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Microwave Assisted Reaction of Condensed Thiophenes With Electron Poor Olefins

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요 약. 아미노싸이에노피리다진(**1a, b**)과 아미노싸이에노쿠마린(**2**)은 DMFDMA와 축합반응을 하여 아미딘(**3a, b**)을 형성한다. 이 화합물들을 N-페닐말레이마이드와 반응시키면 화합물 **9**와 **10**이 얻어진다. 반면에 **3a, b, 4, 18, 19, 20**을 말레산 무수물과 반응시키면 포밀 유도체인 **5a, b, 6, 21, 22, 23** 들이 얻어진다. 아미딘 화합물 3a, b 를 다이에틸 퓨마레이트와 반응시키면 가수분해산물인 아미딘 **14**를 거쳐 **11**이 얻어진다. N-페닐말레이마이드를 마이크로웨이브 오븐에서 반응시키면 [2+2]와 [2+2+2] 고리첨가반응 산물이 얻어진다.

주제어: 마이크로웨이브, 싸이오펜, 고리첨가반응

ABSTRACT. Aminothienopyridazines **1a, b** and aminothienocoumarin **2** condensed with DMFDMA to yield amidines **3a, b** and **4**. These compounds reacted with N-phenylmaleimide to yield **9** and **10**. On the other hand reacting **3a, b, 4, 18, 19** and **20** with maleic anhydride afforded only the formylated derivatives **5a, b, 6, 21, 22** and **23** respectively. The reaction of **3a, b** with diethyl fumarate afforded **11**, formed most likely *via* hydrolysis of the amidine **14** during working up the reaction mixture. Irradiation of *N*-phenylmaleimide in microwave oven afforded [2+2] and [2+2+2] cycloaddition product.

Keywords: Microwave, Thiophene, Cycloaddition Reaction

INTRODUCTION

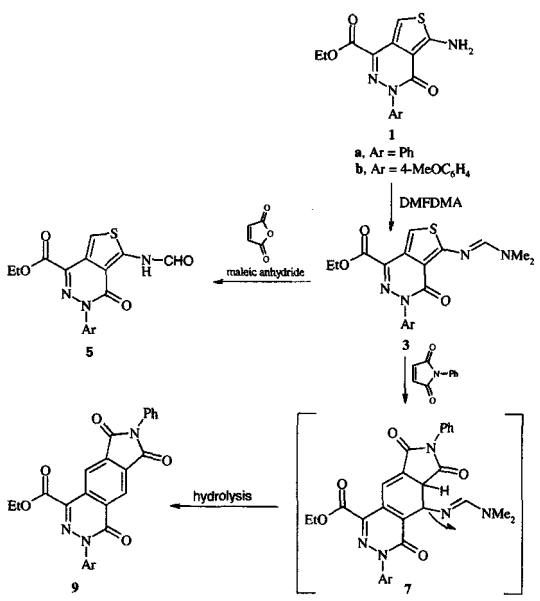
The cycloaddition of condensed aminothiophenes with electron poor olefins has proved to be an efficient route to benzo-fused heteroaromatics.[1-5] It was suggested that presence of free amino groups is essential for reactivity of condensed aminothiophenes as dienes in the Diels-Alder reaction. It occurred to us to see if substituted condensed aminothiophenes could also act as active dienes in the Diels-Alder reaction if drastic reaction condition were used.

RESULTS AND DISCUSSION

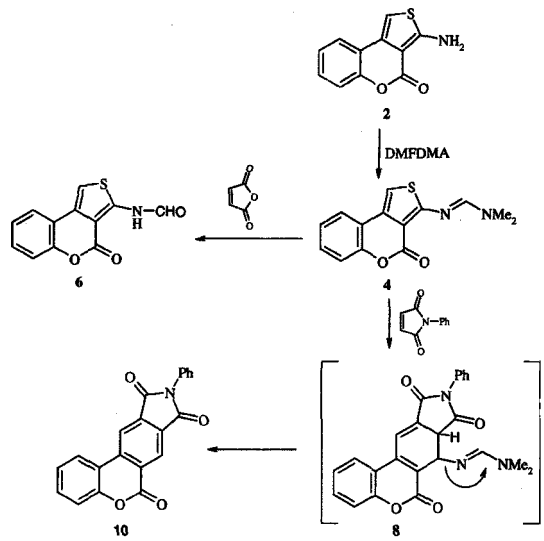
Consequently we condensed amino thienopyridazines **1a, b** and aminothienocoumarine **2** with dimethyl-

formamide dimethylacetal (DMFDMA) and investigated the reactivity of the resulting amidines **3a**, **b** and **4** toward electron-poor olefins. In polar solvents, only hydrolysis of the amidine moiety in **3** and **4** afforded **5** and **6**, respectively. Consequently we investigated the solventless addition of electron-poor olefins under a microwave irradiation, a technology that has been extensively utilized to affect Diels-Alder additions.[6,7] *N*-Phenylmaleimide under this condition reacted smoothly with **3a**,**b** and **4** affording **9** and **10**, which are assumed to be formed *via* intermediacy of cycloadducts **7**, **8** which then loses formamidine to yield the final products **9**, **10** (*Scheme* 1, 2).

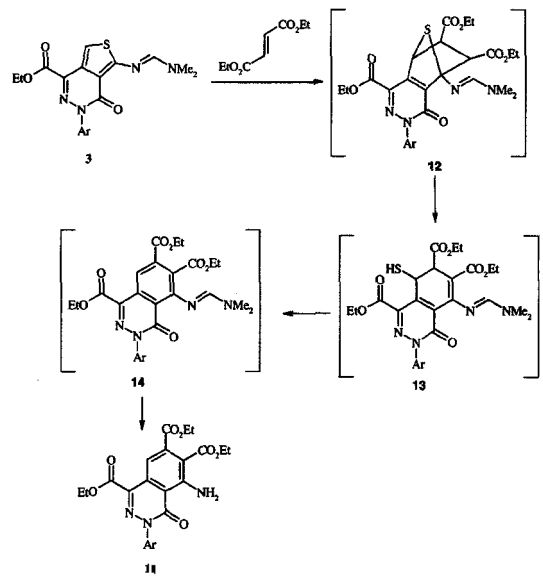
In contrast to this, the reaction of **3** with diethyl fumarate in microwave oven has afforded phthalazine derivatives **11** formed *via* initial formation of cycloadduct when treated with diethyl fumarate **12** then eliminate H_2S to yield the non-isolable intermediate **14** which is then hydrolysed to afford the compound **11**. Compounds **14**, **18**, **19** and **20** were recovered unreacted when treated with diethyl fumarate under the similar condition. Under a variety of conditions, maleic anhydride failed to react with **3a**, **b**, **4**, **18**, **19** and **20**. Only hydrolysis products to the formyl derivatives **5**, **6**, **21**, **22** and **23**



Scheme 1.



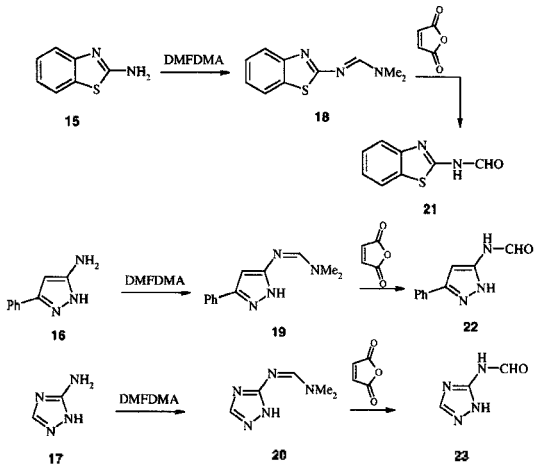
Scheme 2.



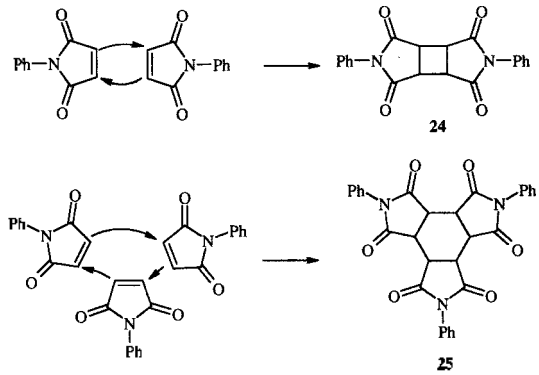
Scheme 3.

were observed (*Scheme* 3, 4). The difference in the behavior of **3** toward maleic anhydride and *N*-phenylmaleimide reflects their different activity as dienes in cycloaddition reactions. Consequently only hydrolysis occurs with maleic anhydride.

Formation of **18**, **19** and **20** has previously been reported *via* condensing **15**, **16** and **17** with dimethylfomamide-dimethylacetal (*DMF DMA*).[8] So treatment of **18**, **19** and **20** with maleic anhydride afforded **21**, **22** and **23** respectively.



Scheme 4.



Scheme 5.

It is of value to report here that products of reacting aminothienocoumarine with *N*-phenylmaleimide were always contaminated with minor quantity of other products of MS=346 and 519. These were the only isolable products from the reaction of **18**, **19** and **20** with *N*-phenylmalimide. The same results were also obtained on irradiating *N*-phenylmaleimide in microwave oven. It is assumed to be the products of, most likely, nonconcerted [2+2] dimerization of two molecules of *N*-phenylmaleimide and the concerted [2+2+2] trimerisation of three molecules of *N*-phenylmalimide and thus assigned structures **24** and **25** respectively. To our knowledge, it is the first reported dimerization and trimerization under *N*-phenylmaleimide in microwave irradiation (*Scheme* 5). It seems that this reaction proceeds much faster than cycloaddition to the diene system in **18**, **19** and **20**. So this is reason why cycloaddition reaction did not proceed with these compounds.

Experimental

All melting points were measured on Gallenkamp electrothermal melting point apparatus and are uncorrected. IR spectra were recorded as KBr pellets on a Pye Unicam SP 3-300 Spectrophotometer. ^{1}H NMR spectra were recorded in deuterated dimethylsulfoxide (*DMSO*-d_6) or deuterated chloroform ($CDCl_3$) on either Varian Gemini (^{1}H NMR at 200 MHz) or Bruker DPX (^{1}H, ^{13}C NMR at 400 MHz) spectrometer using tetramethylsilane (*TMS*) as an internal reference and results are expressed as δ values. Mass spectra were performed on a Shimadzu GCMS-QP 1000 Ex mass spectrometer at 70 eV. Microwave irradiation was carried out using commercial microwave oven and irradiation power of 450 W. Elemental analyses were carried out at the Microanalytical center of Cairo University.

General Procedure for the preparation of 3a, b, 4, 18, 19 and 20

Method A. *N,N*-Dimethylformamide dimethylacetal (0.12 mol) was added to each of (**1a**, **b**, **2**, **15**, **16**, **17**) (0.1 mol) in xylene and the reaction mixture was refluxed for 6 hours. The removal of solvent under reduced pressure yielded the crude product which was crystallized from ethanol.[8]

Method B. Compound (**1a**, **b**, **2**, **15**, **16**, **17**) (0.1 mol) and dimethylformamide dimethylacetal (0.1 mol) was placed in the microwave oven and irradiated at full power for 5-7 mins then left to cool to room temperature, and the solid was collected and crystallized from ethanol.

5-(Dimethylaminomethylenamino)-4-oxo-3-phenyl-3,4-dihydro-thieno[3,4-*d*] pyridazine-1-carboxylic acid ethyl ester 3a

Compound **3a** was obtained as green crystals (method A: 61%, method B: 90%) mp 153 °C. Analysis for $C_{18}H_{13}N_4O_3S$ (370.43): Calcd: C, 58.36; H, 4.90; N, 15.12% Found: C, 58.39; H, 4.91; N, 15.14%. **IR** (ν/cm^{-1}): 1729(CO ester) 1711(ring CO), ^{1}H NMR (δ ppm): 1.38(t, 3H, CH_3CH_2); 3.07(s, 3H, CH_3-N); 3.10(s, 3H, CH_3-N); 4.42(q, 2H, CH_2); 702-7.60(m, 5H, Ar-H); 7.27(s, 1H, thienyl H); 7.89 ppm (s, 1H, amidine H). ms: M$^+$ (370).

5-(Dimethylaminomethylenamino)-3-(4-methoxyphenyl)-4-oxo-3,4-dihydro-thieno[3, 4-*d*]pyridazine-1-carboxylic acid ethyl ester 3b

Compound **3b** was obtained as yellow crystals (method A: 62%, method B: 92%) mp 147 °C. Analysis for $C_{19}H_{20}N_4O_4S$ (400.45): Calcd: C, 56.99; H, 5.03; N, 13.99. Found: C, 56.97; H, 5.11; N, 14.01%. **IR** (ν/cm^{-1}): 1730(CO ester) 1710 cm^{-1}(ring CO), ^{1}H NMR (δ ppm): 1.39(t, 3H, CH_3CH_2); 3.08(s, 3H, CH_3-N); 3.10(s, 3H, CH_3-N); 3.85(s, 3H, OCH_3); 4.42(q, 2H, CH_2); 6.93-7.00(m, 2H, Aryl H-3, 5); 7.28(s, 1H, thienyl H); 7.4-7.5(m, 2H, Aryl H-2, H-6); 7.88 ppm(s, 1H, amidine H). ^{13}C NMR (δ ppm): 14.37 (CH_3CH_2); 34.97 (CH_3-N); 40.84 (CH_3-N); 55.60 (OCH_3); 61.91(OCH_2); 112.29, 114.03, 114.18, 127.84, 128.80, 133.08, 134, 53, 156.51, (aromatic and heterocyclic carbon) 157.97 (amidine CH) and 158.91, 163.45 (2CO). ms: M$^+$ (400).

3-*N,N*-Dimethylaminothieno[3,4:3^1, 4^1]benzo[*b*]pyran-4-one 4

Compound **4** was obtained as green crystals (method A: 72%, method B: 93%) mp 148 °C. Analysis for $C_{14}H_{12}N_2O_2S$ (272.32): Calcd: C, 61.75; H, 4.44; N, 10.29. Found: C, 61.77; H, 4.45; N, 10.30%. **IR** (ν/cm^{-1}): 1690 cm^{-1}(ring CO), ^{1}H NMR (δ ppm): 2.47 (s, 3H, CH_3); 2.51(s, 3H, CH_3); 7.13-7.7 ppm(m, 6H, Ar-H, thienyl H and amidine H). ms: M$^+$ (272).

***N*-Benzothiazol-2-yl-*N,N*-dimethylformamidine 18**

Compound **18** was obtained as colourless crystals (75%) mp 111 °C. Analysis for $C_{10}H_{11}N_3S$(205.28): Calcd: C, 58.51; H, 5.40; N, 20.47. Found: C, 58.57; H, 5.45; N, 20.30%. ^{1}H NMR (δ ppm): 3.02(s, 3H, CH_3-N); 3.04(s, 3H, CH_3-N); 7.13-7.65 (m, 4H, Ar-H); 8.28 ppm (s, 1H, amidine H). ^{13}C NMR (δ ppm): 35.12(CH_3-N); 40.97(CH_3-N); 120.58, 121.23, 122.85, 125.74, 133.33, 152.11(phenyl carbon), 156.54 (thiazole C-2) and 173.61 (amidine). ms: M$^+$ (205).

***N,N*-Dimethyl-*N*-(5-phenyl-*2H*-pyrazol-3-yl) formamidine 19**

Compound **19** was obtained as buff crystals (method A: 78%, method B: 89%) mp 175 °C. Analysis for $C_{12}H_{14}N_4$(214.27): Calcd: C, 67.27; H, 6.59; N, 26.15. Found: C, 67.31; H, 6.60; N, 26.17%. **IR** (ν/cm^{-1}): 3200(NH) and 3074 cm^{-1}(CH), ^{1}H NMR (δ ppm): 2.97(s, 3H, CH_3-N); 3.04(s, 3H, CH_3-N); 6.13 (s, 1H, pyrazol H-4); 7.34-7.78 (m, 5H, Ar-H); 7.86 (s, 1H, amidine CH) and 11.9 ppm (br, 1H, NH). ^{13}C NMR (δ ppm): 34.94 (CH_3-N); 40.66(CH_3-N); 87.83 (pyrazolyl C-4); 125.48, 127.50, 128.60, 133.77 (phenyl carbon), 150.0 (pyrazolyl C-2); 153.0 (pyrazolyl C-5) and 155.27(amidine CH). ms: M$^+$ (214).

***N,N*-Dimethyl-*N*-(*2H*-[1, 2, 4]triazol-3-yl)formamidine 20**

Compound **20** was obtained as colourless crystals (method A: 76%, method B: 85%) mp 97 °C. Analysis for $C_5H_9N_5$ (139.16): Calcd: C, 43.15; H, 6.52; N, 50.33. Found: C, 43.20; H, 6.51; N, 50.36%. **IR** (ν/cm^{-1}): 3325(NH), 2980 cm^{-1}(CH), ^{1}H NMR (δ ppm): 2.96(s, 3H, CH_3-N); 3.04(s, 3H, CH_3-N); 7.96(s, 1H, amidine CH); 8.3 (s, 1H , trizol H-3); and 12.7 ppm (br, 1H, NH). ms: M$^+$ (139).

Reaction of 3a, 3b, 4, 18, 19 and 20 with maleic anhydride

General Procedure.

Method A. A mixture of each of (**3a**, **b**, **4**, **18**, **19**, **20**) (0.1 mol) and maleic anhydride (0.1 mol) was heated at 200 °C for 30-60 mins, left to cool and triturated with ethanol. The solid product, so formed, was collected by filtration and crystallized from ethanol.

Method B. A mixture of each of (**3a**, **b**, **4**, **18**, **19**, **20**) (0.1 mol) and maleic anhydride (0.1 mol) was placed in the microwave oven and irradiated at 450 W for 2-5 mins, then left to cool to room temperature, and the solid was collected and crystallized from ethanol.

5-Formylamino-4-oxo-3-phenyl-3,4-dihydrothieno [3, 4-*d*]pyridazine-1-carboxylic acid ethyl ester 5a

Compound **5a** was obtained as green crystals (method A: 53%, method B, 2 min : 65%) mp 249-250 °C. Analysis for $C_{16}H_{13}N_3O_4S$ (343.36): Calcd: C, 55.97; H, 3.82; N, 12.24. Found: C, 55.99; H, 3.91; N, 12.14%. **IR** (ν/cm^{-1}): 3345(NH), 1731(CO ester) 1690(CO) and 1645 cm^{-1} (ring CO), ^{1}H NMR (δ ppm): 1.43(t, 3H, CH_3); 4.48(q, 2H, CH_2); 7.39-7.60 (m, 5H, Ar-H); 7.96 (s, 1H, thienyl H); 8.55 (s, 1H, CHO); 11.09 ppm (s, 1H, NH). ^{13}C NMR (δ ppm): 14.37 (CH_3CH_2); 62.38 (OCH_2); 112.88, 115.71, 125.38, 126.03, 128.34, 128.99, 133, 96(aromatic

and heterocyclic carbon) and 157.50, 158.95, 162.79 (3CO). ms: M^+ (343).

5-Formylamino-3-(4-methoxyphenyl)-4-oxo-3,4-dihydrothieno[3,4-*d*]pyrida-zine-1-carboxylic acid ethyl ester 5b

Compound **5b** was obtained as yellow crystals (method A: 30%, method B, 2 min: 55%) mp 228-230 °C. Analysis for $C_{17}H_{15}N_3O_5S$ (373.38): Calcd: C, 54.68; H, 4.05; N, 11.25. Found: C, 54.67; H, 4.11; N, 11.28%. **IR** (ν/cm $^{-1}$): 3340(NH), 1731(CO ester) 1690(CO) and 1678 cm^{-1}(ring CO), ^{1}H NMR (δ ppm): 1.39(t, 3H, CH_3CH_2); 3.86(s, 3H, OCH_3); 4.43(q, 2H, CH_2); 7.11(m. 2H, Aryl H-3, 5); 7.49 (m, 2H, Aryl H-2, H-6); 8.02(s. 1H, thienyl H); 8.67(s, 1H, CHO); 11.72ppm (s, 1H, NH). ms: M^+ (373).

3-Formylaminothieno[3,4:3,4]benzo[*b*]pyran-4-one 6

Compound **6** was obtained as pale green crystals (method A: 52%, method B, 3 min: 89%) mp 227-228 °C. Analysis for $C_{12}H_7NO_3S$ (245.25): Calcd: C, 58.77; H, 2.88; N, 5.71. Found: C, 58.79; H, 2.91; N, 5.74%. **IR** (ν/cm^{-1}): 3345(NH), 1693(CO) and 1679 cm^{-1} (ring CO), ^{1}H NMR (δ ppm): 6.95-7.52 (m, 5H, Ar-H); 8.38 (s, 1H, CHO); 11.01 ppm (s, 1H, NH). ms: M^+ (245).

***N*-Benzothizol-2-ylformamide 21**

Compound **21** was obtained as green crystals (method A: 30%, method B, 3 min: 62%) mp 165 °C. Analysis for $C_8H_6N_2OS$ (178.21): Calcd: C, 53.92; H, 3.39; N, 15.72. Found: C, 53.97; H, 3.40; N, 15.80%. ^{1}H NMR (δ ppm): 7.14-7.68(m, 4H, Ar-H); 8.58 (s, 1H, CHO) and 11.12 ppm (s, IH, NH). ms: M^+ (178).

***N*-(5-Phenyl-2*H*-pyrazol-3-yl)formamide 22**

Compound **22** was obtained as beige crystals (method A: 14%, method B, 5 min: 35%) mp 224-226 °C. Analysis for $C_{10}H_9N_3O$ (187.20): Calcd: C, 64.16; H, 4.85; N, 22.45. Found: C, 64.21; H, 4.89; N, 22.47%. ^{1}H NMR (δ ppm): 6.43 (s, 1H, pyrazol H-4); 7.34-7.79 (m, 5H, Ar-H); 8.21 (s, 1H, CHO) 11.01 (1s, 1H, NH) and 12.01 ppm (1s, 1H, NH). ms: M^+ (214).

***N*-(2*H*-[1, 2, 4]Triazol-3-yl)formamide 23**

Compound **23** was obtained as beige crystals (method A: 25%, method B, 5 min: 45%) mp 254 °C. Analysis for $C_4H_6N_4O$(126.12): Calcd.: C, 38.09; H, 4.80; N, 44.42. Found: C, 38.10; H, 4.81; N, 44.46%. ^{1}H NMR (δ ppm): 8.3 (s, 1H , trizol H-3); 9.72(s, 1H, CHO); 10.09 (1s, 1H, NH) and 12.7 ppm (1s, 1H, NH). ms: M^+ (139).

General Procedure for the preparation of 9a, b and 10

Method A. A mixture of each of (**3a**, **b**, **4**) (0.1 mol) and N-phenylmaleimide (0.1 mol) was heated at 200 °C for 1hr, left to cool and triturated with ethanol the solid product, so formed, was collected by filtration and crystallized from ethanol.

Method B. A mixture of each of (**3a**, **b**, **4**) (0.1 mol) and N-phenylmaleimide (0.1 mol) was placed in the microwave oven and irradiated at 450 W for 15-28 mins then left to cool to room temperature, and the solid was collected and crystallized from ethanol.

Compound 9a. was obtained as green crystals (method A: 52%, method B, 15 min: 63%) mp 185 °C. Analysis for $C_{25}H_{17}N_3O_5$ (439.42): Calcd: C, 68.33; H, 3.90; N, 9.56. Found: C, 68.10; H, 4.00; N, 9.650%. **IR** (ν/cm^{-1}): 1728 (CO ester) 1710, 1695 (CO) and 1642 (ring CO), ^{1}H NMR (δ ppm): 1.43 (t, 3H, CH_3), 4.2 (q, 2H, CH_2), 7.16-7.76 (m, 10H, Ar-H); 8.09 (s, 1H, H-9); 9.01 ppm (s, 1H, H-5) ^{13}C NMR (δ ppm): 14.27 (CH_3CH_2); 62.85 (OCH_2); 107.79, 110.12, 116.47, 125.94, 126.46, 128.35, 128.93, 129.12, 129.28, 129.38, 131.34, 134.79, 136.83, 137.99, 140.46, 148.68, 160.87 (aromatic and heterocyclic carbon) and 162.91, 165.96, 168.17, 198.20 (4CO). ms: M^+ (439).

Compound 9b. was obtained as dark yellow crystals (method A: 58%, method B, 28 min: 67%) mp 158 °C. Analysis for $C_{26}H_{20}N_4O_6$ (469.13): Calcd: C, 66.52; H, 4.08; N, 8.95. Found: C, 66.45; H, 4.17; N, 9.82%. **IR** (ν/cm^{-1}): 1738 (CO ester) 1695 (CO) and 1642 (ring CO), ^{1}H NMR (δ ppm): 1.43 (t, 3H, CH_3); 3.67 (s, 3H, OCH_3); 4.2 (q, 2H, CH_2); 7.02-7.52 (m, 9H, Ar-H); 8.08 (s, 1H, H-9); 9.03 ppm (s, 1H, H-5); ms: M^+ (469).

Compound 10. was obtained as oily crystals (method A: 72%, method B, 28 min: 88%) mp 165 °C. Analysis for $C_{21}H_{11}NO_4$ (341.07): Calcd: C, 73.90; H, 3.25; N, 4.10. Found: C, 73.87; H, 3.31;

N, 4.13%. **IR** (ν/cm^{-1}): 1710 (ring CO) 1695 cm^{-1} (amide CO), ^{1}H NMR (δ ppm): 7.2-7.68 (m, 9H, Ar-H), 7.99 (s, 1H, H-7), 8.50 ppm (s, 1H, H-11). ms: M$^+$ (341).

Triethyl 5-amino-3, 4-dihydro-4-oxo-3-phenylphtha lazine-1,6,7-tricarboxylate 11

A mixture of **3a** (0.1 mol) and diethyl fumarate (0.1 mol) was placed in the microwave oven and irradiated at 450 W for 2 min, then left to cool to room temperature, and the solid was collected and crystallized from ethanol.

Compound 11. was obtained as dark red crystals (52%), mp 228 °C. Analysis for $C_{23}H_{23}N_2O_7$ (453.44): Calcd: C, 60.92; H, 5.11; N, 9.27. Found: C, 60.89; H, 5.14; N, 9.29 %. **IR** (ν/cm^{-1}): 3505 and 3320 (NH_2), 1745, 1714, 1710 (ester CO) and 1673 (ring CO), ^{1}H NMR (DMSO-d_6 δ ppm): 1.19-1.44 (m, 9H, $3CH_3$); 3.44 (s, 2H, NH_2); 4.20-4.42 (m, 6H, $3CH_2$); 7.25-7.60 (m, 5H, Ar-H); 8.15 ppm (s, 1H, H-8). ms: (M$^+$+1) (454).

2, 5-Diphenyltetrahydrocyclobuta[1,5-*c*; 3,4-*c'*] dipyrrole-1,3,4,6-tetraone 24 and 2,5,8-Triphenylhexahydro-2,5,8-triazatrindene-1, 3, 4, 6, 7, 9-hexanone 25

Method A. *N*-phenylmaleimide was heated at 200 °C for 1 hr, left to cool and triturated with ethanol the solid product, so formed, was collected by filtration and crystallized from ethanol.

Method B. N-phenylmaleimide (0.1 mol) was placed in the microwave oven and irradiated at 450 W for 5 mins, then left to cool to room temperature, and the solid was collected and crystallized from ethanol.

Compound 24. was obtained as pink crystals (method A: 52%, method B: 60%) mp 252 °C. Analysis for $C_{20}H_{14}N_2O_4$ (346.34): Calcd: C, 69.36; H, 4.07; N, 8.09. Found: C, 69.40; H, 4.09; N, 8.12%. ^{1}H NMR (δ ppm): 3.64-3.66 (m, 4H, H-cyclobutane) and 6.83-7.64 ppm (m, 10H, Ar-H). ^{13}C NMR (δ ppm): 32.4 (cyclobutane carbon); 120.4, 124.1, 128.7, 140.8(aromatic carbon) 175.20 (CO). ms: M$^+$ (346).

Compound 25. was obtained as beige crystals (method A: 50%, method B: 64%) mp 275 °C. Analysis for $C_{30}H_{21}N_3O_6$ (519.50): Calcd: C, 69.36; H, 4.07; N, 8.09. Found: C, 69.39; H, 4.10; N, 8.10%. ^{1}H NMR (δ ppm): 2.88-3.03 (m, 6H, H-cyclohexane) and 6.86-7.67 ppm (m, 15H, Ar-H). ^{13}C NMR (δ ppm): 29.4 (cyclohexane carbon); 120.4, 124.1, 128.7, 140.8 (aromatic carbon) 173.80 (CO). ms: M$^+$ (519).

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