

## The Neighboring Benzil group and Synthesis of Hydantions

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### Abstract:-

Synthesis of some imidazolidine derivatives in the presence of the 4-chloro-4'-dimethyl amino benzyl, 2-nitro-4'-dimethylaminobenzyl, 4-chloro-4-hydroxybenzyl, 4-chloro-3'-bromobenzyl, 4-chloro-4-aminobenzyl with urea.

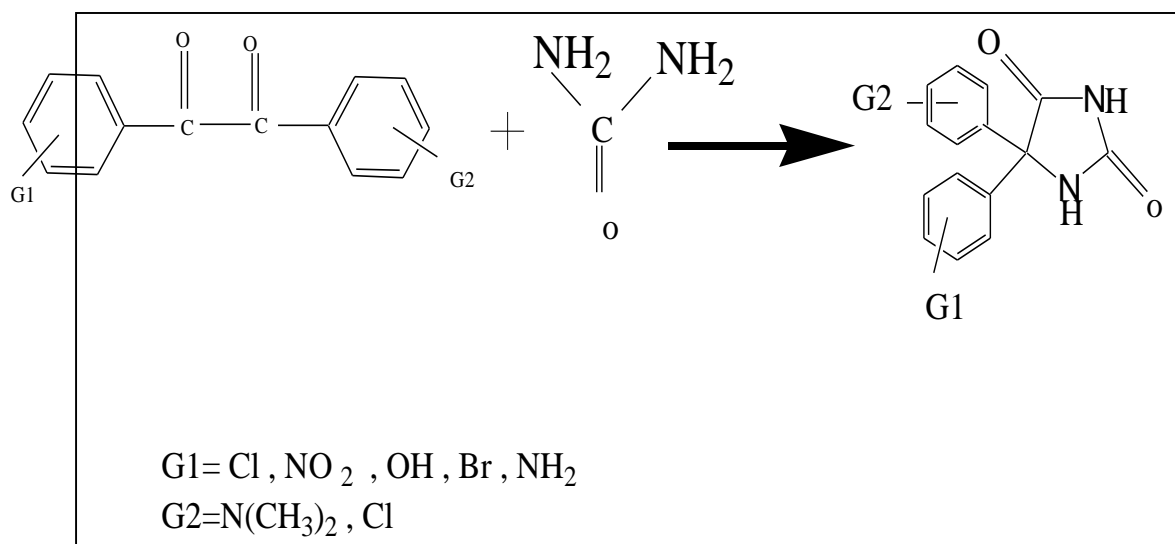
The compounds which have been prepared are 5-(4-dimethylaminophenyl)-5'-(4-chlorophenyl)-imidazolidine-2,4-dione, 5-(4-dimethylaminophenyl)-5'-(2-nitrophenyl)-imidazolidine-2,4-dione, 5-(4-chlorophenyl)-5'-(4-hydroxyphenyl)-imidazolidine-2,4-dione, 5-(4-chlorophenyl)-5'-(3-bromophenyl)-imidazolidine-2,4-dione, 5-(4-chlorophenyl)-5'-(4-aminophenyl)-imidazolidine-2,4-dione. Diagnosis of compound by IR spectrum and C.H.N analysis.

### 1-Introduction :-

The imidazolidine 2,4-dione antiepileptic drug phenyl and of structurally related derivatives. This heterocycle is present in a wide range of biologically active compounds anticonvulsant and antitumor<sup>(1)</sup>.

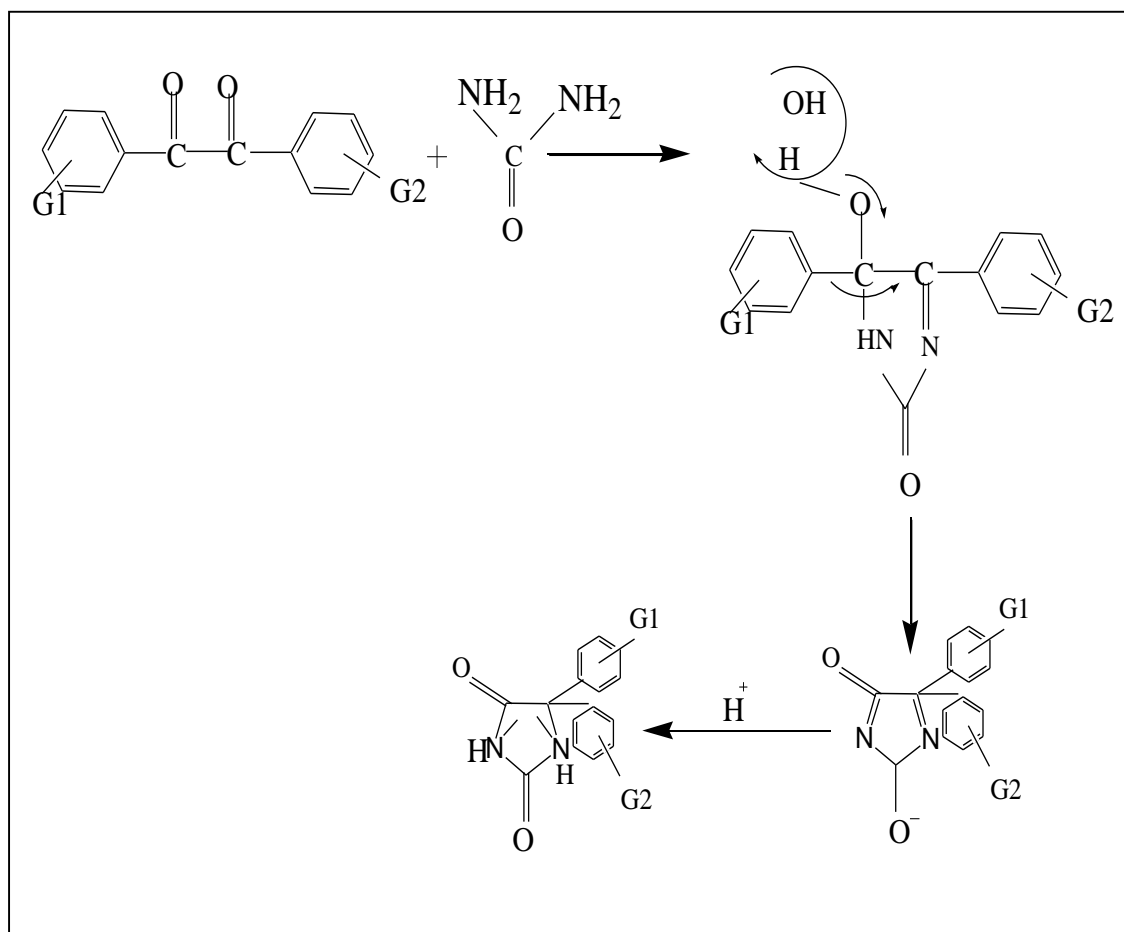
The heterocyclic straight forward condition for the synthesis of phenytoin in the base-catalyzed condensation using benzyl derivatives and urea.

Scheme(1) Known as the benzil synthesis of phenytoin<sup>(2)</sup>.



**Scheme (1) preparation of imidazolidine derivatives**

The reaction described to synthesise selectively and in high yields phenytoin. The great step consists reaction of benzyl derivatives with urea, and conversion of resulting imidazolidine derivatives  
Scheme (2) show the mechanical interaction of the former are<sup>(3)</sup>.



**Scheme (2) The mechanical interaction to preparation imidazolidine derivatives**

## 2-Experimental

### 2.1- Materials and Measurements

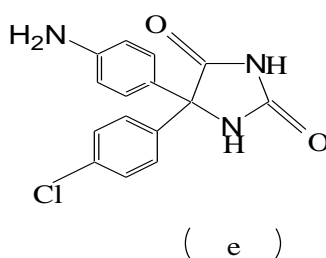
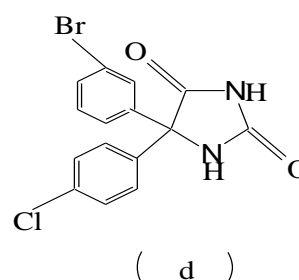
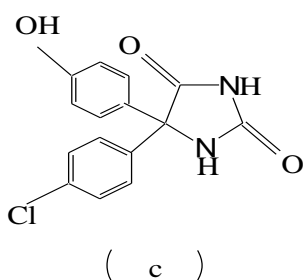
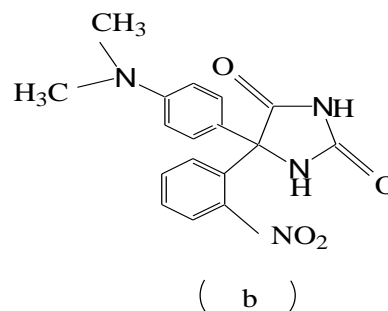
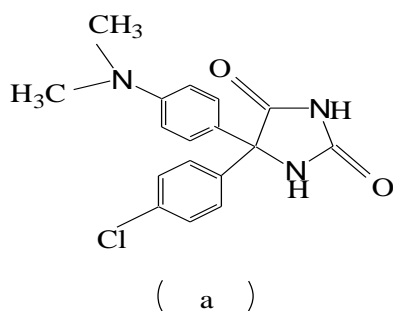
All chemicals were of highest purity and used as supplied from BDH, Aldrich and Fluk company. Elemental analysis were carried out by micro analytical unit 1108 C.H.N. elemental analyzer. Infrared spectra were measured with test scan Shimaduz FTIR-8000 series, in the (4000-400)  $\text{cm}^{-1}$  range using KBr discs and Melting point "P.D-303" Apel was used to measure the melting point their compounds.

### 2.2. General procedures synthesis of hydantoins

To a solution of 5.3gm (0.025mole)of benzil(3gm of urea 0.5mole) in 50ml of 15% NaOH ,and 50ml of 75% ethanol . the resulting mixture was refluxed for 2h and poured into cold water , the precipitate was filtrated and the filtrate was acidified wit acetic acid . the resulting precipitate was collected dried and recrystallized from hote ethanol <sup>(4,5)</sup>

### **3-Result and Discussion:-**

The Biltz synthesis is acommon way to synthesise phenytoin starting from benzil and urea two procedures , the classical one under thermal heating and a new microwave – assisted approach<sup>(6)</sup>study only classical method. The imidazolidine wre obtained:-



The FT-IR spectra of imidazolidine derivatives are shown in figures.(1),(2),(3) ,(4)and(5) .The most important IR assignments of ligand and their compounds (KBr disc)are listed in table (1).

All compounds(a)5-(4-dimethylaminophenyl)-5-(4-chlorophenyl)-imidazolidine-2,4-dione ,(b)5-( 4-dimethyl aminophenyl )-5-( 2-nitrophenyl )-imidazolidine-2,4-dione , (c)5-(4-chlorophenyl)-5-(4hydroxyphenyl)-imidazolidine-2,4-dione, (d)5-(4-chlorophenyl)-5-(3-bromophenyl)-imidazolidine-2,4-dione,

(e)5-(4-chlorophenyl)-5-(4-aminophenyl)-imidazolidine-2,4-dione show strong band observed at 1600 cm<sup>-1</sup> indicate stretching vibration of the  $\nu(\text{C}=\text{O})$  <sup>(7)</sup> their compound show weak band at 3120 cm<sup>-1</sup> due to  $\nu(\text{C}-\text{H})$  aromatic and show absorption band at 1595 cm<sup>-1</sup> due to  $\nu(\text{C}=\text{N})$  of imidazole ring.

In (a), (d) (c), and (e) compounds the spectrum show strong band at ~ (650-800) cm<sup>-1</sup> indicate stretching vibration of the  $\nu(\text{Cl})$  <sup>(8)</sup>

In (a), (b) compounds the spectrum show strong band at 2962 cm<sup>-1</sup> indicate stretching vibration of the  $\nu(\text{C}-\text{H})$  aliphatic .

In (b) compound the spectrum show strong band at (1500-1580), (1300-1380) cm<sup>-1</sup> indicate stretching vibration of the  $\nu(\text{NO}_2)$  <sup>(9,10)</sup>

The characteristic broad band around (3325) cm<sup>-1</sup> indicates the  $\nu(\text{OH})$  stretching in the spectrum of in (c) compound .

In (d) compound the spectrum show strong band at ~ (650-800) cm<sup>-1</sup> indicate stretching vibration of the  $\nu(\text{Br})$  <sup>(11)</sup>

**Table (1) :- Characterisation data for IR absorption bands of the derivatives hydantions in cm<sup>-1</sup> units (KBr disc).**

compound	$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{N})$ imidazole ring.	$\nu(\text{C}-\text{H})$ aromatic	$\nu(\text{Cl})$	$\nu(\text{OH})$	(C-H) aliphatic	$\nu(\text{NH}_2)$	$\nu(\text{NO}_2)$	$\nu(\text{Br})$
a	1600	1595	3120	650-800		2962			
b	1600	1595	3120			2962		1500-1580	
c	1600	1595	3120	650-800	3325				
d	1600	1595	3120	650-800					650-800
e	1600	1595	3120	650-800			3350		

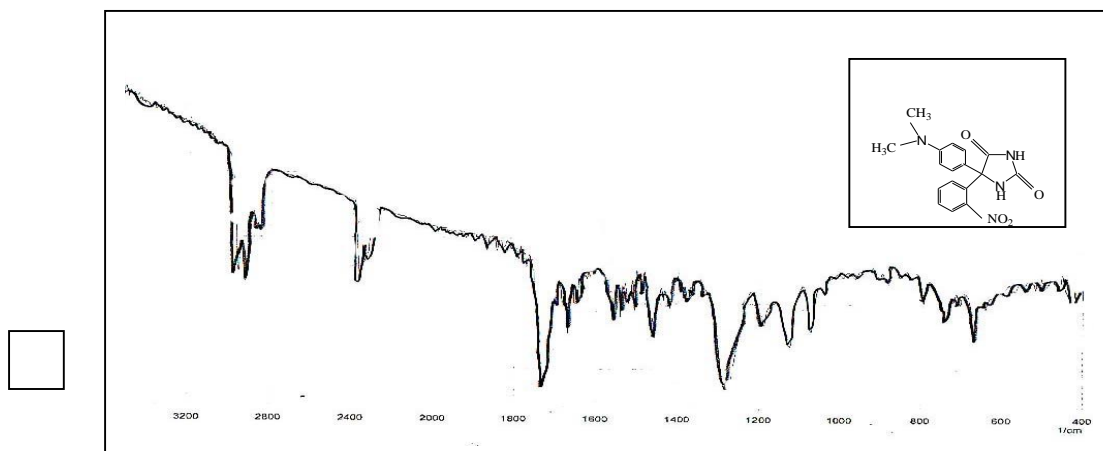


Fig.(2):- IR spectrum of (b)

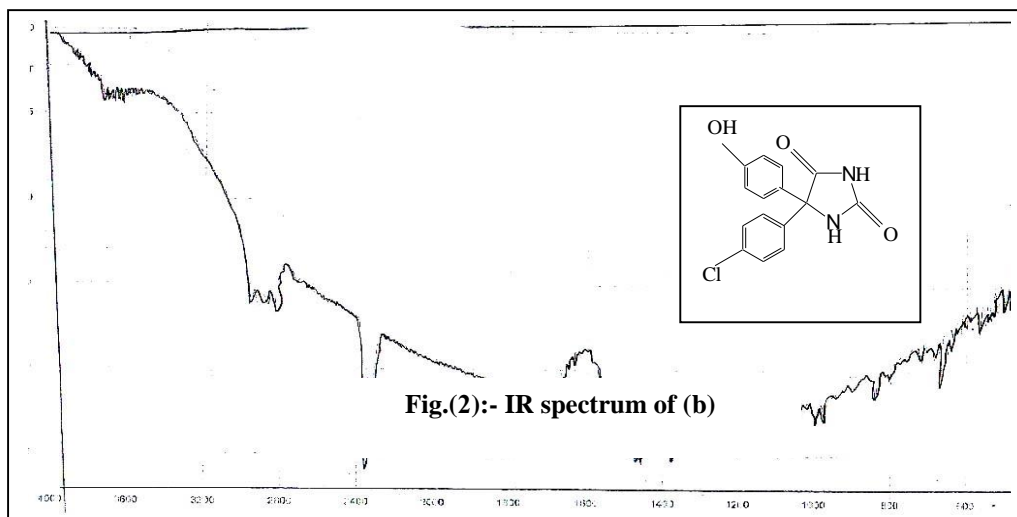
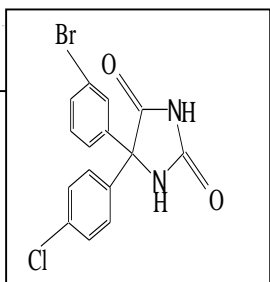
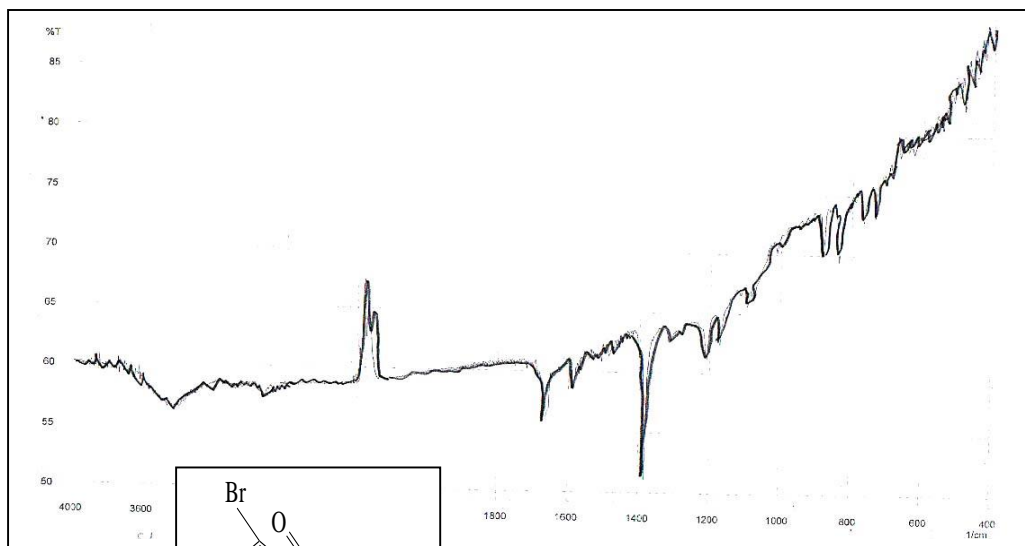
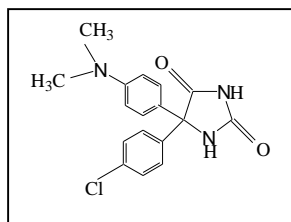


Fig.(2):- IR spectrum of (b)



spectrum of (c)

Fig.(4):- IR spectrum of (d)

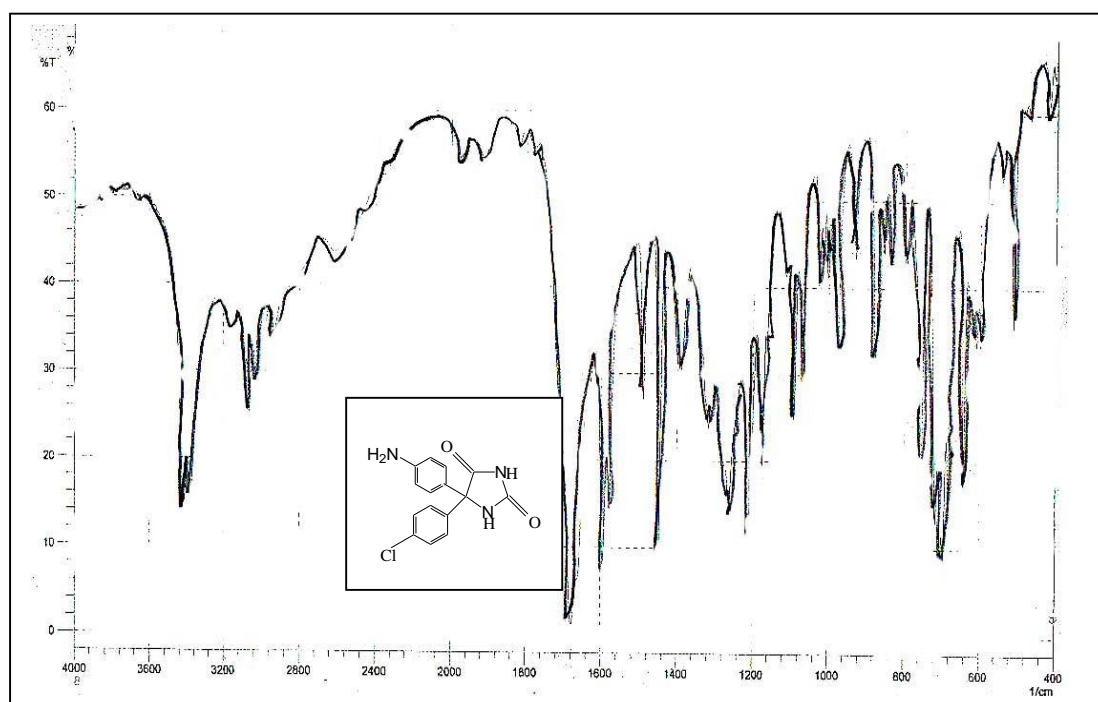
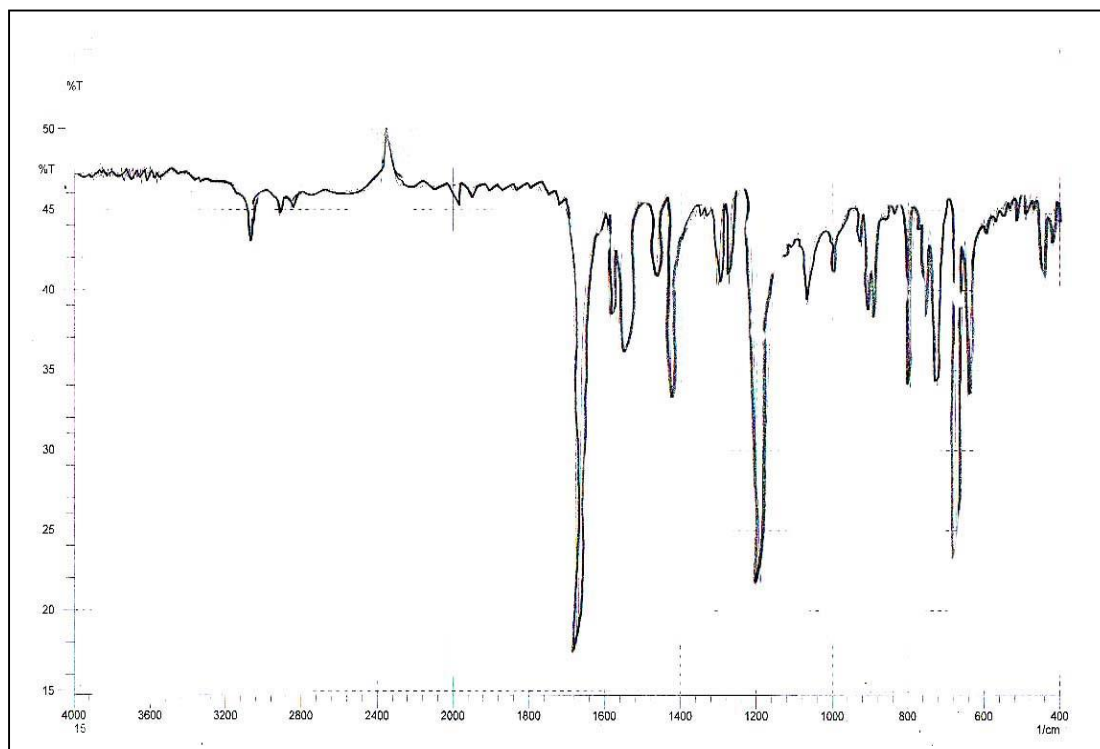


Fig.(5):- IR spectrum of (e)

Table(2):- Analytical and physical data of the derivatives hydantions.

Compound	Color	m.P°C	Yield %	Molecular formula (Mol.Wt)	Found (Calc.)%		
					C	H	N
a	purple	133	88	C <sub>17</sub> H <sub>16</sub> N <sub>3</sub> O <sub>2</sub> Cl (345)	59.130 (59.112)	4.637 (4.534)	12.173 (12.212)
b	red	124.	79	C <sub>17</sub> H <sub>16</sub> N <sub>4</sub> O <sub>4</sub> (340)	60.00 (60.01)	4.7 (4.59)	16.470 (16.512)
c	Dark	120	86	C <sub>15</sub> H <sub>11</sub> N <sub>2</sub> O <sub>3</sub> Cl (302)	59.6 (59.48)	3.642 (3.563)	9.271 (9.154)
d	purple	97	90	C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub> Br <sub>2</sub> Cl (365)	49.315 (49.251)	2.739 (2.439)	7.671 (7.759)
e	red	133	77	C <sub>15</sub> H <sub>12</sub> N <sub>3</sub> O <sub>2</sub> Cl (301)	59.800 (59.740)	3.986 (3.847)	13.953 (13.698)

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## تخليق مركبات الاميدزولين بانتقال مجموعته البنزويل

شيماء عدنان بهجت

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### الخلاصه:-

تم تحضير مشتقات الاميدازول بوجوده 4-كلورو-4-ثنائي مثيل اميون بنزويل ، 2-نايترو-4-ثنائي مثيل امينو بنزويل ، 4-كلورو-4-هيدروكسي بنزويل ، 4-كلورو-3-برومو بنزويل ، 4-كلورو-4-امينو بنزويل مع اليوريا وحصلنا على المركبات التاليه :- 5-(4-ثنائي مثيل امينو فنيل)-5-(4-كلورو فنيل)-اميدازول-4-داي اون ، 5-(4-ثنائي مثيل امينو فنيل)-5-(2-نايترو فنيل)-اميدازول-4-داي اون ، 5-(4-كلورو فنيل)-5-(4-هيدروكسي فنيل)-اميدازول-4-داي اون ، 5-(4-كلورو فنيل)-5-(3-برومو فنيل)-اميدازول-4-داي اون ، 5-(4-كلورو فنيل)-5-(4-امينو فنيل)-اميدازول-4-داي اون وقد تم تشخيص المركبات بواسطه طيف الاشعه تحت الحمراء الـ IR و تقنيه تحليل العناصر الدقيق الـ C.H.N-