Synthesis and Characterization of New Heterocyclic Derived from Pyromellitic Dianhydride

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Abstract

In this work two moles of 2-amino benzothiazole were allowed to react with one mole of pyromellitic dianhydride to produce N,N⁻-Bis-(benzathiazol-2-yl) pyromellitamic diacid [I] which was submitted to esterification via the reaction with dimethyl sulphate in sodium carbonate in acetone as a solvent to synthesize N,N⁻-bis-(benzothiazol-2-yl) pyromellitam diacetate [II] .This ester was used to produce novel compounds through two paths :- Path one:-

Reaction of ester [II] with hydrazine in ethanol as a solvent to form the corresebonding N,N⁻-bis (benzothiazole-2-yl) –pyromellitamic acid hydrazide [III] which react with acetyl acetone in ethanol or with phthalic anhydride in dioxane to yield new pyrazole, N,N⁻-bis[(2-amidobenzothiazol) 3,5-dimethyl pyrazole] pyromellitic [IV] . and new imide; N,N⁻-bis-[(2-amidobenzothiazol)-amido phthalimide] pyromellit [V] respectively. Path two:-

Reaction of ester [II] with thiosemicarbazide to form bis $[N,N^{-}(2-amidobenzothiazole)$ thiosemicarbazide] pyromellitic acid [VI], which is converted to triazole. bis $[N,N^{-}(2-amido benzothiazol) 1,2,4-triazole -3-yl]$ pyromellit [VII] under basic condition treatment of the last product with different alkyl halides to give new series of thio-alkyl triazoles; bis $[N,N^{-}(2-amido benzothiazole)-5-thio alkyl 1,2,4-triazole-3-yl]$ pyromellit[VIII]_n

All the above compoundes were characterized by their melting points, elemental analysis and by their spectral data,FTIR and ¹HNMR for (some of them).

Key word : pyromellitic dianhydride , pyrazol , imide ,1,2,4- triazole , hydrazide.

Introduction

The wide spread applications of pyromellitic dianhydride (PMDA) in several fields, such as synthesis of an important monomer in the preparation of a variety of thermoplastic polymers such as polyimides, epoxy resins and metal carboxylate complexes. Moreover, it is also useful in the preparation of high performance coatings that have been widely employed in many fields in our daily life because of its excellent thermal, oxidative stability and excellent mechanical properties [1].

Pyrazoles played a crucial part in the development of theoretical studies and also useful building blocks in organic synthesis , with wide applications as dyestuff, analytical reagents and agrochemicals . The pyrazole ring system is a useful structural moiety found in numerous biologically active compounds. and in the field of the medicinal chemistry . They display various biological activities such as, analgesic , anti-inflammatory , anti anxiety , antibacterial , antifungal , antitumour , antitubercular , and antiparasitic [2] .

Imides possess excellent mechanical properties, thermal stability and important class of bioactive molecules that show a wide range of pharmacological activities such as androgen receptor antagonistic, anti-inflammatory, anxiolytic, antiviral, antibacterial, and antitumor properties. Moreover, they are synthetic precursors with application in organic synthesis, polymer synthesis, and for the development of new materials and molecular electronic devise [3].

A large number of 1,2,4-triazole-containing ring system have been incorporated into a wide variety of therapeutically interesting drug candidates including Anti-septic, analgesic, anti-convulsant, anti-biotic, anti-allergic, anti-inflammatory, diuretic, fungicidal, insecticidal, herbicidal, anti-bacterial, anti-viral, anti-depressant, anti-microbial, anti-tumor, and antihypertensive [4].

The aim of this work is the synthesis of a novel pyrazole ,imides , 1,2,4- triazole derivatives from pyromellitic anhydride by many reaction steps which are given in scheme 1

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 Shimadzo (Ir prestige-21) FTIR spectroscopy. Uncorrected melting points were determined by using Hot-Stage, Gallen Kamp melting point apparatus, ¹HNMR spectra were carried out by company : Ultra Shield 300 MHz, Bruker, Switzerland, at University of Al-albayt , Jordan, and are reported in ppm(δ), DMSO was used as a solvent with TMS as an internal standard . Elemental analyses of some compounds were performed on a (C.H.N.S) Company: Euro vector, model EA 3000A origin: Italy. University of Al-albayt, Jordan

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 $R{=}C_{n}H_{2n+1}$, n=1-5 x=Br or I

Scheme 1

Synthesis of asymmetrical diamic acid N,N⁻-Bis-(benzothiazol-2-yl) pyromellitamic diacid [I]

To a solution of pyromellitic dianhydride (0.218g, 0.001mole) in (15mL) acetone , a solution of the 2-amino benzothiazol (0.3 g, 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 hrs, the white product was then filtered off and recrystallized from acetone to give the a corresponding N,N⁻-Bis-(benzothiazol-2-yl) pyromellitamic diacid [I] [5], yield 90%, mp >300 $^{\circ}$ C.

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Synthesis of N,N⁻-bis-(benzothiazol-2-yl) pyromellitam diacetate [II]

A mixture of compound [I]. (6.73g, 0.013mol) and anhydrous sodium carbonate (2.7 g, 0.026 mol) was dissolved in 25mL of acetone, to this solution (0.026 mol) of dimethyl sulphate was added then 20 min after, the resulting mixture was heated under reflux for 4 hrs. The reaction mixture was allowed to cool down at room temperature, extraction with chloroform. The product was collected by evaporating the chloroform to give a white compound[II] [6], yield 90% ,mp(174-176)^oC.

Elemental analysis of compound [II] :

Theoretical : C% = 57.14 , H% = 3.29 , N% = 10.25 S%=11.72 Found : C% = 57.30 , H% = 3.42 , N% = 9.16 S%=11.61 Path one:

Synthesis of N,N⁻-bis (benzothiazol-2-yl)-pyromellitamic acid hydrazide [III] A solution of N,N⁻-bis-(benzothiazol-2-yl) pyromellitam diacetate [II] (32.76 g,0.06 mol) and hydrazine hydrate (15mL) in 25 mL of ethanol was heated under reflex for 2 hrs. The mixture was then cooled at room temperature, and the obtained pale orange solid was filtered and recrystallized from ethanol .yield 86% ,mp135-137^oC.

Synthesis of N,N⁻-bis[(2-amido benzothiazol) 3,5 dimethyl pyrazole] pyromellitic [IV]

A mixture of acid hydrazide [III] (1.09 g, 0.002 mol) and acetyl acetone (0.002 mol) in absolute ethanol 4mL was heated under reflux for 4 hrs. The reaction mixture was cooled and the formed precipitate was filtered off and recrystallized to give yellow compound [IV], yield 71%, mp162-164^oC.

Synthesis of N,N⁻-bis-[(2-amidobenzothiazol)-amido phthalimide] pyromellit[V]

A mixture of acid hydrazide [III] (1.09g, 0.002mol) and phthalic anhydride (0.59 g,0.002mol) was refluxed in dry dioxane (20) mL for 5 hrs. The reaction mixture was left overnight for slow evaporation. The browen product was recystallized from dioxane to give new imide [V], yield 60 %, mp >300^oC.

Path two:

Synthesis of symmetrical bis [N,N⁻- (2-benzothiazol) thiosemicarbazide] pyromellitamic acid [VI]

To a solution of compound [II] (0.54 g, 0.01mol) in absolute ethanol (20mL) was added thiosemicabazide (0.18 g, 0.01mole), the mixture was refluxed for 4 hrs .And after cooling the white precipitate was filtered and recretallized from ethanol [7], yield 93%, mp $150-152^{0}$ C.

Synthesis of symmetrical bis[N,N⁻-(2- benzothiazol) -1,2,4 -triazole-3-yl] pyromellit amide [VII]

A stirring solution of compound [VI] (6.64 g, 0.01 mol) and 10 mL 4% aqueous sodium hydroxide solution was refluxed for 4 hrs. After that, the mixture was filtered and the filtered was acidified with dilute hydrochloric acid. The pale browen precipitate was filtered and washed several times with water , yield 58% , mp (100-101) $^{\circ}$ C.

Synthesis of a new series of symmetrical bis $[N,N^--(2-benzothiazole) -5-thio alkyl - 1,2,4 -triazole -3-yl]$ pyromellit amide $[VIII]_{1-5}$

Potassium hydroxide (6.72 g, 0.12mole) dissolved in minimum volume of water, was added dropwise to a stirred solution of triazole (5.74 g, 0.01 mole) in (10mL) of dioxane at 250° C. After heating, the mixture for (15min.) and cooling , n-alkyl halide ((methyl



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iodide,ethyl bromide ,propyl bromide ,butyl bromide or bentyl bromide) (0.01mole) was added dropwise . The solution was refluxed for (3 hrs) . Afterwards the solvent was evaporated on a rotator evaporator, ice-water (100 mL) was added, the resulting precipitate was collected , and recrystallized .The physical properties data of a new series $[VIII]_{1-5}$.are given in table 1.

Result and Discussion

N,N⁻-Bis-(benzothiazol-2-yl) pyromellitamic diacid [I] was synthesized by the reaction of one mole of pyromellitic dianhydride with two moles of 2-amino benzothiazole in acetone as a solvent. The mechanism involves nucleophilic addition reaction, as follows scheme 2.[5]



Scheme 2

The structure of amic acid was studied by it's melting point and FTIR, FTIR spectrum which shows the disappearance of absorption bands of NH_2 group 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 (3284-2450)cm⁻¹, C=O (carboxylic acid) stretching at (1685)cm⁻¹, C=O (amid) stretching at (1654) cm⁻¹ and C=N (endocyclic) stretching at (1627)of hetrocyclic unite .The FTIR data support the proposed structure.

N,N⁻-Bis-(benzothiazol-2-yl) pyromellitamic diacid [I] was converted to ester by the common esterifiction process using dimethyl sulphate in the presence of anhydrous sodium carbonate in dry acetone . N,N⁻-bis-(benzothiazol-2-yl) pyromellitam diacetate **[II]** was charecteized by melting point, C.H.N.S and FTIR sprcetrum . the FTIR spectrum shows absorption band at 1734 cm⁻¹ due to stretching vibration of the (C=O) for ester , also appearance band at 1188 cm⁻¹ was due to (C-O) bending of ester ,besides to disappearance two bands of O-H and C=O of carboxylic moiety . the (C.H.N.S) analysis is consistent with their proposed structure .

The new N,N⁻-bis-(benzothiazol-2-yl) pyromellitam diacetate [II] was used to synthesize new compounds by using two paths as follows :

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Path one:-

The acid hydrazide [III] was synthesized by the reaction of compound [II] with hydrazine hydrate and refluxing in ethanol to form compound [III] which was characterized by melting point, FTIR and ¹HNMR spectram . FTIR spectrum shows absorption bands at (3278-3170) cm⁻¹ due to the asymmetric and symmetric stretching vibration of the (-NH-NH₂) group and stretching vibration band at 1680 cm⁻¹ due to (C=O) for amide .

¹HNMR spectrum (in DMSO as asolvent) of acid hydrazid [III] showed abroad signals at $\delta 2.77$ ppm for two protons of CONH group, many signals at $\delta (7.17-7.66)$ ppm that could be attributed to the ten aromatic protons The spectrum also showed a douplet signal at $\delta 1.2$ ppm for protons of NH₂ group and a triplet signal at $\delta (3.43-3.72)$ ppm due to of NH protons of hydrazide moiety.

The pair of electrons on nitrogen atoms in compound [III] facilitate the reaction of this compound in two ways:

A) acetyl acetone as shown in the following mechanism (scheme 3) to produce the pyrazol derivatives ,which was identified by it's melting point,FTIR, ¹HNMR spectroscopy .FTIR spectrum of pyrazole compound showed the following the disappearance a stretching bands at (1680) cm⁻¹due to C=O amide groups of hydrazide moity. Appearance of new absorption bands of NH at (3419) cm⁻¹ and C=N group endocyclic of pyrazole at (1618) cm⁻¹ was shown.

¹HNMR of pyrazole compound [IV] showed two sharp signals at δ 1.74ppm and δ 1.79ppm due to two CH₃ groups of pyrazole ring [8], a singlet signal at δ 3.15 ppm could be attributed to two protons of NH group and many signals in the region δ (6.76-7.28) ppm for aromatic protons.



B) phthalic anhydride to form the imide as afinal product as shown in (scheme 4) was studied by melting point, FTIR spectrum . The FTIR spectrum exhibited significant two peaks at 1735cm⁻¹ and1720cm⁻¹ which could be attributed to asymmetry and symmetry

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stretching vibration of the carbonyl group (imide cyclic) [9] and two absorption bands around 1186 cm^{-1} and 750 cm^{-1} for asymmetrical and symmetrical of C-N-C (cyclic) .[10-12]



Path two:-

bis [N,N⁻- (2-benzothiazol) thiosemicarbazide] pyromellitamic acid [VI], was obtained by treatment compound [II] with thiosemicarbazide under reflux in ethanol, the structural assignments of [VI] are based on melting points, and their spectral data (FTIR and ¹HNMR spectroscopy). The FTIR absorption-spectrum of this compound [VI] showed (C=O) amid absorption band at 1690 cm⁻¹, the spectrum showed many peaks in the region 3367-3178cm⁻¹ which could be attributed to asymmetric and symmetric stretching vibration of NH and NH₂ groups besides to 1310cm⁻¹ and 2652 cm⁻¹ due to C=S groups and S-H group, respectively.

The ¹HNMR spectrum of this compound showed a good signal at $\delta 3.70$ ppm due to two protons of NH₂ group .Abroad signal at $\delta 4.48$ ppm for two protons of amide group (CO-NH-Ph), two signals doublet and singlet at $\delta 7.45$ -7.58ppm and $\delta 7.98$ ppm for aromatic protons of para substitution benzene rings and central benzene ring, respectively .The spectrum showed two signals due to protons of two NH group of (CO-NH-NH-CS) moiety at $\delta 8.62$ ppm and $\delta 9.80$ ppm

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Scheme 5

The triazole compound [VII] was synthesized by oxidative cyclization of carboxylic thiosemicarbazide derivatives[VI] in the presence of (4%) aqueous NaOH. The mechanism of the reaction may be outlined as follows in scheme (6)



Scheme 6

This compound was characterized by melting point and FTIR ,¹HNMR spectroscopy . The FTIR spectrum showed disappearance of absorption bands due to ν C=O

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(amide) and NH₂ group of compound [VI] together with the appearance of three characteristic stretching bands at 3248cm⁻¹,1645cm⁻¹ and 1205cm⁻¹, which are assigned to ν N-H, ν C=N and ν C=S groups, respectively.

¹HNMR spectrum of triazole compound [VII] showed the following characteristic chmical shefts (in DMSO as a solvent) showed two singlet signals at $\delta 8.63$ ppm and $\delta 8.16$ ppm that could be attributed to protons of two NH groups of triazole (tautomerism stat between thion and thiol forms SH+NH), many signals (triplet, doublet and singlet) in the region $\delta 6.94$ -8.54 ppm due to ten aromatic protons.

The spectrum also showed a good sharp singlet signal SH group and a weak peak at δ 4.05ppm due to protons of amide group (NHCO).

Thio alkyl derivatives [VIII]₁₋₅ of triazole was obtained by the treatment of compound [VII] with selective alkyl halides and potassium hydroxide in dry dioxane as a solvent. These compounds were identified by melting points and FTIR, ¹HNMR.



The FTIR spectra show disappearance bands of S-H ,C=S and NH together with the appearance of new bends at 2966-2854cm⁻¹ due to vC-H aliphatic and 869-855 cm⁻¹ for C-S stretching group [13] the spectral data of new series [VIII]₁₋₅ are listed in table 2.

¹HNMR spectrum of 5-thio methyl triazole compound [VIII]₁ showed the following signals (in DMSO as a solvent) : asinglet signal at δ 3.07 ppm for two protons of NH (amide) group ,a good signal at δ 8.17 ppm for two protons of NH(triazol) group .

The spectrum also showed three doublet signals which could be assigned to the eight aromatic protons of pare substation benzene ring in the region $\delta 6.97$ -7.67 ppm and a singlet signal at $\delta 7.50$ ppm due to two aromatic protons of central benzene ring and a singlet for six aliphatic protons at $\delta 1.29$ ppm of SCH₃ group.

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Table No. (1): The physical properties of compounds [VIII] 1-5									
Comp. No.	Nomenclature	Structural formula	Molecuar formula	M. P^0C	Yie d%	Color			
[VIII]1	bis[N,N'-(2- benzothiazole)- 5-thio methyl - 1,2,4- triazol- 3yl] pyromellit amide	$ \begin{array}{c} $	$C_{27}H_{17}O_2N_{10}S_4$	158-160	67	Pale orange			
[VIII] ₂	bis[N,N'-(2- benzothiazole)- 5-thio ethyl - 1,2,4- triazol- 3yl] pyromellit amide	$\begin{array}{c} 0 \\ N-N \\ S \\ N-N \\ C_2H_5S \\ N \\ O \\ N \\ O \\ N \\ O \\ S \\ N \\ O \\ S \\ O \\ S \\ S \\ S \\ S \\ S \\ S \\ S$	$C_{28}H_{19}O_2N_{10}S_4$	130-132	92	Pale Brown			
[VIII] ₃	bis[N,N'-(2- benzothiazole)- 5-thio propyl- 1,2,4- triazol- 3yl] pyromellit amide	$\begin{array}{c c} & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & &$	C ₂₉ H ₂₁ O ₂ N ₁₀ S ₄	94-95	93	Pale yellow			
[VIII]4	bis[N,N'-(2- benzothiazole)- 5-thio butyl- 1,2,4- triazol- 3yl] pyromellit amide	$\begin{array}{c c} & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ &$	$C_{30}H_{23}O_2N_{10}S_4$	108-109	83	Pale Brown			
[VIII]5	bis[N,N'-(2- benzothiazole)- 5-thio pentyl - 1,2,4- triazol- 3yl] pyromellit amide		$C_{31}H_{25}O_2N_{10}S_4$	118-120	95	Pale Brown			

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Table No.(2): Characteristic FTIR absorption band of compound[VIII]₁₋₅

Comp.	√NH of	√С-Н	√C=O	√C=N	$\sqrt{C=C}$	√C-S
No.	triazole	Aliph.	amid	Of triazole	arom.	
$[VIII]_1$	3248	2954-2854	1645	1636	1598	869
[VIII] ₂	3246	2926-2854	1653	1642	1597	858
[VIII] ₃	3248	2966-2864	1656	1645	1597	856
[VIII] ₄	3248	2962-2856	1658	1645	1597	855
[VIII] ₅	3248	2945-2854	1655	1640	1599	865

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تحضير مركبات حلقية غير متجانسة مشتقة من انهدريد البايروملتيك ودراستها

سحر فاضل عباس عماد تقي علي جمبد هرمز توما قسم الكيمياء / كلية التربية للعلوم الصرفة (ابن الهيثم) / جامعة بغداد

استلم البحث في: 28 آيار 2013 ، قبل البحث في :26 آب 2013

الخلاصة

تم في هذا البحث مفاعلة مولين من (2-امينو بنزوثايزول) مع مول واحد (انهدريد البايروملتيك) للحصول على N,N'-بس- (بنزوثايزول-2-يل) بايرومالتاميك داي اسيد [I]. المقابل من خلال مفاعلة مع داي مثيل سلفيت وبوجود كاربونات الصوديوم واستعمل الاستون مذيباً للحصول على 🛛 بس -N.N' -(بنزوثایزول-2-یل)بایرومالتام دای اسیتیت[[]] استعمل هذا الاستر لانتاج وتحضير مركبات جديدة من خلال عمليتين: 1-العملية الاولى :-تفاعل الاستر مع الهيدر ازين في الايثانول مذيباً للحصول على N,N-بس-(بنزوثایزول- 2-یل)بایرومالتامیك اسید هیدرازاید [III] الذي بدور ه تم مفاعلته مع الاستيل اسيتون في الا يثانول مذيباً او مع انهدريد الفثالك في الدايوكسان للحصول على مركبات حلقية غير متجانسة جديدة (البايروز لات) N,N'-بس-[(2-اميدو بنزوثايزول)5,3 داي ميثيل بايرزول] بايروملتيك [Iv] و الايميدات المقابلة على التوالي N,N'-بس-[(2-امیدو بنزوثایزول) –امیدوفثالماید] بایروملیت [v] 2-العملية الثانية: تفاعل الاستر مع ثايوسيمي كارباز ايد لمحاولة الحصول على بس- [/N N- (2- بنزوثايزول) ثايوسيمي كاربازايد] بايروملتياميك اسيد [vl]

الذي تم تحويله الى الترابيوزول المقابل بس [' N N- (2- بنزوثايزول) 4,2,1-ترايزول -3-يل] بايرومليت اميد [vII] باستعمال مُحيط قاعدي مناسب استعمل المركب الاخير مع هاليدات الكيل متنوعة للحصول على مجموعة من مشتقات الثايو-الكيل ترابيوزول بس ['N N- (2- بنزوثايزول)-5-ثايو الكيل 4,2,1-ترايزول -3-يل]بايرومليت اميد n [vIII] شخصت جميع المركبات المحضرة اعلاه ودرست من خلال ايجاد درجة الانصهار، وتحليل العناصر، واطياف الاشعة تحت الحمراء، وطيف الرنين النووي المغناطيسي للبعض منها، اذ حيث وجد تطابق النتائي النظرية والعمالية للمركبات

لحف الحمراء، وطيف الرئين اللووي المعناطيسي للبعض منها، أد حيث وجد لطابق اللثائج اللطرية والعملية للمرحبات المحضرة الكلمات المفتاحية :- انهدريد البايروملتيك ،البايروزول ،الايميدات ،4,2,1 - ترايزول ،هيدرزايد