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Synthesis and Biological Evaluation of New Dithiocarbamate Derivatives and New α-aminophosphonate Conjugates Containing 1,3,5-Triazine Nucleus



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Abstract

The reaction of perchlorophenyl carbonochloridodithioate 1 with 1, 2-phenylene diamine 2a or 1, 4-phenylene diamine 2b dissolved in DMF produced the aminophenylamino dithiocarbamate derivatives 3a, 3b. Aminophenylamino dithiocarbamate derivatives 3a, 3b, benzaldehyde 4a, and triphenyl phosphite 5, along with LiClO4 acting as a Lewis acid catalyst, were added to form α -aminophosphonate derivatives 6a, 6b. The interaction of cyanuric chloride 7 with 1, 2-phenylene diamine 2a or 1, 3-phenylene diamine 2c dissolved in acetone produced new 2, 4-diaminophenylaminotriazine derivatives 8a, 8b. In contrast, a one-pot reaction using the produced 2, 4-diaminophenylaminotriazine derivatives 8a, 8b, benzaldehyde 4a or m-chlorobenzaldehyde 4b, and triphenylphosphite 5, along with LiClO4 acting as a Lewis acid catalyst resulted in the synthesis of α -aminophosphonate derivatives 9a, 9b, 10a, 10b. All of the generated compounds' chemical structures were ascertained using the FT-IR, ¹H-NMR, ¹³C-NMR, ³¹P-NMR and mass spectroscopic techniques. Each synthesized compound was tested *in vitro* for its biological effectiveness against gram-positive bacteria, gram-negative bacteria and fungi pathogens. Most synthesized compounds exhibited moderate to good antibacterial activity.

Keywords: α-aminophosphonate, 1,3,5-triazine, triphenylphosphite, Lewis Acid, α-aminophosphonate, antimicrobial activity

1. Introduction

 α -Aminophosphonates compounds are of significant interest to us because of their biological activity [1-6], pharmacological characteristics [7-9], industrial applications [10, 11], metabolic stability, and minimal toxicity to mammals.

α-Aminophosphonates have been shown to have antibacterial and anticancer activity [3, 4]. These compounds are also used as herbicides, antiviral, antifungal, and enzyme inhibitors [6, 7]. In the removal of heavy metals and uranium sorption, it also has industrial and environmental applications in this field. This led to the perception of aaminophosphonates as a viable pharmacophore core in the areas of drug design and discovery [10, 22]. Numerous methods have recently been researched to make the synthesis of α -aminophosphonates more practical. The synthesis methods of αaminophosphonates will be determined by the type of reactants and catalyst.

Due to their widespread biological applicability, dithiocrbamate **II** and 1,3,5-triazine **III** appear to be an essential building motif for the development of novel medications [22–24].



Figure 1: I α -aminophosphonates, II dithiocarbamate, III 1,3,5-Triazine

As phosphonic acid or other analogous groups are replaced for a carboxylic moiety in α aminophosphonate compounds **I**, they are structural mimics of amino acids [26–32]. They perform the

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role of amino acid antagonists by blocking the enzymes involved in amino acid metabolism, which has an effect on the biological operation of the cell. Because of this, the substituted α -aminophosphonate derivatives play a key role as intermediates in the synthesis of large biologically active compounds, which have a variety of applications in the medical field [33, 45].

We would want to talk about how new dithiocarbamate derivatives, 1,3,5-triazine derivatives and their α -aminophosphonate conjugates are made and how they affect gram-positive bacteria, gram-negative bacteria, and fungi pathogens biologically.

2. Result and discussion

According to **Scheme 1**, the aminophenylamino dithiocarbamate derivatives **3** were produced in good yields by reacting perchlorophenyl carbonochloridodithioate **1** with excess amounts of 1,2-phenylene diamine **2a** or 1,4-phenylene diamine **2b** dissolved in dimethyl formamide DMF along with triethyl amine Et₃N acting as a base under refluxing for 6 hours.



Scheme 1: Synthesis of aminophenylamino dithiocarbamate derivatives 3

Scheme 2 provides an illustration of the S_{NAr} amination reaction process for aminophenylamino dithiocarbamate derivatives **3**.



Scheme 2: Suggested mechanism of the synthesis of aminophenylamino dithiocarbamate derivatives

FT-IR, ¹H-NMR, ¹³C-NMR, and mass spectroscopic analyses were used to determine the structure of the aminophenylamino dithiocarbamate derivatives 3. Broad absorption bands at 3262 and 3228 cm⁻¹, which belong to the NH group, were visible in the FT-IR of 3a and 3b. While C=S grouprelated absorption bands could be seen at 1170 and cm^{-1} . 1175 Additionally, absorption bands corresponding to C-S occurred at 684 cm⁻¹. New compounds 3a and 3b ¹HNMR spectra were captured in DMSO-d₆ solvent. The presence of broad at δ : 3.71 and 3.66 ppm verified the presence of NH.

Scheme 3 detailed the synthesis of the α aminophosphonate derivatives 6. As shown in Scheme 3, benzaldehyde 4a, triphenyl phosphite 5, and aminophenylamino dithiocarbamate derivatives 3 were added to methylene chloride in an equivalent molar ratio. Additionally, 10 mmol% of lithium perchlorate was added as a Lewis acid to produce the α -aminophosphonate derivative 6 in a good yield. This reaction required stirring for [24-32] hrs.



Scheme 3: Synthesis of α-aminophosphonate derivatives 6

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By using FT-IR, ¹H-NMR, ¹³C-NMR, ³¹P-NMR, and mass spectroscopic analyses, the structure of the α aminophosphonate derivatives **6** was identified. Broad absorption bands at 3406, 3291, 3348, and 3227 cm⁻¹, which belong to the NH group, were visible in the FT-IR of **6a** and **6b**. While P=O groupcorresponding absorption bands at 1222 and 1253 cm⁻¹ were visible. Additionally, POC-corresponding absorption bands at 814 and 830 cm⁻¹ were seen. New compounds **6a** and **6b** ¹HNMR spectra were captured in DMSO-d6 solvent.

The presence of broad at 6.08, 7.95, 3.82, and 7.95 ppm verified the presence of NH.

The production of the α -aminophosphinates moiety was further supported by the ³¹P-NMR spectra of **6a** and **6b**, which showed a distinct signal at -0.79 and - 3.8 ppm. ppm.

Using 4-di scheme 4, 2. а new aminophenylaminotriazine derivatives 8a. 8b synthesis was shown. The cyanuric chloride 7 as a starting material reacted with excess amounts of 1,2phenylene diamine 2a or 1,3-phenylene diamine 2c dissolving in acetone, along with triethyl amine Et₃N, acting as a base, with stirring at room temperature for produce (24)hours) to new 2,4diaminophenylaminotriazine derivatives 8 in good yields as shown in scheme 4.



Scheme 4: Synthesis 2, 4- diaminophenylaminotriazine derivatives 8

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Scheme 5: Suggested mechanism for the synthesis of 2, 4diaminophenylaminotriazine derivatives 8

FT-IR, ¹H-NMR, ¹³C-NMR, and mass spectroscopic analyses were used to investigate the 2, 4-diaminophenylaminotriazine derivatives **8a** and **8b** structural characteristics. Broad absorption bands at 3233 and 3234 cm⁻¹, which belong to the NH group, were visible in the FT-IR of **8a** and **8b**. While C=N group-related absorption bands could be seen at 1573 and 1554 cm⁻¹. Additionally, C-N-corresponding absorption bands at 1402 and 1398 cm⁻¹ were visible. New compounds **8a** and **8b** ¹HNMR spectra were captured in DMSO-d6 solvent. The existence of broad at: 9.08, 9.19, and 9.99 ppm proved the presence of NH.

Schemes 6, 7 provided instructions for the production of α -aminophosphonate derivatives 9, 10. As shown in schemes 6, 7, benzaldehyde 4a or mchlorobenzaldehyde 4b, triphenyl phosphite 5, and 2,4-di aminophenylaminotriazine derivatives 8 were added in methylene chloride in an equivalent molar ratio. 10 mmol % of lithium perchlorate was then added as a Lewis acid to produce the α aminophosphonate derivatives 9, 10. This reaction required stirring for [24-36] hrs.



Scheme 6: Synthesis of α-aminophosphonate derivatives 9



Scheme 7: Synthesis of α-aminophosphonate derivatives 9

By using FT-IR, ¹H-NMR, ¹³C-NMR, ³¹P-NMR, and mass spectroscopic investigation, the structure of the α -aminophosphonate derivatives **9**, **10** was identified. Broad absorption bands belonging to the NH group were visible in the FT-IR of samples **9a**, **9b**, **10a**, and **10b**.

At the same time, P=O group-corresponding absorption bands at 1209, 1206, 1189, and 1183 cm⁻¹ were visible. In addition, absorption bands

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corresponding to POC at 842, 794, 870, and 873 cm-1 were seen. Newly synthesised compounds **9a**, **9b**, **10a**, and **10b**'s ¹HNMR spectra were captured in DMSO-d₆ solvent. By having broad at δ :9.35, 8.97, 9.3, and 8.42 ppm, NH was proven to be present. Additionally, the aminophosphinates moiety was proven to have formed based on the ³¹P-NMR spectra of samples **9a**, **9b**, **10a**, and **10b**, which showed a distinct signal at 16.12, 15.37, 16.51 and 15.67 ppm.

In scheme 8, the proposed technique for forming the α -aminophosphonate [41] derivatives 6, 9, and 10 is described. First, according to scheme 8, the appropriate imine-intermediates C were produced by the reaction of the aldehyde 4 with the amino-compounds 3, 8, and lithum percholorate as a Lewis acid (LA) catalyst. Next, nucleophilic phosphite 5 attacks the imine intermediate C, which results in the creation of a phosphonium intermediate D. It is most likely that the Lewis acid catalyses this step (LA). After phenol was eliminated, the target α -aminophosphonate derivative 6, 9, and 10 were produced by reaction of phosphonium intermediates D with water, as shown in Scheme 8.



Scheme 8: Reaction mechanism of α -aminophosphonate derivatives 9, 10

3. Antimicrobial Screening

Due to the various biological activities of aminophenylamino dithiocarbamate derivatives, 2, 4di aminophenylaminotriazine derivatives, and their α aminophosphonates, we tested and studied against gram-positive bacteria (Staphylococcus aureus and Bacillus subtilis), gram-negative bacteria (Escherichia coli and Proteus vulgaris), and fungi (Aspergillus fumigatus and Candida albicans). Ketoconazole and gentamycin served as the reference drugs, and DMSO served as the control substance. After a 24-hour incubation period at 37°C, the inhibition zone diameter was measured in mm. Staphylococcus aureus and Bacillus subtilis, which are gram-positive bacteria, were most effectively **Table (1)** combated by compounds **3a**, **6a**, and **10a**, whereas gram-negative bacteria were most effectively combated by compound **3b** (Escherichia coli and Proteus vulgaris). According to the **table** (1), compound **6a** had the best antimicrobial activity against the Aspergillus fumigatus and Candida albicans.

Antimicrobial activity for aminophenylamino dithiocarbamate derivatives (**3a**, **3b**), their α -aminophosphonate derivatives (**6a**, **6b**), 2, 4diaminophenylaminotriazine derivatives (**8a**, **8b**) and their α -aminophosphonate derivatives (**9a**, **9b**, **10a**, **10b**)

Compound	The inhibition zone diameter (mm)					
	Fungi		Gram + Bacteria		Gram -Bacteria	
	Aspergillus	Candida	Staphylococ	Bacillus	Escherichia	Proteus
	fumigatus	albicans	cus aureus	subtilis	coli	vulgaris
3a	0	0	11	12	8	0
3b	10	0	11	8	10	9
6a	10	11	9	13	10	0
6b	9	10	10	14	9	9
8a	0	10	0	10	0	11
8b	0	8	9	13	0	0
9a	0	8	8	12	8	0
9b	0	11	0	11	0	9
10a	0	8	11	12	9	0
10b	0	9	0	13	0	10
Ketoconazole	17	20				
Gentamycin			24	26	30	25
DMSO	0	3	0	5	0	0

4. Conclusion

New aminophenylamino dithiocarbamate derivatives, 2, 4-di aminophenylaminotriazine derivatives, and their α -aminophosphonates were synthesised, characterised by spectroscopic analysis, and tested for their biologiacl properties against gram-positive bacteria, gram-negative bacteria, and fungi. The majority of them had medium to good antibacterial activity.

5. Materials

At the faculty of science at El-Zigzag University in Egypt, all ¹HNMR, ¹³CNMR, and ³¹PNMR spectra

were measured at 400 MHz with $(DMSO-d_6)$ as the solvent. Chemical shifts were expressed in ppm with regard to the solvent's location.

The IR spectra were conducted at the Menoufia University in Egypt's faculty of science. Azhar University in Egypt measured the mass spectra. The screening of *in vitro* antimicrobials was done at Al-Azhar University in Egypt. Stuart Scientific's melting point device was used to measure and record melting points in an uncorrected. On kiesel gel F254 precoated plates, thin layer chromatography (TLC) was used to monitor all synthesised products (Merck). Dimethylformamide (DMF), triethylamine (Et₃N), acetone, methylene chloride (MC), and

lithium perchloride (LiClO₄) were the starting components that were either commercially accessible

or listed in the literature.

6. Experimental

General procedure of aminophenylamino dithiocarbamate derivatives 3a, 3b

Perchlorophenyl carbonochloridodithioate 1 (1 mmol) and 1, 2-phenylene diamine 2a or 1, 4-phenylene diamine 2b (2 mmol) were refluxed for 6 hours in dimethyl formamide DMF along with 10 eqs triethyl amine Et3N acting as a base. To guarantee the completion of the reaction, TLC monitoring was employed. The resulting crude was then put into freezing water, where the solid precipitate that developed was then collected and dried.

Perchlorophenyl (4-aminophenyl) carbamodithioate (3b)

Display the information below m.p = 198-201 °C, dark gray, Yield =77 %, IR (KBr, cm⁻¹): 3329 (NH₂), 3228 (NH), 1175(C=S), 684(C-S). ¹HNMR (CDCl₃, 400MHz): δ ppm = 3.66-3.71(m, 1H, -NH), 6.71 – 6.74(m, 2H, -HAr), 6.97–7.04 (m, 1H, - HAr), 7.26 (s, 1H, - HAr), 8.02 (s, H, -NH₂), ¹³C-NMR (DMSO d6, 100MHz): δ =162.3, 121.1, 114.3, MS (*m*/*z*): 433.7(M, 27 %).

General procedure of α-aminophosphonate derivatives 6a, 6b:

Lithium perchlorate (10 mmol%), benzaldehyde **4a** (1.2 mmol), triphenyl phosphite **5**, and aminophenylamino dithiocarbamate derivatives **3a** and **3b** (1 mmol each) were added to and stirred in 5 mL of methylene chloride (24-32hrs).To guarantee the completion of the reaction, TLC monitoring was employed. The required product was taken by filtering, producing the matching products **6a** and **6b** with a good yield.

Perchlorophenyl

2-

(((diphenoxyphosphoryl)(phenyl)methyl)amino) phenylcarbamodithioate (6a)

Display the information below m.p =163-165 °C, brownish black, Yield =73 %, IR (KBr, cm⁻¹): 3406 (NH), 3291 (NH), 1222(P=O), 1080(C=S), 814(POC), 753 (PCH), 683(C-S). ¹HNMR (DMSO d6, 400MHz): δ ppm =5.92(s, 1H, -CHP), 6.08 (s, 1H, -NH), 6.97 – 7.09(m, 5H, -HAr), 7.14 – 7.27(m, 10H, - HAr), 7.52– 7.59(m, 4H, - HAr), 7.95 (s, 1H, -NH), ¹³C-NMR (100 MHz, DMSO): δ =162.4, 157.4, 129.4, 128.95, 118.85, 115.27, ³¹P -NMR (DMSO, 400MHz) δ : -0.79ppm, MS (*m*/*z*): 755.3(M, 6 %).

Perchlorophenyl carbamodithioate (3a)

(2-aminophenyl)

4-

Display the information below m.p = $178-181 \,^{\circ}$ C, dark brown, Yield =71 %, IR (KBr, cm⁻¹): 3338 (NH₂), 3262 (NH), 1170(C=S), 684(C-S). ¹HNMR (CDCl₃, 400MHz): δ ppm = 3.71-3.73(d , J=7.2Hz, 1H, -NH), 7.123 – 7.258(m, 2H, -HAr), 7.60–7.68(m, 2H, -HAr), 8.02-8.06 (m, 2H, -NH₂), ¹³C-NMR (DMSO d6, 100MHz): δ =129.6, 119.3, 115.3, MS (m/z): 433.8(M, 10 %)

Perchlorophenyl

(((diphenoxyphosphoryl)(phenyl)methyl)amino) phenylcarbamodithioate (6b)

Display the information below m.p =148-151 °C, dark gray, Yield =65 %, IR (KBr, cm⁻¹): 3348 (NH), 3227 (NH), 1253(P=O), 1095(C=S), 830(POC), 730 (PCH), 684(C-S). ¹HNMR (DMSO d6, 400MHz): δ ppm = 1.77-1.79 (m, 2H, -CH₂), 3.82- 3.85 (m, 1H, -NH), 5.95(s, 1H, -CHP), 7.08 – 15(m, 5H, -HAr), 7.21 – 7.21(m, 10H, - HAr), 7.37-7.43(m, 4H, -HAr), 7.95 (s, 1H, -NH), ¹³C-NMR (100 MHz, DMSO): δ =162.3, 147.7, 142.9, 142, 122.1, 121.6, 120.7, 120, 114.6, 113.7, ³¹P -NMR (DMSO, 400MHz) δ : -3.8ppm, MS (*m/z*): 755.9(M, 6 %).

General procedure of 2, 4-

diaminophenylaminotriazine derivatives 8a, 8b:

Cyanuric chloride 7 (1 mmol) was combined with either 1, 2-phenylene diamine 2a or 1, 3-phenylene diamine 2c (3 mmol) in acetone while being stirred at room temperature for 24 hours. To make sure that the reaction was finished, TLC monitoring was employed. The solid precipitate that developed after pouring the resultant crude into freezing water was recovered by filtration and dried.

N1, N1'-(6-chloro-1,3,5-triazine-2,4diyl)bis(benzene-1,2-diamine) (8a)

Display the information below m.p =141-143 °C, white, Yield =75 %, IR (KBr, cm⁻¹): 3304 (NH₂), 3233(NH), 1573(C=N), 1402(C-N). ¹HNMR (DMSO d6, 400MHz): δ ppm = 3.32-3.51 (m, 2H, -NH₂), 4.92 (s, 2H, -NH₂), 6.52 - 6.69(m, 4H, -HAr), 7.16 - 7.24(m, 4H, - HAr), 9.08-9.27(m, 2H, -NH). ¹³C-NMR (DMSO d6, 100MHz): δ =168, 164.3, 163.7, 139.7, 137.9, 129.4, 128.9, 118.8, 118.1, 45.6, MS

(m/z): 328.7(M, 24 %).

N1, N1'-(6-chloro-1,3,5-triazine-2,4diyl)bis(benzene-1,3-diamine) (8b)

Display the information below m.p = 153-156 °C, dark green, Yield =71 %, IR (KBr, cm⁻¹): 3321 (NH₂), 3234(NH), 1554(C=N), 1398(C-N). ¹HNMR (DMSO d6, 400MHz): δ ppm = 3.83-3.91 (m, 4H, -NH₂), 6.58 - 7(m, 3H, -HAr), 7.13 - 7.67(m, 5H, -HAr), 9.19-9.35(m, 1H, -NH), 9.99-10.1(m, 1H, -NH). ¹³C-NMR (DMSO d6, 100MHz): δ =164.8, 126.7, 125.8, 123, 116.4, 115.9, 64.9, 45.4, MS (*m/z*): 328.7(M, 24 %).

General procedure of α-aminophosphonate derivatives 9a, 9b, 10a, 10b:

Lithium perchlorate (10 mmol%), benzaldehyde **4a** or m-chlorobenzaldehyde **4b** (1.2 mmol), 2, 4diaminophenylaminotriazine derivatives **8a**, **8b** and triphenyl phosphite **5** (1 mmol each) were added to and stirred in 5 mL of methylene chloride (24-36hrs).To be sure the reaction had finished, TLC monitoring was employed. To produce the matching products **9a**, **9b**, **10a**, and **10b** in good yield, the desired product was filtered off.

Diphenyl(2-((4-((2-aminophenyl)amino)-6-chloro-1,3,5-triazin-2-

yl)amino)phenylamino)(phenyl)methylphosphonat e (9a)

Display the information below m.p = 112-116 °C, brown, Yield =76 %, IR (KBr, cm⁻¹): 3352 (NH₂), 3275(NH), 1565(C=N), 1408(C-N), 1209(P=O), 842(POC), 690 (PCH). ¹HNMR (DMSO d6, 400MHz): δ ppm =3.65-3.81 (m, 2H, -NH₂), 5.89(s, 1H, -CHP), 6.65 – 6.87 (m, 4H, -HAr), 6.98 – 7.2(m, 9H, - HAr), 7.27 – 7.54 (m, 10H, -HAr), 9.35(s, 3H, -NH). ¹³C-NMR (DMSO d6, 100MHz): 165.9, 165, 157.3, 130.3, 129.8, 129.4, 129.3, 128.9, 128.6, 128.4, 126.9, 126, 120, 118.8, 117, 115.2. ³¹P -NMR (DMSO, 400MHz) δ : 16.12ppm. MS (*m/z*): 650.5(M, 16%).

Diphenyl(2-((4-((2-aminophenyl)amino)-6-chloro-1,3,5-triazin-2-yl)amino)phenylamino)(3chlorophenyl)methylphosphonate (9b)

Display the information below m.p = 187-190 °C, brown, Yield =73 %, IR (KBr, cm⁻¹): 3353 (NH₂), 3261(NH), 1562(C=N), 1402(C-N), 1206(P=O), 794(POC), 684 (PCH). ¹HNMR (DMSO d6, 400MHz): δ ppm =3.61-3.79 (m, 2H, -NH₂), 5.91(s, 1H, -CHP), 6.5(s, 1H, -NH), 6.74 - 6.89 (m, 5H, -HAr), 7.04 - 7.35(m, 9H, - HAr), 7.44 - 7.66 (m, 8H, -HAr), 8.97(s, 1H, -NH), 10(s, 1H, -NH). ¹³C-NMR (DMSO d6, 100MHz):159.1, 157.3, 130.3, 129.4, 126, 118.8, 115.3, 45.5, ³¹P -NMR (DMSO, 400MHz) δ: 15.37ppm. MS (*m*/*z*): 685.3(M, 19%). **Diphenyl(3-((4-((3-aminophenyl)amino)-6-chloro-**

1,3,5-triazin-2-

yl)amino)phenylamino)(phenyl)methylphosphonat e (10a)

Display the information below m.p =195-199 °C, gray, Yield =69 %, IR (KBr, cm⁻¹): 3358 (NH₂), 3261(NH), 1559(C=N), 1405(C-N), 1189(P=O), 870(POC), 684 (PCH). ¹HNMR (DMSO d6, 400MHz): δ ppm =3.85-3.91 (m, 2H, -NH₂), 6.74 – 6.96 (m, 3H, -HAr), 7.08 – 7.31(m, 10H, - HAr), 7.46 – 7.95 (m, 10H, -HAr), 9.3 (s, 1H, -NH), 10.02(s, 1H, -NH), 10.3(s, 1H, -NH). ¹³C-NMR (DMSO d6, 100MHz): 165.15, 164.9, 138.5, 130.9, 129.9, 129.6, 129, 119.9, 119.6, 119. ³¹P -NMR (DMSO, 400MHz) δ : 16.51ppm. MS (*m/z*): 650.2(M, 22%).

Diphenyl(3-((4-((3-aminophenyl)amino)-6-chloro-1,3,5-triazin-2-yl)amino)phenylamino)(3chlorophenyl)methylphosphonate (10b)

Display the information below m.p =139-143 °C, dark gray, Yield =74 %, IR (KBr, cm⁻¹): 3335 (NH₂), 3240(NH), 1553(C=N), 1401(C-N), 1183(P=O), 873(POC), 682 (PCH). ¹HNMR (DMSO d6, 400MHz): δ ppm =3.90-3.93 (m, 2H, -NH₂), 6.79 – 6.93 (m, 4H, -HAr), 7.17 – 7.27(m, 10H, - HAr), 7.76 – 8.15 (m, 8H, -HAr), 8.42 (s, 1H, -NH), 9.19-9.32(m, 1H, -NH), 9.99-10.3(m, 1H, -NH). ¹³C-NMR (DMSO d6, 100MHz): 168.2, 164.7, 164.2, 163.9, 163.7, 162.5, 139.9, 138.3, 137.8, 129.4, 129.1, 119.4, 118.6, 117.8, 115.6, 45.3, ³¹P -NMR (DMSO, 400MHz) δ : 15.67ppm. MS (*m*/*z*): 685(M, 19%).

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