromobenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	 (*E*)-4-(2-bromobenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	 (*E*)-4-(4-chlorobenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	 (*E*)-4-(furan-2-ylmethyleneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	 (*E*)-1,5-dimethyl-4-(2-nitrobenzylideneamino)-2-phenyl-1*H*-pyrazol-3(2*H*)-one	 1-(2,4-dinitrophenyl)-2-(furan-2-ylmethylene)hydrazine	 1-(2,4-dinitrophenyl)-2-(2-nitrobenzylidene)hydrazine
IR Characteristic Absorption frequencies, cm-1	3133 ν C-Harom., 3010 ν C-H Olef., 2963 ν CH3 Alk., 1700 ν C=O Amide3°, 1590 ν C=N Imine, 1589,1483 ν C=Carom.,olef., 1435 δ C-H Alk.	3139 ν C-Harom., 3018 ν C-H Olef., 2963 ν CH3 Alk., 1700 ν C=O Amide3°, 1603ν C=N Imine, 1590,1512 ν C=C arom.,olef., 1435 δ C-H Alk.	3096 ν C-H arom., 3030 ν C-H Olef., 2970 ν CH3 Alk., 1646 ν C=O Amide3°, 1600 ν C=N Imine 1582ν C=C arom., 1465 δ C-HAlk., 766 ν P-C-Cl	3070 ν C-Harom., 3045 ν C-H Olef., 2958 ν CH3 Alk., 1644 ν C=O Amide3°, 1610 νC=N Imine, 1590νC=Carom.,olef., 1480δ C-HAlk., 1301νC-O ether.	3073 ν C-Harom., 3024 ν C-HOlef. 2895 ν C-H Alk., 1646 ν C=O Amide3°, 1601 ν C=N Imine, 1567,1485 ν C=C arom.,olef., 1518,1355 ν C-NO2	3276 ν N-H, 3151 ν C-Harom., 3119 ν C-H Olef, 1620 ν C=N Imine, 1578,1418 ν C=C arom.,olef., 1500,1396 ν C-NO2, 962νC-O ether.	3287 ν N-H, 3093 ν C-Harom., 3087 ν C-HOlef., 1623νC=N Imine, 1584,1499 ν C=Carom.,olef., 1550,1331 ν C-NO2
Yield%	90	87	82	82	86	90	95
M.P	220-222	157-160	238-240	190-193	198-200	180-182	229-232
Color	yellow	yellow	Yellow	Brown	Orange	Red	Orange
Reaction Time.	120min.	120min.	90min.	150 min.	90 min.	180 min.	60 min.

Code	3h	3i	3j
Chemical Formula			
M.wt g\mole			
C.H.N Cal. (Found) C%			
H%			
N%			
Yield%	93	77	82
M.P	211-213	249-252	265-267
Color	Black	Orange	Dark Red
Reaction time	120 min.	90 min.	120 min.

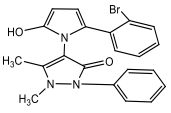
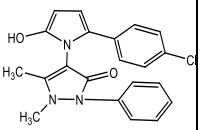
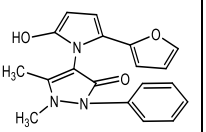
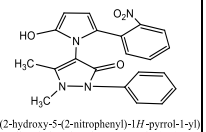
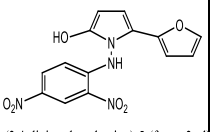
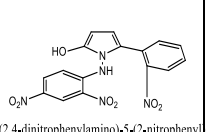
Table (2): The chemical formula, molecular weights, C.H.N %, yield, melting points, colors, and the reaction time of 1,5-Disubstituted 2-hydroxypyrroles 4(a-j)

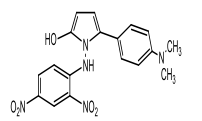
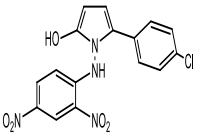
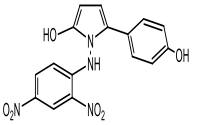
Code	4a	4b	4c
Chemical Formula	$C_{21}H_{18}BrN_3O_2$	$C_{21}H_{18}BrN_3O_2$	$C_{21}H_{18}ClN_3O_2$
M.wt g\mole	424.29	424.29	379.84
C.H.N Cal. (Found) C%	59.45 (59.32)	59.45 (59.11)	66.40 (66.09)
H%	4.28 (4.13)	4.28 (4.09)	4.78 (4.51)
N%	9.90 (9.78)	9.90 (9.62)	11.06 (10.79)
Yield%	75	69	79
M.P	187-190	171-173	220-222
Color	Brown	Brown	Pink
Reaction time	60 min.	60 min.	60 min.

Code	4d	4e	4f	4g	4h	4i	4j
Chemical Formula	$C_{19}H_{17}N_3O_3$	$C_{21}H_{18}N_4O_4$	$C_{14}H_{10}N_4O_6$	$C_{16}H_{11}N_5O_7$	$C_{18}H_{17}N_5O_5$	$C_{16}H_{11}ClN_4O_5$	$C_{16}H_{12}N_4O_6$
M.wt g\mole	335.36	390.39	330.25	385.29	383.36	374.74	356.29
C.H.N Cal. (Found) C%	68.05 (67.88)	64.61 (64.08)	50.92 (50.41)	49.88 (49.67)	56.39 (55.97)	51.28 (50.76)	53.94 (53.57)
H%	5.11 (5.02)	4.65 (4.43)	3.05 (2.67)	2.88 (2.79)	4.47 (4.27)	2.96 (2.61)	3.39 (3.11)
N%	12.53 (12.13)	14.35 (13.83)	16.96 (16.38)	18.18 (17.91)	18.27 (17.84)	14.95 (14.59)	15.73 (15.32)
Yield%	72	84	78	68	87	90	70
M.P	179-181	178-180	204-207	198-200	202-205	235-238	252-255
Color	Brown	Orange	Red	Yellow	Gray	Orange	Red
Reaction time	90min.	60 min.	75 min.	30 min.	30 min.	30 min.	30 min.

Table 3: Molecular structure, IR Characteristic Absorption, Chemical Shift δ ppm of 1,5-Disubstituted 2-hydroxypyrroles 4(a-j).

Code	Molecular structure	IR Characteristic Absorption frequencies, cm^{-1}	Chemical Shift δ ppm
4a	4-(2-(4-bromophenyl)-5-hydroxy-1*H*-pyrrol-1-yl)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	3133 υ C-H arom., 3010 υ O-H enolic, 2963 υ CH3 Alk., 1700 υ C=O Amide3', 1594 υ C=O Keto, ,1483 υ C=Carom, 1435 δ C-H Alk., 1329 δ O-H enolic,	(3H s) δ=2.4 (C-CH_3), (3H s) δ=3.19 (N-CH_3), (11H m)δ=7.2-7.8(C-$H_{arom.}$), (1H s) δ=9.5 (O-H).

4b	 4-(2-(2-bromophenyl)-5-hydroxy-1*H*-pyrrol-1-yl)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	**3133 υ C-Harom., 3010 υ O-Henolic , 2963 υ CH3 Alk., 1700 υ C=O Amide3', 1589 υ C=O Keto , 1483υ C=C arom., 1435 δ C-H Alk., 1320 δ O-H enolic.**	----------
4c	 4-(2-(4-chlorophenyl)-5-hydroxy-1*H*-pyrrol-1-yl)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	**3079 υ C-H arom., 3056 υ O-H enolic, 2942 υCH3 Alk.,1643 υ C=O Amide3', 1591 υ C=O Keto., 1563 υ C=C Het., 1483 δ C-H Alk.,1376 δ O-H enolic., 767 υ P-C-Cl**	**(3H s) δ=2.4 (C-CH_3), (3H s) δ=3.19 (N-CH_3), (11H m)δ=7.2-7.8 (C-$H_{arom.}$), (1H s) δ=9.5 (O-H).**
4d	 4-(2-(furan-2-yl)-5-hydroxy-1*H*-pyrrol-1-yl)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	**3075 υ C-H arom., 3046 υ O-H enolic, 2959 υ CH3 Alk., 1639 υ C=O Amide3', 1590 υ C=O Keto , 1545 υ C=C Het., 1481 δ C-H Alk.,1413 δ O-H enolic 1301 υ C-O ether.,**	----------
4e	 4-(2-hydroxy-5-(2-nitrophenyl)-1*H*-pyrrol-1-yl)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	**3074 υ C-H arom., 3027 υ O-H enolic 2864υ CHAlk., 1645υ C=O Amide3', 1597υ C=O Keto , 1566 υ C=C Het., 1518, 1355 υ C-NO2, 1413 δ O-H enolic**	**(3H s) δ=2.4 (C-CH_3), (3H s) δ=3.2 (N-CH_3), (11H m)δ=7.2-8.2 (C-$H_{arom.}$), (1H s) δ=9.8 (O-H).**
4f	 1-(2,4-dinitrophenylamino)-5-(furan-2-yl)-1*H*-pyrrol-2-ol	**3276 υ N-H, 3151 υ O-H enolic 3119υ C-H arom., 1610 υ C=O Keto ., 1580 υ C=C arom., 1518 υ C=C Het., 1500, 1396, υ C-NO2., 1320 δ O-H enolic., 1132 υ C-O phenol,962 υ C-O**	**(1H s) δ=3.57 (N-H), (8H m) δ=6.6-8.8 (C-$H_{arom.}$), (1H s) δ=11.68 (O-H).**
4g	 1-(2,4-dinitrophenylamino)-5-(2-nitrophenyl)-1*H*-pyrrol-2-ol	**3287 υ N-H, 3115 υ O-H enolic 3091 υ C-H arom., 1591 υ C=O Keto ., 1499 υ C=C arom., 1539,1332 υ C-NO2, 1522υ C=C Het., 1320 δ O-H enolic.**	----------

4h	 5-(4-(dimethylamino)phenyl)-1-(2,4-dinitrophenylamino)-1*H*-pyrrol-2-ol	**3276 υ N-H, 3097 υ O-H enolic 3095 υ C-H arom., 2993 υ CH3 Alk., 2873 υ CH Alk., 1597 υ C=O Keto. 1510 υ C=C arom., 1497 υ C=C Het., 1448, 1385υ C-NO2., 1323δ O-H enolic**	**(6H s) δ=3.0 (-CH_3), (1H s) δ=3.57 (N-H), (9H m) δ=6.7-8.8 (C-$H_{arom.}$), (1H s) δ=11.54 (O-H).**
4i	 5-(4-chlorophenyl)-1-(2,4-dinitrophenylamino)-1*H*-pyrrol-2-ol	**3285 υ N-H, 3101 υ O-H enolic 3092 υ C-H arom., 1612 υ C=O Keto . 1583 υ C=C arom., 1507 υ C=C Het., 1421 υ C-NO2 ,1313δ O-H enolic, 613 υ P-C-Cl**	**(1H s) δ=3.57 (N-H), (9H m) δ=6.7-8.8 (C-$H_{arom.}$), (1H s) δ=11.54 (O-H).**
4j	 1-(2,4-dinitrophenylamino)-5-(4-hydroxyphenyl)-1*H*-pyrrol-2-ol	**3266 υ N-H , 3250 υ O-H Phenol, 3117 υ O-H enolic ,2975 υ C-H arom., 1619 υ C=O Keto . 1588 υ C=C arom., 1508 υ C=C Het.,1416 δ O-H enolic., 1330 υ C-NO2**	----------

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تكوين مشتقات 5،1 – ثنائية التعويض 2- هايدروكسي بيرول من تفاعل 2- ثلاثي فنيل فوسفوريلدين سكسنيك انهدريد مع قواعد شف

عبيد حسن عبد عمر جمال مهدي

الخلاصة

حضرت قواعد شف من تفاعل البنزالديهايدات والفورفالالديهايد مع 4- امينو انتي بايرين و 4،2- ثنائي نايترو فنيل هيدرازين وفوعلت مع 2- ثلاثي فنيل فوسفوريلدين سكسنيك انهدريد بالتصعيد في 4،1- ثنائي الدايوكسان الجاف. كانت النواتج مشتقات 5،1 – ثنائية التعويض 2- هايدروكسي بيرول والتي حددت صيغها التركيبية من خلال نقاط الانصهار وأطياف الاشعة تحت الحمراء وأطياف الرنين النووي المغناطيسي للبروتون.