Synthesis of New Compounds Bis-(N-Naphthal) Phthalisoimid, Bis-(N-Phenyl) Phthalisoimid and Studying Thermal Stabilty and Bio Activity

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الخلاصة

تحضير مركبات حلقية من الصعب الحصول عليها بالطرق الاعتيادية. تطوير معلوماتنا حول تحضير مركبين جديدين من (بيس فثالاميك) من تفاعل مركبات اروماتية ثنائية الامين مع فثالك انهدريد. إضافة مركبات جديدة من (بيس فثالايسوئيميد) إلى مركبات الايسوئيمايدات ، من خلال إضافة كاشف (DCC) إلى بيس- فثالاميك ودراسة خصائصها الطيفية وتشخيصها طيفياً بـ (I.R.)، أضافتا إلى التحليل الكمي الدقيق للعناصر . ودراسة الثبات الحراري لهذه المركبات وكذلك تمت دراسة التأثير البايولوجي للمركبات المحضرة تجاه بعض البكتريا موجبة لصبغة كرام وسالبة لصبغة كرام وكذلك تجاه بعض الفطريات . وبالإضافة إلى تحضير هذه المركبات فان هذه لطريقةِ تعطينا عِدّة فوائد مثل نسب المنتوج الجيدِ، وطريقة عمل بسيطة في ظروف مناسبة. وذات تأثير جيد على بعض البكتريا والفطريات.

ABSTRACT

Synthesis Cyclic Compounds which are difficult to obtain in normal ways. Developing our in formation concerning the Synthesis of two new Bis-Phthalamic Compounds from reaction between diamines Aromatic Compounds with Phthalic anhydride and add new Compounds to (Compounds Isoimids) by Synthesis two new Bis-Phthalisoimids from adding reagent (DCC) to the Bis- Phthalamic and Studying and diagnosing them by Spectrally (I.R.) in addition to the accurate quantitative elemental analysis. Studying the thermal stability to yield compound and the antimicrobial activity of the synthesized compound was tested against Gram positive and Gram negative bacteria and some fungi. This method provides several advantages such as good yield,

simple work-up procedure and environment friendly and have good effect upon some becteria and fungi.

Introduction

Isoimids Organic compounds are rare, and the difficulty of preparing in one hand, due to lack of fixity other hand [(1) (2)]. And Isoimids Compounds are either open or chain also indicated[(3)]. To Cycloisoimids, and were able to find three different ways to prepare[(4),(5)] Cycloisoimids and longer these methods (important turning point in the history of research on Isoimids) Although the method were not general, as failed to prepare saturated Cycloisoimids.

The following are general reagents in synthesis isoimids

1- Ethyl chloro format – triethyl amine
2- Tri floroacetic anhydride- triethyl amine
3- N,N- dicyclohexyl carbodiimide

The Aryl Phthalides were synthesized by one of two of major methods: acid or base[(19)] catalysed reduction O- aroyl benzoic acid or O-Phthalic acid and substituted Benzen in the Presences of cocnc. H_2SO_4 at Variable temperatures[(19)].

Synthesized Bis-Phthalmic acid from reaction between two moles from Anhydrides Aromatic Compounds with Di-amines Aromatic Compounds and used it to synthesis Bis-Phthalamids Aromatic Compounds by reagent which pull one Molecular weater[(8)].

Experimental

Instrumention:

1. M.P.: Electro thermal Melting Points Apparatus. Univ. Anbar, College of Sci. Dep. of Chem.
2. I.R.: Infrared Spectrophotometer, type (Philips), PU9706, Univ. Anbar, College of Sci. Dep. of Chem.
3. C.H.N.: Element Analysis, Analyzer, type 1106, carlo Eerba Univ. Mousal, College of Sci. Dep. of Chem.

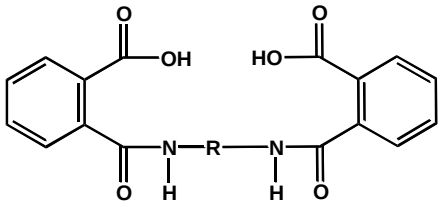
Using methods in the literature [(6),(7),(8)] for the preparation of these Compounds with some changes for Compounds required. The modus operandi can be divided into the following:

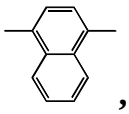
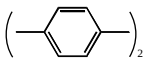
First step: preparation Bis- Phthalamic acid compounds:

Add gradually (9.84 g, 0.0756 mol) of (1,4-Diaminonaphthalene) through the suppression of separation by dissolved in (80 ml of Acetone 95%) and adding (20 g, 0.1493 mol) of the (Phthalic anhydride) dissolved

(150 ml Tetrahedrofuron T.H.F.) in the form of droplets with constant stirring at room temperature. Addendum lasted (one h. and 50 min) to the observing of white deposit. The process of stirring after completion of the addendum for one hour, nominated deposit of (Bis-((N-naphthal) phthalamic acid) washing re-crystallization with acetone the weight (8.2 g) per. (83.3%) and a melting point (195-196 C^0) (1a). Were diagnosed produced by Spectrally (I.R). (See Table (4)) in addition to the accurate quantitative elemental analysis.

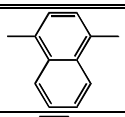
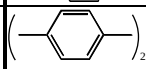
In the same way (Bis ((N-phenyl) fathalamic acid) (1b) is preparing. (See. Table (1), Fig. (1) and (2)).



R=  , 

(1)

Table (1): Characterization data for the synthesized of Bis-Phthalamic compounds

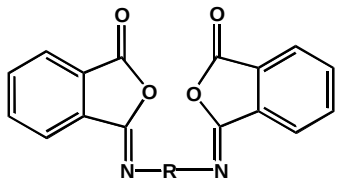
No.	R	formula (M.Wt.)	M.P/C^0	Form yield	Yield %	Analysis Clcd./ (Found)		
						C%	H%	N%
1a		$C_{26}H_{18}O_6N_2$ (454.46)	195-196	Deposit yellow	83.3	68.71 (67.22)	4.00 (3.87)	6.17 (5.99)
1b		$C_{28}H_{20}O_6N_2$ (480.5)	182-183	Deposit yellow	92.0	69.99 (69.33)	4.20 (4.01)	5.83 (4.75)

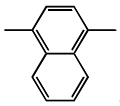
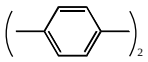
Second step: prepare Bis- Phthalisoimids Compounds

Add (120 ml) of dry methylne chloride (CH_2Cl_2) to (6 g, 0.0132 mol) of the (Bis ((N-nafthal) fathalamic acid) with continuous shaking (using Magnetic stirrer), so it forms foamy solution, then add (5 .4 g, 0.0264 mol) of the detector (N: N-Dicyclohexalcarbon-diimid (DCC))(3) dissolved (60 ml) of methylne chloride (CH_2 Cl_2) through separation funnal linked to interaction flask, and them addendum are gradually with the constant movement and cooling, the addendum lasted an hour and a half, it is observed during the formation of a white deposit, shaking lasted for three hours, after shaking lasted for a period (24 hours) at room

temperature, then left to stagnate deposit, nominated solution of deposit (N: N-Dicyclohxalurea (DCU)). Taking Alerach and vapor by Evaporator spinner at room temperature, then deposit the yellow weight (4.3 g) and by (71.7%) melting point (97-98C^0) (2A), Was diagnosing them Spectrally (I.R.) (Table (5)), in addition to the accurate quantitative elemental analysis.

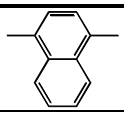
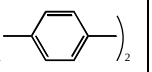
In the same way been preparing the boat Bis- ((N-Phenyl) Phthalisoimid) (2B). (See. Table (2), Fig. (3) and (4)).



R=  , 

(2)

Table (2): Characterization data for the synthesized of Bis- Phthalisoimids compounds

No.	R	formula (M.Wt.)	M.P/C^0	Form yield	Yield %	Analysis Clcd./ (Found)		
						C%	H%	N%
2A		$C_{26}H_{14}O_4N_2$ (418.46)	98-97	Deposit yellow	71.7	74.62 (73.87)	3.38 (3.01)	6.70 (5.65)
2B		$C_{28}H_{16}O_4N_2$ (444.46)	89-88	Deposit yellow	77.2	75.66 (74.54)	3.64 (2.88)	6.30 (6.05)

Thermal Stability to prepare Bis- Phthalisoimids Compounds:

Add (1.5 g, mol) from Bis-((N-phenyl) phthalisoimid) in round flask of double hole and add (15 ml) from chloroform with stirring get yellow solution, $CaCl_2$ anhydrous tube is linked at the of linked reflux and the Nitrogen cylinder is linked with other hole, heated the reaction in water bath upon chloroform reflux (60 C^0) for (3 h.) with continuous stirring under Nitrogen atmosphere. Let yellow solution to cooling in room temperature. Solution evaporation under rarefied pressure and yield yellow deposit, dry it, yield compound M.P. (88-89)C^0 (2A), and diagnosing them by Spectrally (IR.) it compound noticed it symmetrical with Spectrally (IR.) original compound reflux before it. Re method the using Benzene and m-xylene in different times and all re- experiment in

Bis-((N-naphthal) phthalisoimid)(2B) compound prepared. (See Table(3), Fig. (3) and (4)).

Table (3): Characterization data for the Thermal Stability of Bis-Phthalisoimids compounds

No.	Solvent	Sol.C^0	Time ref./ h.	Yield from ref.
A	chloroform	60	3	Bis-((N-naphthal) fathalisoimid)
	chloroform	60	2	Bis-((N-naphthal) fathalisoimid)
	Benzene	78-80	3	Bis-((N-naphthal) fathalisoimid)
	Benzene	78-80	2	Bis-((N-naphthal) fathalisoimid)
	m-xylene	138-140	3	Bis-((N-naphthal) fathalisoimid)
	m-xylene	138-140	2	Bis-((N-naphthal) fathalisoimid)
B	chloroform	60	3	Bis- ((N-phenyl) fathalisoimid)
	chloroform	60	2	Bis- ((N- phenyl) fathalisoimid)
	Benzene	78-80	3	Bis- ((N- phenyl) fathalisoimid)
	Benzene	78-80	2	Bis- ((N- phenyl) fathalisoimid)
	m-xylene	138-140	3	Bis- ((N- phenyl) fathalisoimid)
	m-xylene	138-140	2	Bis- ((N- phenyl) fathalisoimid)

Results and Discussion

Synthesis Aromatic Bis-Phthalisoimid could be two steps,[(10),(11)] as follows:

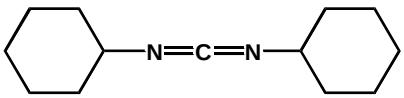
The first step preparing Aromatic Bis-Phathalamic acid:

Got reaction two mol of aromatic anhydrides with one mol of the Compounds aromatic primary diamine to give a aromatic Bis-phathalamic acid interview, and also explained the following formula.

The second step prepare Compounds aromatic Bis- Phthalisoimids:

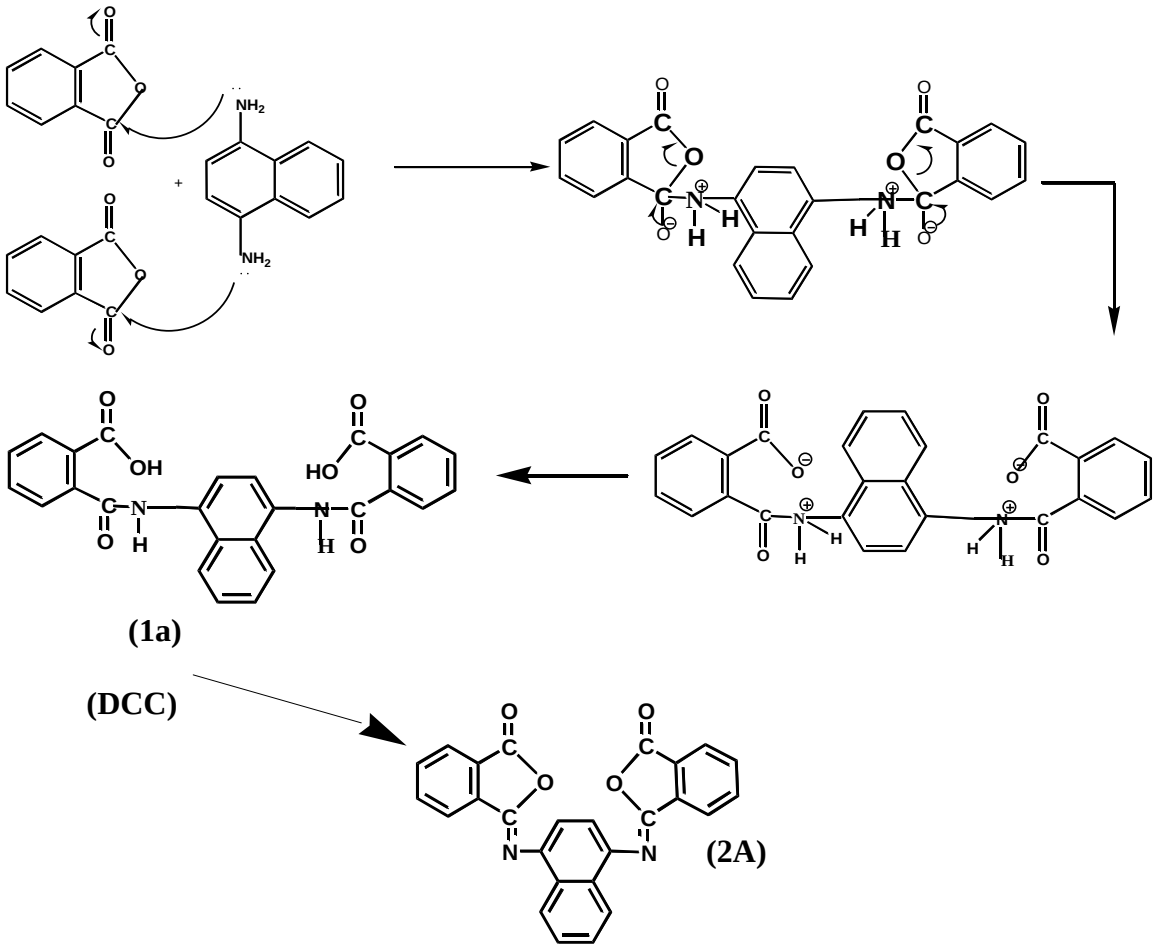
Using acetic aromatic Bis-phthalamic acid Prepared in this research as raw materials in the preparation of Compounds aromatic Bis-(phthalisoimids) or (phthalimids) interview, and that the withdrawal of two molecular water from acid Bis-fathalamic acid (and choose the type of detector user in withdrawing of two molecular water), when used acetic acid anhydride existence sodium acetic anhydride degrees when heat ranging from (85-100 C^0) is obtained Compounds from Bis-phthalisoimids.

As for Compounds Bis-(Phthalisoimids) from Phthalamic acid aromatic interview, are using detector [(4),(5),(12)] (Dicyclohexylcarbodiimide (DCC)) and is one of the reagents powerful withdrawal of water



(DCC)
(3)

also explained the following formula:



Spectrum I.R. Compounds Prepared:

Diagnosed aromatic Bis-Phthalamic acid Prepared and structures Bis-(Phthalisoimids) Prepared using infrared I.R spectrum in (KBr) disk as follows:

For Bis-Phthalamic acid Prepared:

Peak emerged strong absorption at the site (3310-3300 cm^{-1})[13] Vibrated to the frequency of the bond (N-H) and the peak is very strong and broad in site (3110-3350 cm^{-1}) attributed to the frequency of the bond (O-H), a peak of strong intensity at the site (1450-1660 cm^{-1}) Vibrated to the frequency of the bond of stretching imid (C = O) (13),(14) and a group of beam intensity at different locations (1560,1590,1610 cm^{-1}) Vibrated to the frequency stretching bond (C = C) and aromatics set of beams in sites (790,850 cm^{-1}) [11] Vibrated to the frequency of bond angle (C-H) outside the workshop. (See Table (4), Fig. (1) and (2)).

Table (4): IR. Spectra of Bis-Phthalamic acid Prepared

NO.	***N-H*** cm^{-1}	***C=O*** cm^{-1}	O-H cm^{-1}	C-N cm^{-1}	C-O cm^{-1}	C=C aromatic	C-H Out of plane	C=O amide
a	2900	1700	3100- 3225	1300	1210	1560-1610	790	1450
b	2920	1690	3110- 3300	1320	1210	1590-1440	850	1660

As for the Compounds Bis -Phthalisoimids Prepared:

Showed some differences in the type and location bond when compared with Bis-Phthalamic acid as follows:

since disappeared frequency absorption of a two (O-H), (N-H) and Compounds belonging to Bis-fathalamic acid, emerged a powerful new peak distress at the site (1580-1590 cm^{-1}) [13],[15] attributed to the frequency seismic vibration symmetry (C = N). The package of strong intensity in the region (1660,1650 cm^{-1}) (11) and the frequency of sustained analog seismic (C = O). (See Table (5), Fig. (3) and (4)).

Table (5): IR. Spectra of Bis-Phthalisoimids Prepared

NO.	C=O cm^{-1}	C=C aromatic	C-H	C=Ncm^{-1}
A	1660	1440	3250	1590
B	1650	1420	3250	1580

Biological activity

All Bis-Phthalisoimids (2A and 2B) prepared compounds were screened for their antimicrobial activity against [16][17] the Gram-positive bacteria *(1-Staphylococcus aureu, 2-Bacillus subtilis, 3- Bacillus cereus)*, Gram-negative bacteria *(4-Psedomonase aeruginosa, 5- Esherichia Coli)*, as well as fungi: *a) Aspergllus niger, b) penicillium italicum, C) fusarium oxysporum*. Standard antibiotic drug Amoxicillin for bacteria were used at a concentration of (0.1, 1, 10, 20) mg/ml and Mycostatin for fungi were used at a concentration of (1000 ppm) for comparisons. The biological activity for these compound have been evaluated by filter paper disc method [16] after dissolved in N,N-dimethyl formamide to obtain a 1 mg/ml solution (1000 ppm). The inhibition Zones of microbial growth surrounding the filter paper disc (5mm) were measured in millimeters at the end incubation period of 3 days at $37C^0$ for *Esherichia Coli* and $28C^0$ for other bacteria and fungi, N,N-dimethyl formamide alone showed on inhibition zone. The results are illustrated in. (See. Table (6)).

Table (6): Antibacterial activity of the synthesized compounds

Comp.	Con. Mg/ml	Organism*							
		1	2	3	4	5	A	B	C
A	0.1	22	27	16	24	20	14	17	22
	1	28	22	19	18	12	12	15	10
	10	10	25	14	20	9	14	18	20
	20	26	16	22	13	---	16	18	18
B	0.1	17	18	13	9	---	15	16	22
	1	25	20	12	6	11	13	22	16
	10	25	20	10	10	19	13	17	16
	20	20	12	23	11	22	16	12	10
Amoxicillin	1000 ppm	29	20	12	26	10			
Mycostatin							12	19	25

Organism*: 1- *Staphylococcus aureu.* **2-** *Bacillus subtilis.* **3-** *Bacillus cereus.* **4-** *Psedomonase aeruginosa.* , **A**- Aspergllus niger. **B**- penicillium italicum. **C**- fusarium oxysporum.

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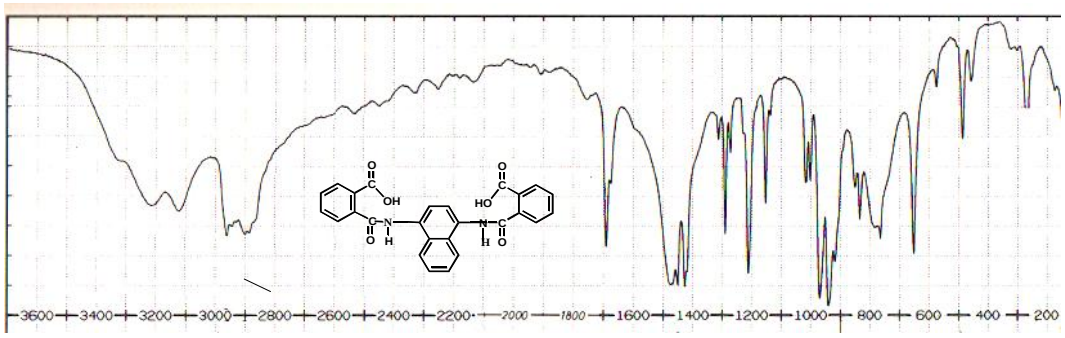

Fig. (1): Spectrum IR. For Comp.(1a).

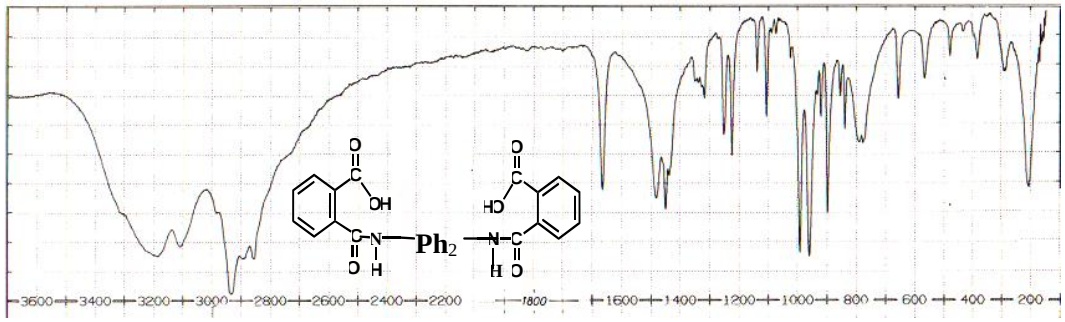

Fig. (2): Spectrum IR. For Comp.(1b).

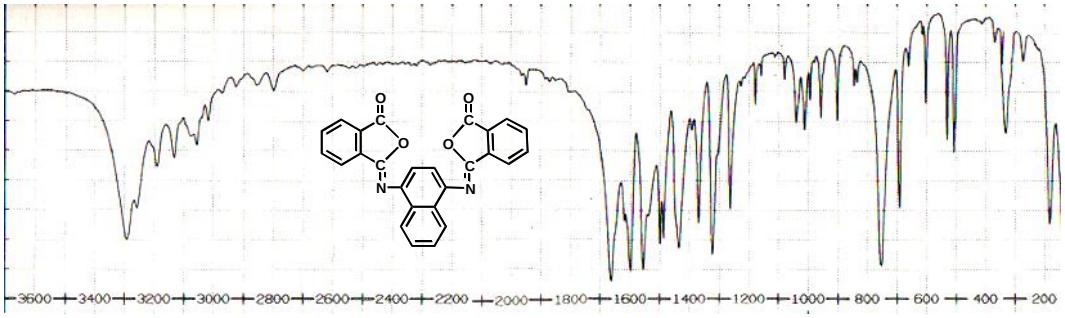

Fig. (3): Spectrum IR. For Comp.(2A).

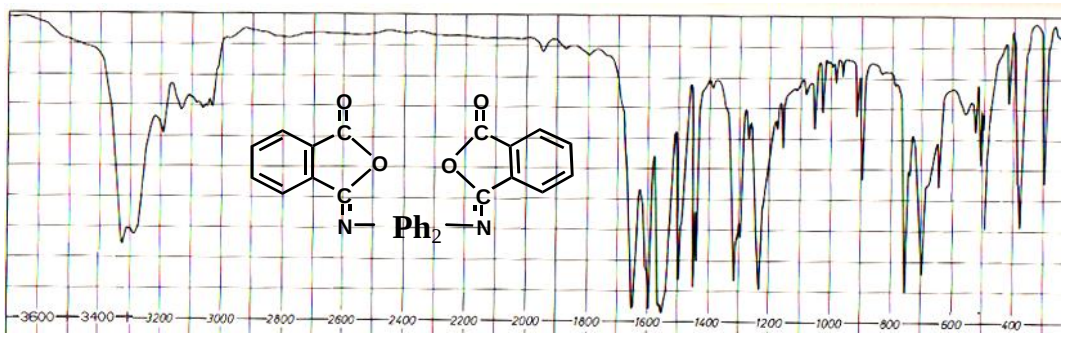

Fig. (4): Spectrum IR. For Comp.(2B).