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## The Preparation and Characterization of Some Schiff Bases by Direct Fusion

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### Abstract

Schiff bases were prepared via direct fusion (solvent free) of vanillin with *P*- Toluidine , 2, amino-4, 6-dimethylpyridine. Indole-3-carboxaldehyde with *o*-aminophenol , *P*- Toluidine and 2, amino-4, 6-dimethylpyridine.

The synthesized compounds has been characterized on the bases of IR and <sup>1</sup>H NMR spectra. The study reveals that the method is simple and takes a short time in addition it is a simple method of purification in a moderate yield.

**Keywords:** Schiff bases, green chemistry, *o*- vanillin, indole-3-carboxaldehyde.

### 1. Introduction

Schiff bases are extensively studied due to their different application owing to their characteristic properties such as coordinating ability [1,2] with different methods, thermal stability[2], biological activities [3],and catalytic properties[4], etc.. The experimental conditions for Schiff bases formation depend on the nature of amine and carbonyl compounds. Usually aldehydes condensed with amine without

removing the water formed during the reaction[ 5]. So mostly such reactions are carried out in refluxed ethanol or methanol[6]. But with diary ketons or arylalkyl ketones with aromatic amine, needs to remove the water formed. The reactions of aromatic ketons with aromatic amines have higher temperature , longer time, and catalyst is also required.

### 2. Experimental Materials and Methods

*o*.vanillin, *P*. Toluidine 2, amino phenol, and 2-amino-4, 6 dimethy pyridine were purchased from Fluka and were used as received. Indol-3- carboxaldehyde from Himedia.

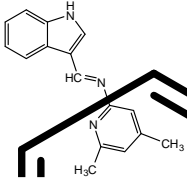
Melting points(uncorrected) were done using melting point apparatus. FTIR spectra were recorded on SHIMIDZU-84005 on a KBr pellets, <sup>1</sup>H NMR were recorded, DMSO-d<sub>6</sub> as asolvent on TMS as astandard Bruker-400 MHz.

## 2.1.Synthesis (General Procedure)

Equimolar of aldehydes (*o*.vanillin or indole carboxaldehyde) and amines ( *P*. Toluidine 2, amino phenol, and 2-amino-4, 6 dimethy pyridine) were ground together and heated gently in an oil bath for about 30 minutes.

The resulting mixture was dissolved in acetone and then cold distilled. water was added and left in refrigerator overnight. The solid product obtained was filtered off and dried. The physical properties and yield are collected in Table 1.

**Table 1:Some physical properties of synthesized Schiff bases**

No.	Structure	Physical State	m.p (°C)	Yield%
R <sub>1</sub>	(E)-2-methoxy-6-((p-tolylimino)methyl)phenol	Orange powder	98	25
R <sub>2</sub>	(E)-2-((4,6-dimethylpyridin-2-ylimino)methyl)-6-methoxyphenol	Yellow- light orange powder	104-105	60
R <sub>3</sub>	(E)-2-((1H-indol-3-yl)methyleneamino)phenol	Brown Crystal	193-194	16
R <sub>4</sub>	(E)-N-((1H-indol-3-yl)methylene)-4-methylaniline	White powder	156-157	24
R <sub>5</sub>	 (E)-N-((1H-indol-3-yl)methylene)-4,6-dimethylpyridin-2-amine	Brown Crystal	172-174	30

### 3. Results and Discussion

#### 3.1. Results:

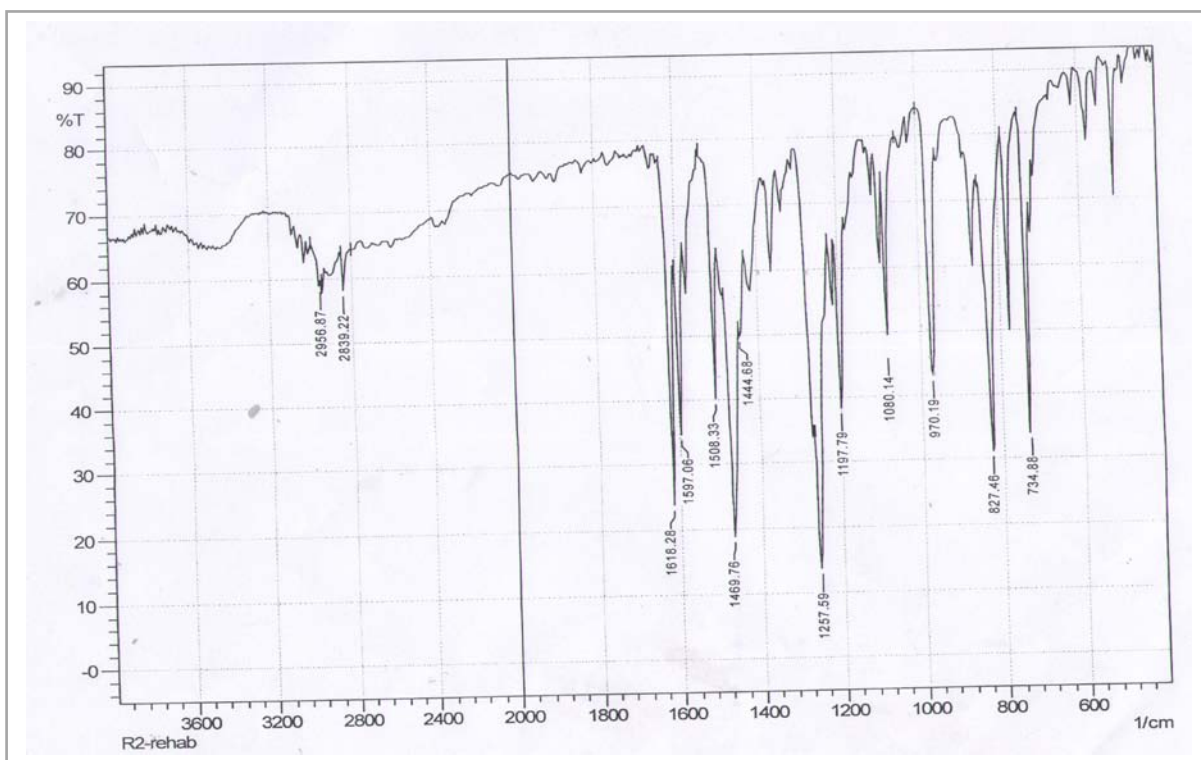
All the synthesized Schiff bases were stable to air at room temperature, they were all soluble in DMSO, DMF and THF. And they were insoluble in alcohol, acetone and chloroform.

#### 3.2. Infrared

The synthesized compounds showed a sharp band at 1604-1633  $\text{cm}^{-1}$  assigned to the azomethine group ( $-\text{C}=\text{N}$ ), thus clearly gave an evidence of condensation between an aldehydes and amines. Also the spectra of compound (Fig. 1) R1 and R2 showed a

All compounds have crystalline nature and melted sharply. All compounds have yellow to orange colours.

broad band resulting from hydroxyl group stretching at  $\sim 3400 \text{ cm}^{-1}$  also the compounds R3-R5 showed a sharp band at 3147-3167  $\text{cm}^{-1}$  (Fig. 2) assigned to N-H of indole moiety. IR data of other bands are recorded in Table 2.



**Figure 1: IR spectrum of Compound R1**

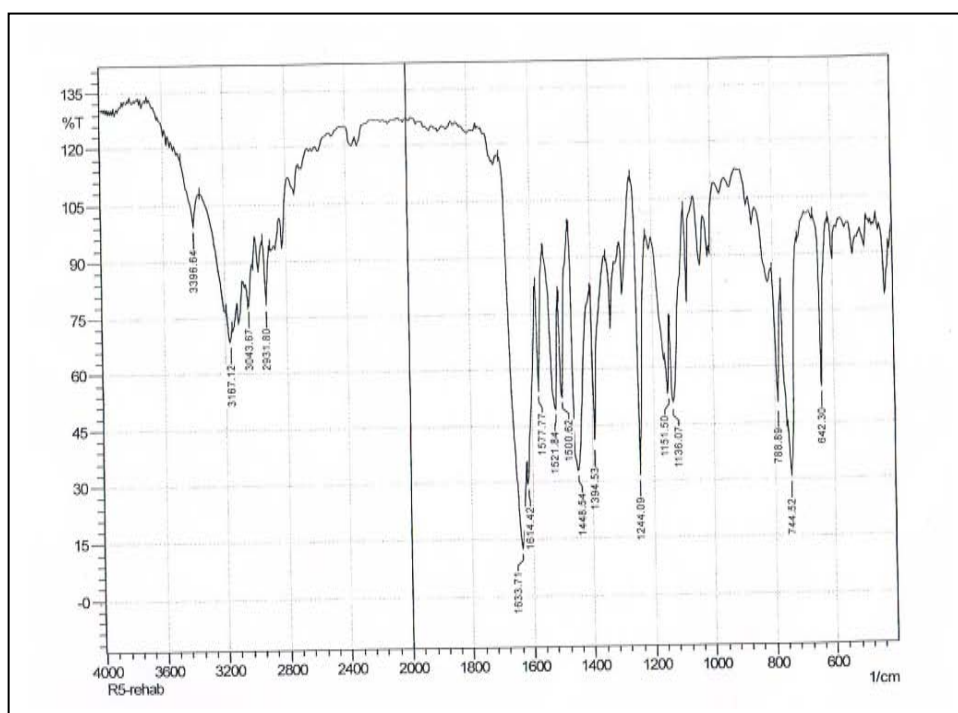


Figure 2: IR spectrum of Compound R5

Table 2: IR data on KBr,  $\nu$   $\text{cm}^{-1}$

Compound	$\nu$ OH	$\nu$ NH	$\nu$ CH aliphatic	$\nu$ CH aromatic	$\nu$ C=N aliphatic	$\nu$ C-N	$\nu$ C-O
R1	3300-3400 br	—	2956 asym 2839 sym	3025 m	1618.28	1257 vs	1197 s
R2	3450 br	—	2960 2830	3055 w	1604	1251 vs	1201 vs
R3	3199 br	3167 br	2931 2867	3043 m	1633.7	1392 vs	1244 vs
R4	—	3396 br	2931 2865	3043 m	1633	1394 vs	1244 vs
R5	—	3147 br	2952 2881	3065 m	1600	1244 vs	1122 vs

m, moderat ; w, weak ; br, broad ; s, strong ; vs, very strong

### $^1\text{H}$ NMR

The  $^1\text{H}$  NMR spectrum of R1 in DMSO-d<sub>6</sub> showed a signal at 13.39 ppm attributed to hydroxyl proton while a signal at 8.95 ppm assigned to azomethine proton [7]. The aromatic protons appear in the region 6.9-7.35 ppm as a multiple signal. The signal of OCH<sub>3</sub> protons appear at 3.82 ppm and the signal of CH<sub>3</sub> proton at 2.34 ppm (Fig. 3).  $^1\text{H}$  NMR spectrum of R2 showed a signal of OH proton at 13.34 ppm and the signal of azomethine proton at 9.44 ppm, the aromatic protons in the region 6.9-7.34

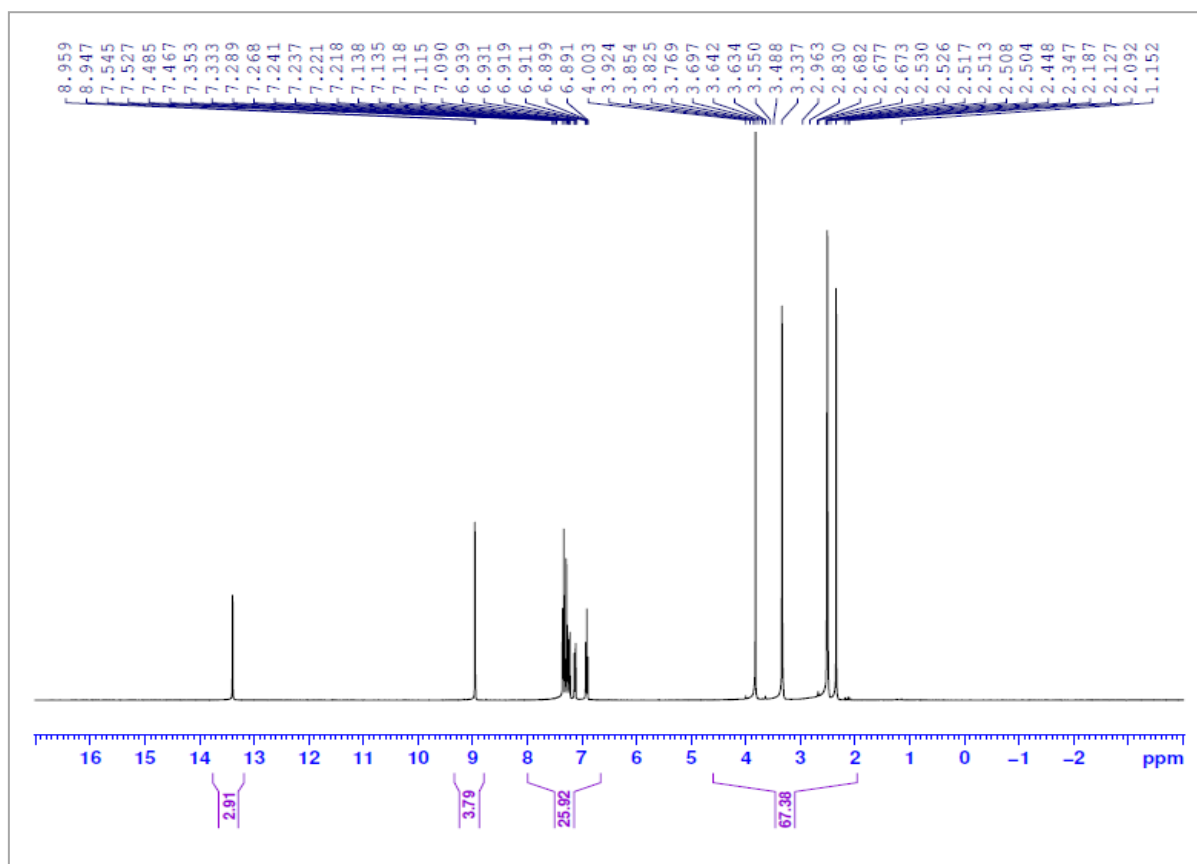
ppm, the signal of OCH<sub>3</sub> proton at 3.83 ppm and the signal of 2 CH<sub>3</sub> protons at 2.34 ppm (Fig. 4).

$^1\text{H}$  NMR spectrum of R<sub>3</sub> showed a signal of phenolic proton at 12.14 ppm and a signal at 9.93 assigned to N-H proton of indole moiety [8]. The azomethine proton signal appears at 8.29 and the aromatic protons at the region 7.2-8.29 ppm (Fig. 5).

$^1\text{H}$  NMR spectrum of R<sub>4</sub> showed a signal of indole N-H at 12.3 as a broad band, the azomethine proton at 9.9 ppm. The other

aromatic protons in expected region.  $^1\text{H}$  NMR spectrum of  $\text{R}_5$  showed a signal of indole N-H at 11.86, and the azomethine

proton at 9.31 ppm. The two methyl protons signals appear at 2.1, 2.3 ppm (Fig. 6). also the aromatic protons appear at 7.2-8.4 ppm.



**Figure 3:  $^1\text{H}$  NMR spectrum of Compound R1**

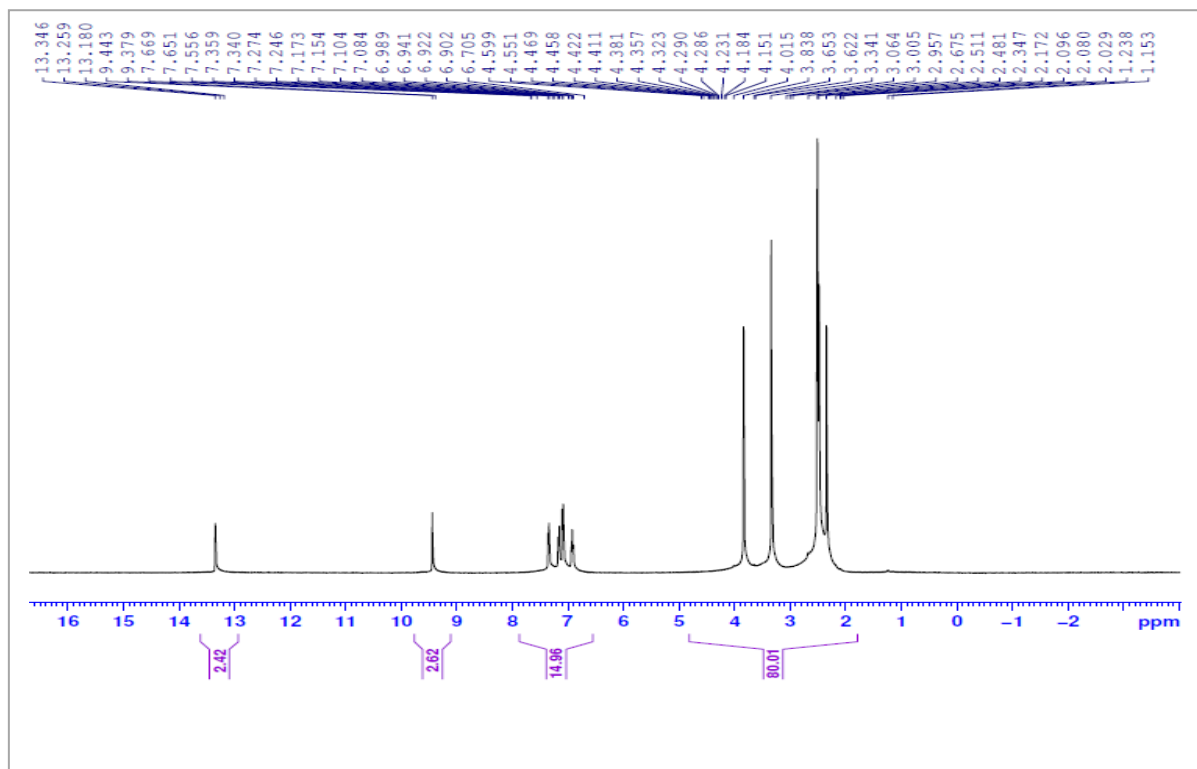


Figure 4: <sup>1</sup>H NMR spectrum of Compound R2

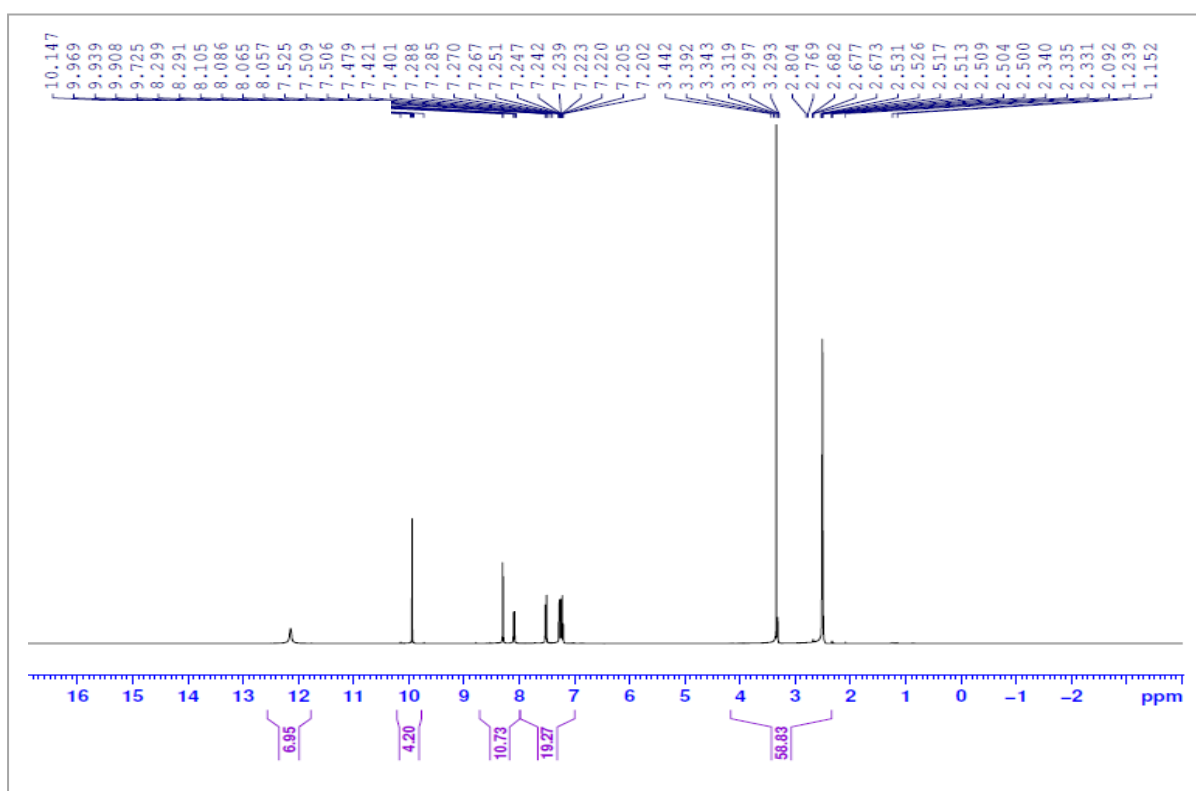


Figure 5: <sup>1</sup>H NMR spectrum of Compound R3

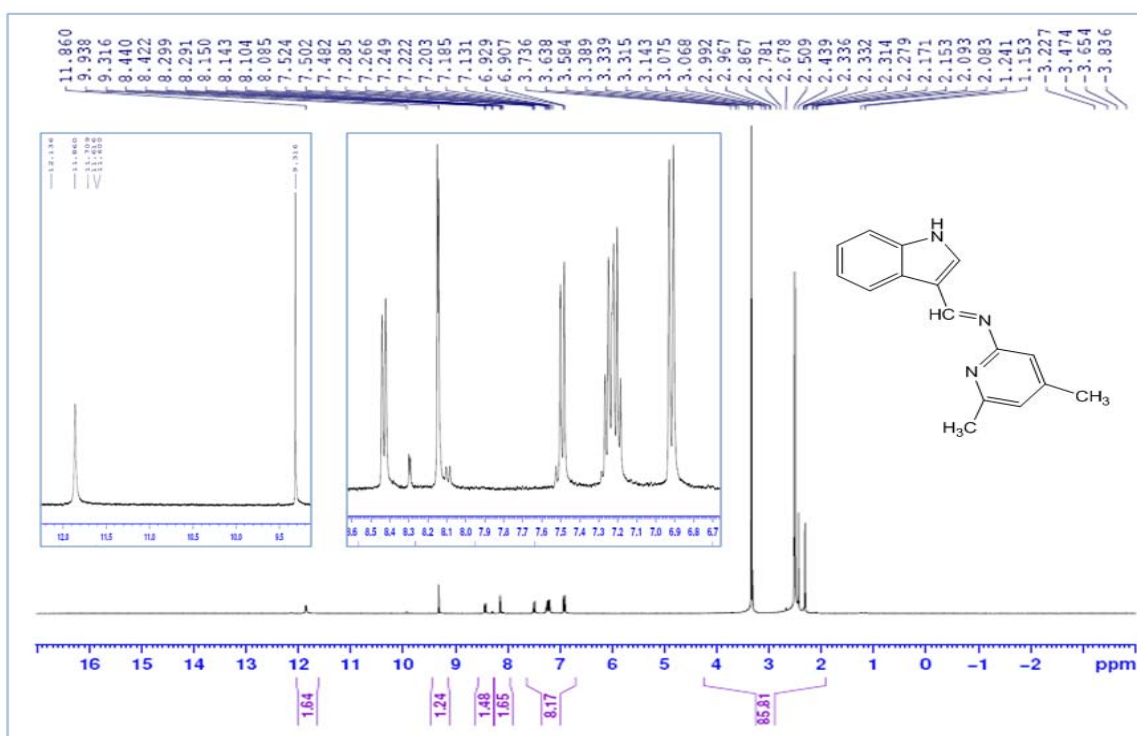


Figure 6 : <sup>1</sup>H NMR spectrum of Compound R5

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## تحضير وتشخيص بعض قواعد شف بطريقة الصهر المباشر

رحاب غني عبود

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العراق ، بصره

### الخلاصة

حضرت مركبات قواعد شف بطريقة الصهر (بدون مذيب) من تفاعل الفانيلين مع كل من بارا- تولودين و 2-امينو - 4,6 ثنائي مثيل بريدين . و تمت مفاعلة الاندول - 3- كاربوكسليدهايد مع اورثو - امينو فينول و بارا تولودين و 2-امينو - 6,4 ثنائي مثيل بريدين . وشخصت المركبات المحضرة بواسطة مطيافية الرنين النووي المغناطيسي للبروتون ومطيافية الأشعة تحت الحمراء وقد تم الاستنتاج ان طريقة التحضير سريعة ولم تحتاج الى مذيب او وقت طويل لاكمال التفاعل فضلاً عن أن عملية التنقية كانت أيضاً سهلة .