Synthesis of Schiff Bases of Homocyclic and Heterocyclic Phthalimides

[1] Mohammed A. Sheat, [2] Anwar A. G. Fathi and [2] Eslam K. Saeed
[1] *Chemistry Department, College of Science, University of Mosul, Mosul, Iraq*
[2] *Chemistry Department, College of Education, University of Mosul, Mosul, Iraq*

Abstract
Schiff bases for homocyclic and heterocyclic phthalimides were prepared from the condensation reactions of phthalic anhydride and pyridine-2,3-dicarboxylic anhydride with *p*-phenylene diamine. The products were allowed to react through condensation reactions with aldehydes and ketones to yield these compounds. The structural formula of the synthesized compounds were conforemed by physical and spectroscopic methods.

Introduction
Phthalimides are of high interest organic compounds used in organic synthesis and other industerial fields such as in drugs synthesis for the acetylenic phthalimides showed pharmaceutical activity to be anticholinergic agents and anti-Parkinsonian agent [1,2], other substituted phthalimides were found to possess satisfactory analgesic characteor and used as analgesic drug [3], also some of them have been employed as inhibitors against mammalian, plant, bacterial and fungal copper-containing amine oxidases [4]. Whereas other substituted phthalimides demonstrated inhibitory effect on the tested microorganisms [5,6], moreover they also employed as prevulcanization inhibitor used in sulphur-cured rubber polymer systems [7].
On the other hand, Schiff bases were also found to possess biological activity and used as analgesic and antipyretic agents [8], bactericidic, fungicidic and algicidic agents [9] and anti-inflammatory agents [10], whereas the Schiff bases complexes with some transitional elements have been employed in the synthesis of stabilizers to increase the wheather resistant of polymers [11] and as excellent catalysts in the dehydrogenation process of hydrazones into diazocompounds [12], also in the oxidation process of sulfides into sulfoxides [13].

Experimental
Melting points were measured by Electrothermal 1A9000 Digital-Series Melting point (1998). Apparatus and are uncorrected. I.R. spectra were recorded by FTIR-Tensor 27-Bruker Spectrophotometer as KBr disc. U.V. spectra were obtained from Shimadzu U.V-V is spectrophotometer in $CHCl_3$.

Methods of preparation
1. Preparation of p-Phthalimido Aniline (1) [14]:
A mixture of *p*-phenylene diamine (0.1 mole) and phthalic anhydride (0.1 mole) in acetic acid (50 ml) was refluxed for 1 hr, then water was added till solid precipitate was obtained. Filtration and recrystallisation of the precipitate from acetic acid-water yield the desired product as a light brown powder of 54% yield and m.p. (292-294)°C.

2. Preparation of p-(Pyridine-2,3-Dicarboximido) Aniline (2) [14]:
A mixture of *p*-phenylene diamine (0.02 mole) and pyridine-2,3-dicarboxylic anhydride (0.02 mole) was refluxed in acetic acid (50 ml) for 1hr. The reaction mixture was filtered while hot and evaporated to dryness. The residue was recrystallized from acetic acid to give the desired product as a brown crystals of 43% yield and m.p. (320-322)°C.

Preparation of Schiff Bases Compounds (3_{a-f}) [15]:
Equimolar (0.003 mole) of *p*-phthalimido aniline (1) and aldehyde or ketone, each one dissolved in absolute ethanol (10 ml) with heating to give hemogenous solutions. The solution of aldehyde or ketone was added gradually to the solution of compound (1). The mixture was refluxed for 1hr with stirring. After cooling, the precipitate was filtered and recrystallized from ethanol to obtain the desired products (3_{a-f}) as a fine crystals as shown in Table (1).
On following the same procedure, *p*-(pyridine-2,3-dicarboximido) aniline was allowed to react with aldehydes or ketones to produce Schiff bases compounds (4_{a-e}).

Table (1): Physical and spectroscopic data of compounds (1-4)

Comp. No.	Yield %	m.p. °C	I.R. (KBr) cm^{-1}			U.V. ($CHCl_3$) λ_{max} (nm)
			$\upsilon^*_{C=O}$	$\upsilon_{C=O}$	$\upsilon_{C=N}$	
			Imidic			
1	54	292-294	1715	1634	-	293
2	43	320-322	1711	1628	-	287
3_a	61	369-371	1698	1615	1595	306
3_b	70	300-303	1692	1624	1592	311
3_c	52	342 dec.	1685	1617	1596	307
3_d	68	320 dec.	1685	1619	1590	316
3_e	56	370-373	1680	1621	1586	311
3_f	48	300 dec.	1695	1623	1595	309
4_a	40	341-344	1694	1620	1593	302
4_b	45	315-317	1696	1618	1587	310
4_c	53	343-345	1692	1615	1594	305
4_d	58	297-300	1695	1621	1591	314
4_e	49	307 dec.	1685	1617	1595	304

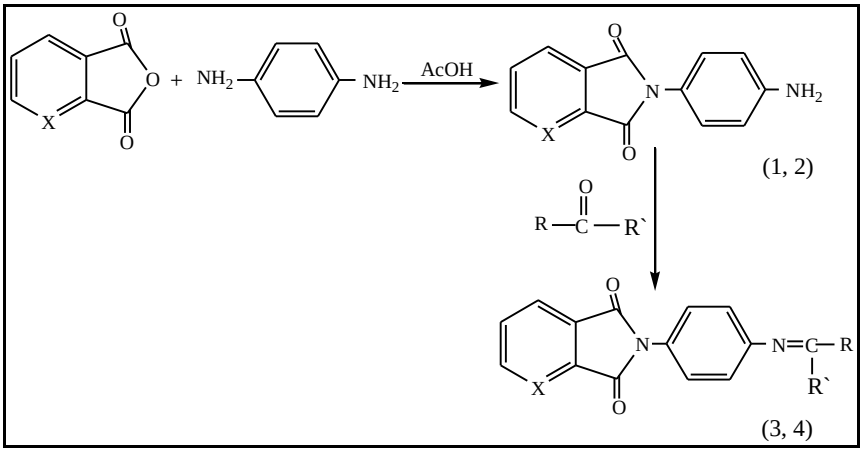
Note: Compound (1 and 2) showed two broad bands at 3310-3345cm^{-1} for υ_{N-H}.

Result and Discussion

The biological activity which was found in a number of previous prepared phthalimides and Schiff bases compounds create a desire to prepare new Schiff bases for homocyclic and heterocyclic phthalimides. The idea of synthesis of Schiff bases containing phthalimido groups may leads to obtain compounds with some biological activity.

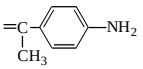
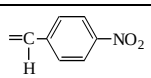
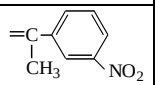
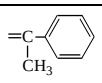
Therefore, a number of Sciff bases for homocyclic and heterocyclic phthalimides (3_{a-f}, 4_{a-e}) were prepared from two steps. The first involved reaction of *p*-phenylene diamine with phthalic anhydride or pyridine -2,3-dicarboxylic anhydride to give compounds (1, 2), while the second step involved condensation reactions of compounds (1, 2) with aldehydes and ketones to obtain the new Schiff bases compounds (3_{a-f}, 4_{a-e}) as shown in Scheme (1).

The formation of Schiff bases was occured through addition-elemination mechanism [16] as illustrated in Scheme (2).

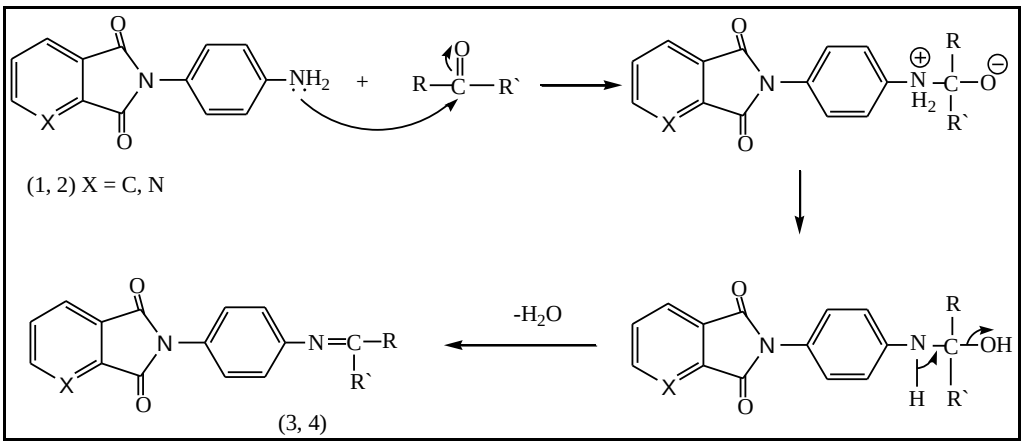


1,3; X=C

2,4; X=N

Comp. No.	$=C(H)R`$	Comp. No.	$=C(CH_3)R`$
3_a, 4_a		3_d, 4_d	
3_b, 4_b		3_e, 4_e	
3_c, 4_c		3_f	

Scheme (1)



Scheme (2)

The synthesized Schiff bases (3_{a-f}, 4_{a-e}) have been investigated according to their physical and spectroscopic data (I.R. and U.V.), and these investigations were supported by the positive Hinsberg test for the amino group in compounds (1,2) and negative test in the synthesized Schiff bases compounds except compounds (3_d, 4_d) [17].

The I.R. spectra of the synthesized Schiff bases (3_{a-f}, 4_{a-e}) showed two absorption bands (strong and weak) at lower frequencies, one at (1680-1698) cm^{-1} and the other at (1615-1624) cm^{-1} due to the asymmetric and symmetric stretching vibrations of the imidic carbonyl groups [18]. Another absorption band was appeared at (1600-1590) cm^{-1} for the $\upsilon_{C=N}$ (imine group) in these compounds.

The U.V. spectra of the compounds (3_{a-f}, 4_{a-e}) showed absorption bands at longer wavelengths (λ_{max} 302-316nm) compared with that in compounds (1 and 2). This bathochromic shift is due to the increasing of conjugation effect on the ($n\rightarrow\pi^*$) transition which occured in these compounds [19].

Table (1): Physical and spectroscopic data of compounds (1-4)

Comp. No.	Yield %	m.p. °C	I.R. (KBr) cm^{-1}			U.V. ($CHCl_3$) λ_{max} (nm)
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Note: Compound (1 and 2) showed two broad bands at 3310-3345cm^{-1} for υ_{N-H}.

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تحضير قواعد شيف للفثاليميدات المتجانسة وغير المتجانسة

[١] محمد علي شيت و [٢]أنوار عبد الغني فتحي و [٢] إسلام كمال سعيد

[١] قسم الكيمياء – كلية العلوم ، جامعة الموصل ، الموصل ، العراق

[٢] قسم الكيمياء – كلية التربية ، جامعة الموصل ، الموصل ، العراق

الخلاصة

حضرت قواعد شيف للفثاليميدات المتجانسة وغير المتجانسة من خلال تفاعلات التكثيف لانهيدريد الفثاليك وانهيدريد البريدين– ٢،٣– ثنائي الكاربوكسليك مع البارا– فنلين ثنائي الأمين، واستخدمت النواتج في تفاعلات التكثيف مع الالدهايدات والكيتونات للحصول على هذه المركبات، وأثبتت الصيغة التركيبية للمركبات المحضرة باستخدام الطرائق الفيزياوية والطيفية.