

# Synthesis and Characterization of New Symmetrical Pyromellitdiimide Derivatives and Their Amic Acids

E. T. Ali , K. M. Lazim AL-Aliawy and J. H. Tomma

Department of Chemistry, College of Education , Ibn Al-Haitham University of Baghdad

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## Abstract

The new symmetry pyromellitdiimide [VII]<sub>a-c,n</sub> were synthesized by two-step reactions from the corresponding pyromellitic dianhydride . A new symmetrical amic acid [VI]<sub>a-c,n</sub> was synthesized by the reaction of pyromellitic dianhydride with different heterocyclic amines in dry acetone . The second reaction step includes intramolecular cyclization of amic acid in the presence of sodium acetate -acetic anhydride system at 85<sup>0</sup>C. Structures of the synthesized compounds have been ascertained by their melting points , C.H.N analysis , UV-Vis, FTIR and <sup>1</sup>HNMR spectroscopy.

**Key words :** Pyromellitdiimide , Pyromellitic dianhydride , Pyromellitamic diacid

## Introduction

Pyromellitdiimides are best known as segments of highly insulating polyimides dielectrics, it is though nevertheless quit surprising that two attempt has been made to fabricate transistor from pyromellitdiimide derivatives . Pyromellitdiimides derivatives can be easily prepared by one –step reaction between pyromellitic dianhydride and various amines . The facile synthesis of pyromellitdiimide derivatives offers an advantage in larges scale synthesis due to its high conversion yield and ease of purification . Many workers synthesized new pyromellitimide derivatives by using this method in different conditions[1-7].

Another method to synthesis pyromellitdiimide derivatives includes two step, firstly synthesis of amic acid by nucleophilic addition of amine to acid anhydride, secondly condensation amic acid by heating to yield pyromellitdiimide by ring closure [8-11].

The pyromellitimides containing heterocyclic units very rare [10,12] , therefore we decided to synthesis and characterized the new amic acid containing thiazole, benzothiazole or oxadiazole unit and their pyromellitdiimide derivatives.

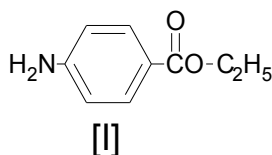
## Experimental

**Materials:** All the chemicals were supplied from Merck , Fluka ,GCC and Aldrich chemicals Co. and used as received.

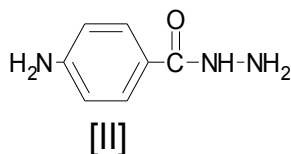
**Techniques :** FTIR spectra were recorded using potassium bromide discs on a shimadzu (8400S) and shimadzu (IR Prestige-21). <sup>1</sup>HNMR spectra were carried out by company : Bruker , model: ultra shield 300 MHz , origin : Switzerland and are reported in ppm(S), DMSO was used as solvent with TMS as an internal standard. Measurements were made at chemistry department, AL-Albyat university, Elemental microanalysis (C.H.N) were carried out by a (C.H.N) company : Euro vector , model EA3000A origin : Italy . AL-Albayt University, Jordan . Uncorrected melting points were determined by using Hot-Stage, Gallen Kamp melting point apparatus .UV-Vis spectra of solution were performed on CECL 7200 England Spectrophotometer using CHCl<sub>3</sub> as a solvent.

### Synthesis methods

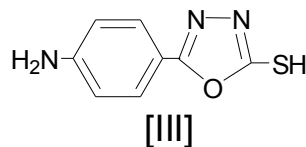
Ethyl-4-Amino benzoate [I]: was prepared following the procedure by Vogel [13] , yield 99%, m.p.91<sup>0</sup>C, lit 91<sup>0</sup>C .



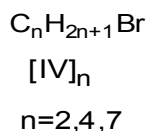
4-amino phenyl hydrazide[II]: This compound was prepared according to the let.[14] , yield 99.5%, m.p. 223<sup>0</sup>C, lit 223<sup>0</sup>C.



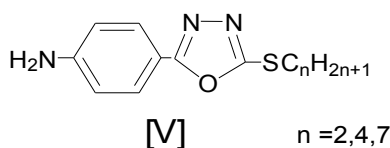
5-(4-amino phenyl)-1,3,4-Oxadiazole-2-thiol [III]: This compound was prepared according to the let. [15], yield 79%, m.p. 239<sup>0</sup>C.



n-alkyl bromide[IV]<sub>n</sub>: was prepared following the procedure by Vogel [13].

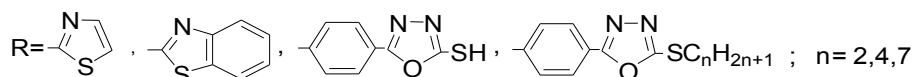
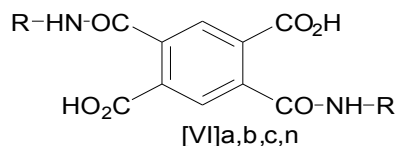


2-alkyl-thio-5-[4-amino phenyl ]-1,3,4-oxadiazole[V]:



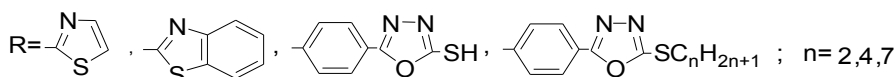
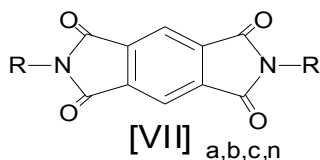
Potassium hydroxide (0.68g, 0.12mole) dissolved in minimum volume of water, was added drop wise to a stirred solution of oxadiazole (1.93 g , 0.01 mole) in (10mL) of dioxane at 25°C. After heating the mixture for (15min.) and cooling , n-alkyl bromide (0.01mole) was added drop wise . The solution was refluxed for (2 hours) ; afterwards the solvent was evaporated on a rotator evaporator. Ice-water (100 mL) was added, the resulting precipitate was collected [16] , and recrystallized from ethanol. Yield(85-92)% .

**symmetrical ( N,N-pyromellitic diacid)[VI]:**



To a solution of pyromellitic dianhydride (0.218g, 0.001mole) in (15mL) acetone, a solution of the amine (0.002 mole) in (15mL) acetone was added dropwise during one hour, the mixture was then left at room temperature with continuous stirring for (24 hours), the product was then filtered off and recrystallized from acetone or chloroform. The physical properties of these compounds are given in Table 1.

Symmetrical ( N,N-pyromellitdiimide)[ VII]



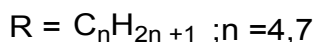
A(0.01 mole) of N,N-pyromellitic diacid was placed in (250 mL) round bottom flask, a mixture of anhydrous sodium acetate ( 0.02mole ) and acetic anhydride (15mL) was added. The mixture was maintained between(85-90)<sup>o</sup>C by means of a water -bath and stirred for half hour . The mixture was allowed to stir for one hour at room temperature . The mixture was poured on distilled water (400mL ) and worked out as specified in each case. The physical data of diimides[VII] are listed in Table (2).

#### Elemental analysis of compound [VII]<sub>7</sub> :

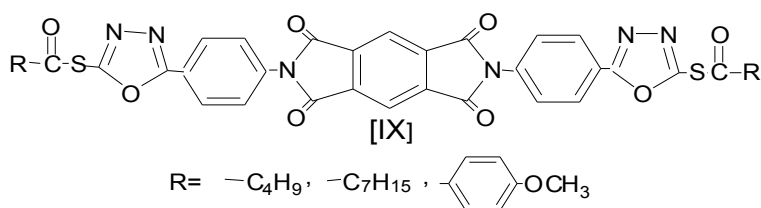
**Theoretical :** C% = 62.82 , H% = 5.23 , N% = 10.99

**Found :** C% = 62.856 , H% = 5.315 , N% = 11.032

Carboxylic Acid Chlorides [VIII]<sub>b,c</sub>: was prepared following the procedure by Vogel [13].



N,N-Bis-[4-(2-thioacyl-1,3,4-oxadiazole-5-yl)-phenyl]-pyromellitdiimid [IX]<sub>a-c</sub>



To a stirred of compound [VII]<sub>C</sub> (0.568g, 0.001mol), triethylamine (0.2g, 0.002mol) in dried mixture of (5ml DMF:10ml THF), was added dropwise carboxylic acid chloride [VIII] (0.001mol) at (0-4)<sup>o</sup>C. After the addition had been completed the resulting suspension was stirred at the same temperature for 3hrs .The triethylaminhydrochloride salt was precipitate . It was filtered and the filtrate was poured with stirring into (100 mL) ice-water then the mixture was extracted by adding (50mL) of diethyl ether. Yield(77-90)% .

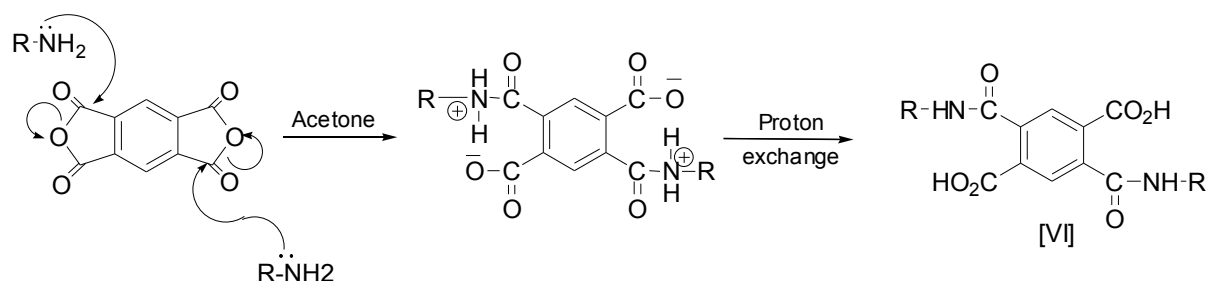
## Result and Discussion

Ethyl amino benzoate [I] was obtained from esterification of the carboxylic acid moiety of 4-aminobenzoic acid by using absolute ethanol saturated with dry (HCl) gas. Condensation this ester [I] with hydrazide hydrate yielded the 4-amino phenyl hydrazide [II], which is characterization by higher melting point and by FTIR-spectrum, revealed stretching vibration at  $3237\text{ cm}^{-1}$  of N-H group as well as stretching absorption at  $1632\text{ cm}^{-1}$  due to C=O (amid) group. The reaction of acid hydrazide[II] with  $\text{CS}_2$  in KOH medium leads to the formation of thio-oxadiazole [III] by intramolecular cyclization reaction. The structure of oxadiazole was confirmed by melting point and by spectroscopy.

The FTIR-absorption spectrum showed the disappearance of absorption bands due to C=O (amid) and N-H groups of acid hydrazide together with the appearance of a stretching bands at  $1630\text{ cm}^{-1}$ ,  $1308\text{ cm}^{-1}$ ,  $3102\text{ cm}^{-1}$  which are assigned to C=N, C=S and N-H groups, respectively of oxadiazole unit. It also shows stretching bands in the region  $(1050-1260)\text{cm}^{-1}$  due to (C-O-C) oxadiazole ring.

2-alkyl-thio-5-[4-aminophenyl]-1,3,4-oxadiazole  $[\text{V}]_n$  were prepared by the reaction of 2-mercapto-oxadiazole with different n-alkyl bromides in basic medium. The structure of these compounds were identified by FTIR spectroscopy. The FTIR spectra of these compounds showed disappearance of the two peaks attributed to N-H and C=S stretching and appearance of many peaks in the region  $(2855-2967)\text{ cm}^{-1}$  due to aliphatic C-H stretching.

The amic acid [VI] were synthesized by the reaction of one mole of pyromellitic dianhydride with two moles of different amines in acetone as solvent. The mechanism involves nucleophilic addition reaction, as follows scheme (1).



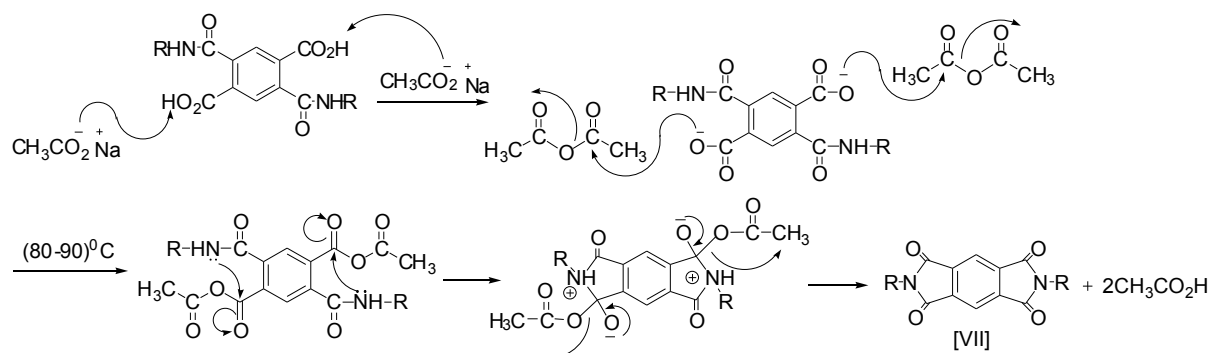
**Scheme (1)**

The structure of these acids was studied by FTIR, UV-Vis and  $^1\text{HNMR}$  spectroscopy. The FTIR absorption-spectra showed the disappearance of absorption bands due to  $\text{NH}_2$  and other peaks characterized of cyclic anhydride of the starting materials together with the appearance of new absorption stretching bands due to O-H of carboxylic moiety at  $(2500-3450)\text{cm}^{-1}$ , C=O (carboxylic acid) at  $(1688-1716)\text{cm}^{-1}$ , C=O (amid) at  $(1645-1678)\text{cm}^{-1}$  and C=N (endocyclic) at  $(1609-1628)\text{cm}^{-1}$  of heterocyclic unit.

The FTIR data of functional groups which are characteristic of the these new amic acid  $[\text{VI}]_{a-f}$  and the UV-Vis data ( $\lambda_{\text{max}}$ ) are listed in Table 3.

$^1\text{H}$ NMR spectrum of compound  $[\text{VI}]_{n=4}$ , showed the following characteristic chemical shift (DMSO as a solvent): A triplet band at  $\delta$  1.40-1.45ppm due to terminal methyl proton. Four protons multiplet at  $\delta$  1.69-1.97ppm that could be related to  $-\text{CH}_2\text{CH}_2-$  protons and two protons triplet at  $\delta$  3.23-3.27ppm due to  $\text{SCH}_2$ . A singlet signal at  $\delta$  4.77ppm for protons of N-H, eight aromatic protons appear as two doublets bands at  $\delta$  6.64-6.67ppm and  $\delta$  7.58-7.61ppm. A good sharp signal at  $\delta$  7.94 ppm due to the two protons of centre aromatic ring. Finally, two protons of carboxylic moiety appear as a broad weak band at  $\delta$  12.8-14.1ppm.

Pyromellitimides  $[\text{VII}]_{a-f}$  were obtained by the intramolecular cyclization of amic acid  $[\text{VI}]_{a-f}$  in acetic anhydride and sodium acetate at  $85^\circ\text{C}$ . The mechanism involves nucleophilic substitution reaction as follows of scheme (2).



Scheme (2)

The structure of these compounds was studied by FTIR, UV-Vis and  $^1\text{H}$ NMR spectroscopy.

The FTIR spectra exhibited significant two peaks in region  $(1700-1800)\text{cm}^{-1}$  which could be attributed to stretching vibration of the carbonyl of the (imide cyclic) and two absorption bands around  $1180\text{cm}^{-1}$  and  $775\text{cm}^{-1}$  for asym. and sym. of C-N-C (cyclic). They also show the disappearance of absorption stretching bands of N-H, O-H and C=O (carboxylic moiety) groups of amic acid.

The FTIR absorption bands data of these compounds  $[\text{VII}]_{a-f}$  and the UV-Vis data ( $\lambda_{\text{max}}$ ) are listed in Table (4).

$^1\text{H}$ NMR spectrum of pyromellitimide  $[\text{VII}]_c$ , (DMSO as a solvent) showed a good peak at  $\delta$  2.69 ppm due to S-H group, eight aromatic protons of (AB) which are typical for p-disubstituted benzene appear as two doublets at  $\delta$  7.80-7.91 ppm [17]. A singlet signal at  $\delta$  7.95ppm for two protons of centre benzene ring and a sharp single at  $\delta$  10.38 ppm due to N-H group.

Elemental analysis result of compound  $[\text{VII}]_7$  is in good agreement with the theoretical data.

N,N-Bis-[4-(2-thioacyl-1,3,4-oxadiazole-5-yl)-phenyl] pyromellitimide  $[\text{IX}]_{a-c}$  synthesized by the reaction of  $[\text{VII}]_c$  with acid chloride  $[\text{VIII}]_{a-c}$  in mixture of (DMF+THF) in the basic medium  $\text{Et}_3\text{N}$ . The FTIR spectra showed a good peak at  $(1686-1715)\text{cm}^{-1}$  due to C=O thioester. The FTIR data of thioester  $[\text{IX}]_{a-c}$  showed in Table 4.

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**Table (1) The physical properties of symmetrical diamic acid [VI]<sub>a</sub>-[VI]<sub>7</sub>**

| Com. No.          | Nomenclature  | Structural formula | Molecular formula  | M.P °C  | Yield % | Color  |
|-------------------|---|--------------------|--|---------|---------|--------|
| [VI] <sub>a</sub> | N,N'-Bis-(2-thiazole)-pyromellitic diacid                                     |                    | C <sub>16</sub> H <sub>10</sub> N <sub>4</sub> O <sub>6</sub> S <sub>2</sub> | >300    | 89      | white  |
| [VI] <sub>b</sub> | N,N'-Bis-(2-benzothiazole)-pyromellitic diacid.                               |                    | C <sub>24</sub> H <sub>14</sub> N <sub>4</sub> O <sub>6</sub> S <sub>2</sub> | >300    | 91      | white  |
| [VI] <sub>c</sub> | N,N'-Bis-[4-(5-phenyl)-2-thio-1,3,4-oxadiazole]-pyromellitic diacid.          |                    | C <sub>26</sub> H <sub>16</sub> N <sub>6</sub> O <sub>8</sub> S <sub>2</sub> | >300    | 96      | orange |
| [VI] <sub>2</sub> | N,N'-Bis-[4-(2-ethylthio-1,3,4-oxadiazole-5-yl)-phenyl] pyromellitic diacid.  |                    | C <sub>30</sub> H <sub>24</sub> N <sub>6</sub> O <sub>8</sub> S <sub>2</sub> | >300    | 89      | Orange |
| [VI] <sub>4</sub> | N,N'-Bis-[4-(2-butylthio-1,3,4-oxadiazole-5-yl)phenyl]-pyromellitic diacid.   |                    | C <sub>34</sub> H <sub>32</sub> N <sub>6</sub> O <sub>8</sub> S <sub>2</sub> | 276-278 | 90      | Orange |
| [VI] <sub>7</sub> | N,N'-Bis-[4-(2-heptylthio-1,3,4-oxadiazole-5-yl)-phenyl]-pyromellitic diacid. |                    | C <sub>40</sub> H <sub>44</sub> N <sub>6</sub> O <sub>8</sub> S <sub>2</sub> | 238     | 88      | Yellow |



Table (2):The physical properties of symmetrical pyromellitdiimide

[VII]<sub>a-c,n</sub> and [IX]a-c.

| Co<br>m.<br>N<br>o. | Nomenclature  | Structural formula | Molecular<br>formula   | M.P <sup>o</sup><br>C    | Yiel<br>d% | Colo<br>r      |
|---------------------|---|--------------------|--|--------------------------|------------|----------------|
| [VII]<br>]a         | N,N <sup>-</sup> -Bis-(2-thiazole)-pyromellitdiiimide                                       |                    | C <sub>16</sub> H <sub>6</sub> N <sub>4</sub> O <sub>4</sub><br>S <sub>2</sub> | 198-<br>200              | 78         | Pale<br>yellow |
| [VII]<br>]b         | N,N <sup>-</sup> -Bis-(2-benzothiazole)-pyromellitdiiimide                                  |                    | C <sub>24</sub> H <sub>10</sub> N <sub>4</sub> O<br>4S <sub>2</sub>            | >300                     | 90         | yellow         |
| [VII]<br>]c         | N,N <sup>-</sup> -Bis-{[4-(2-thio-1,3,4-oxadiazole)-5-yl]-phenyl} - pyromellitdiiimid.      |                    | C <sub>26</sub> H <sub>12</sub> N <sub>6</sub> O<br>6S <sub>2</sub>            | 182-<br>184              | 92         | Pale<br>yellow |
| [VII]<br>]2         | N,N <sup>-</sup> -Bis-[4-(2-ethylthio-1,3,4-oxadiazole-5-yl)-phen-yl] - pyromellitdiiimide. |                    | C <sub>30</sub> H <sub>20</sub> N <sub>6</sub> O<br>6S <sub>2</sub>            | 160-<br>162              | 85         | orange         |
| [VII]<br>]4         | N,N <sup>-</sup> -Bis-[4-(2-butylthio-1,3,4-oxadiazole-5-yl) phenyl]- pyromellitdiiimide.   |                    | C <sub>34</sub> H <sub>28</sub> N <sub>6</sub> O<br>6S <sub>2</sub>            | 138-<br>140              | 88         | Pale<br>yellow |
| [VII]<br>]7         | N,N <sup>-</sup> -Bis-[4-(2-heptylthio-1,3,4-oxadiazole-5-yl)phenyl] - pyromellitdiiimide   |                    | C <sub>40</sub> H <sub>40</sub> N <sub>6</sub> O<br>6S <sub>2</sub>            | 92-94                    | 75         | Yello<br>w     |
| [IX]<br>a           | N,N-Bis-[4(2-thio-4-(methoxyphenoxy)-5-yl)-phenyl]- pyromellitdiiimide                      |                    | C <sub>42</sub> H <sub>24</sub> N <sub>6</sub> O<br>10S <sub>2</sub>           | oily                     | 80         | yellow         |
| [IX]<br>b           | N,N-Bis-[4-(2-thio-pentyloxy-1,3,4-oxadiazole-5-yl)-phenyl ] - pyromellitdiiimide           |                    | C <sub>36</sub> H <sub>28</sub> N <sub>6</sub> O<br>8S <sub>2</sub>            | oily                     | 77         | Brown          |
| [IX]<br>c           | N,N-Bis-[4(2-thio-heptyloxy-1,3,4-oxadiazole-5-yl)-phenyl ]- pyromellitdiiimide             |                    | C <sub>42</sub> H <sub>40</sub> N <sub>6</sub> O<br>8S <sub>2</sub>            | Crysta<br>l<br>Gum<br>my | 90         | yellow         |

**Table(3):Characteristic FTIR absorption bands and UV data ( $\lambda_{\max}$ ) of compounds [VI]<sub>a</sub>-**

| Comp<br>.No.      | $\lambda_{\max}$<br>(nm) | V N-<br>H | VO-H          | VC-H<br>aromatic | VC-H<br>aliphatic | VC=O<br>carboxylic | VC=<br>O<br>amid | VC=N<br>endocyclic | VC=C<br>aromatic |
|-------------------|--------------------------|-----------|---------------|------------------|-------------------|--------------------|------------------|--------------------|------------------|
| [VI] <sub>a</sub> | 266                      | 3302      | 3450-<br>2400 | 3113             |                   | 1695               | 1659             | 1616               | 1580             |
| [VI] <sub>b</sub> | 264                      | 3289      | 3200-<br>2450 | 3115             |                   | 1688               | 1655             | 1628               | 1591             |
| [VI] <sub>c</sub> | 318                      | 3352      | 3450-<br>2500 | 3103             |                   | 1709               | 1678             | 1625               | 1607             |
| [VI] <sub>2</sub> | 306                      | 3368      | 3400-<br>2400 | 3086             | 2967-<br>2855     | 1709               | 1670             | 1609               | 1587             |
| [VI] <sub>4</sub> | 300.<br>5                | 3350      | 3435-<br>2450 | 3077             | 2957-<br>2860     | 1716               | 1645             | 1609               | 1582             |
| [VI] <sub>7</sub> | 304                      | 3348      | 3250-<br>2500 | 3075             | 2955-<br>2855     | 1710               | 1645             | 1609               | 1589             |

**Table(4):Characteristic FTIR absorption bands and UV data ( $\lambda_{\max}$ ) of compounds [VII]<sub>a-c,n</sub> and [IX]a-c**

| Com p.No.          | $\lambda_{\max}$ (nm) | VC-H arom. | VC-H alipha.  | VC=O<br>Imides<br>Cyclic<br>asym.,sym | VC=N<br>endocycl<br>ic | VC=C<br>aromati<br>c | VC-N-C<br>asym.,sym | Other                              |
|--------------------|-----------------------|------------|---------------|---------------------------------------|------------------------|----------------------|---------------------|------------------------------------|
| [VII] <sub>a</sub> | 268                   | 3113       |               | 1800,1728                             | 1685                   | 1600                 | 1172,779            |                                    |
| [VII] <sub>b</sub> | 250                   | 3113       |               | 1787,1740                             | 1634                   | 1597                 | 1195,772            |                                    |
| [VII] <sub>c</sub> | 302                   | 3100       |               | 1728,1695                             | 1622                   | 1591                 | 1180,760            | VN-H at<br>3337<br>VC=S at<br>1317 |
| [VII] <sub>2</sub> | 302.5                 | 3100       | 2960-<br>2850 | 1740,1701                             | 1605                   | 1576                 | 1179,775            |                                    |
| [VII] <sub>4</sub> | 300                   | 3110       | 2955-<br>2868 | 1736,1700                             | 1607                   | 1578                 | 1182,765            |                                    |
| [VII] <sub>7</sub> | 300                   | 3051       | 2955-<br>2865 | 1740,1710                             | 1601                   | 1570                 | 1175,768            |                                    |
| [IX] <sub>a</sub>  | 310                   | 3080       | 2959-<br>2859 | 1728,1700                             | 1640                   | 1606                 | 1165,779            | VC=O<br>thioester<br>1710          |
| [IX] <sub>b</sub>  | 306.5                 | 3100       | 2989-<br>2872 | 1724,1701                             | 1645                   | 1610                 | 1175,780            | VC=O<br>thioester<br>1715          |
| [IX] <sub>c</sub>  | 315                   | 3080       | 2959-<br>2872 | 1740,1710                             | 1640                   | 1607                 | 1171,774            | VC=O<br>thioester<br>1686          |

# تحضير وتشخيص مشتقات جديدة متماثلة للبايرومليت ثنائي الايميد وحوامض

## الاميك لها

عماد تقي علي ، كاظم ماذي لازم العلياوي ، جمبد هرمز توما

قسم الكيمياء ، كلية التربية-ابن الهيثم ، جامعة بغداد

استلم البحث في : 29 آذار 2011

قبل البحث في : 30 آيار 2011

## الخلاصة

حضرت مركبات البايرومليت ثنائية الايميد [VII] عن طريق تفاعل ذي خطوتين ، من ثنائي انهدريد البايروميليتيك المقابل ، اذ حضرت حوامض الاميك الجديدة من تفاعل ثنائي انهدريد البايروميليتيك مع أمينات مختلفة تحتوي حلقة غير متجانسة باستعمال الاسيتون الجاف مذيباً. بينما تتضمن خطوة التفاعل الثاني الغلق الحلقي الضمني لجزيئة حامض الاميك بوجود انهدريد الخليك-خلات الصوديوم وبدرجة حرارة  $(80-90)^{\circ}\text{C}$  .

اثبتت صحة تراكيب المركبات المحضرة بوساطة قياس درجات انصهارها وتحليل العناصر وأطياف-UV

: FTIR ,  $^1\text{HNMR}$  Vis لها.

**الكلمات المفتاحية :** بايرومليت ثنائي الايميد ، بايرومليتيك ثنائي الانهدريد ، حامض البايروميليتاميك