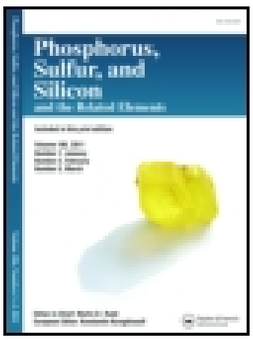

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The Pudovik reaction: the synthesis of bioactive α-aminophosphonates with long alkyl chains

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ABSTRACT

In this research we investigated the reactions of substituted naphthaldehyde with amines. The condensation of do-, tetra-, hexa-, octadecan-1-amines with 2-hydroxy-1-naphthaldehyde yielded a series of azomethines in good yields. Subsequent reaction of these compounds with didodecylphosphine oxide yielded α-aminophosphonates. The in vitro microbiological activity of the synthesized phosphorus compounds against gram-positive, gram-negative bacteria and the yeast Candida albicans was determined in comparison to standard agents. The synthesized compounds showed a high antibacterial and antimycotic activity against human and animal pathogenic microflora. Every newly synthesized compound was characterized by elemental analyses, IR, ^{1}H NMR, ^{31}P NMR spectral studies. The thermal stability was studied by synchronous thermogravimetry and differential scanning calorimetry (TG/DSC).

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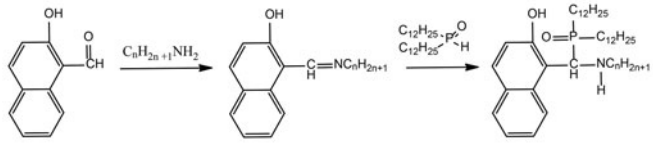
KEYWORDS
α-aminophosphonates; long alkyl chains; antimicrobial activity

GRAPHICAL ABSTRACT



Introduction

In organophosphorus chemistry, the Pudovik reaction is a method for preparing of the various aminophosphonates and phosphorylated imines. The amination of 2-hydroxy-1-naphthaldehyde with long-chain aliphatic amines gave products of condensation (1–5). The phosphorylation of azomethines lead to the formation of α-aminophosphonates (6–10). All new obtained compounds with aliphatic long-chain, due to their unique reactivity, are important in the construction of various multifunctional compounds with a broad spectrum of biological activity.[1–6] The search for novel agents to combat resistant bacteria has become one of the most important areas of antibacterial research today.[7,8] Pharmaceutical and organic chemists are trying to synthesize new drugs with better pharmacokinetic and dynamic properties. We have earlier described the methods of preparation various azomethines and us α-aminophosphonates reported on their structure and reactivity.[9] However, literature survey has indicated that all obtained by us α-aminophosphonates have not been synthesized and their biological activities have not been tested.

Results and discussion

Chemistry

In this paper we present the synthesis and antimicrobial activity of a series of 1-(alkylamino)methyl)naphthalene-2-oles (1–5) on the basis of 2-hydroxy-1-naphthaldehyde and aliphatic alkyl amines and corresponding us α-aminophosphonates bases (6–10) by the reaction of azomethines (1–5) with didodecylphosphine oxide. All the synthesized compounds were characterized by elemental analysis, IR, ^{1}H NMR, and ^{31}P NMR spectroscopy.

We have found that the amination of 2-hydroxy-1-naphthaldehyde with long-chain aliphatic amines ie decan-, dodecan-, tetradecan-, hexadecan and octadecan-1-amines in rations in ethanol at room temperature under vigorous stirring gave products of condensation – azomethines (1–5). Heating in ethanol these compounds with didodecylphosphine oxide leads to the formation of (6–10).

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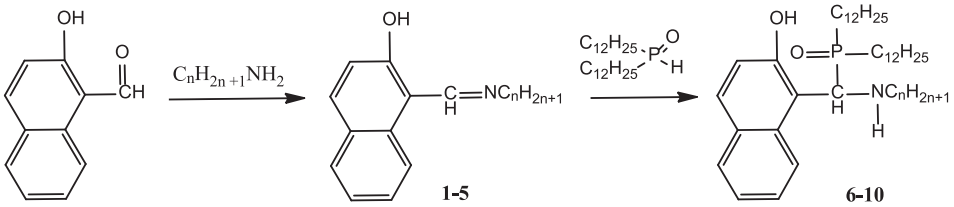
Table 1. Physical data for the azomethines **1–5** and us α-aminophosphonates **6–10**.

Comp. No.	Mw (g/mol)	Yield (%)	Empirical formula	Found (calc) (%)				m.p. (°C)
				C	H	N	P	
1	311	90.7	$C_{21}H_{29}NO$	81.03 (81.37)	9.32 (9.51)	4.50 (4.47)	—	67.7
2	339	85.2	$C_{23}H_{33}NO$	81.42 (81.13)	9.73 (9.28)	4.13 (3.95)	—	74.9
3	367	90.8	$C_{25}H_{37}NO$	81.74 (81.38)	10.08 (10.31)	3.81 (4.17)	—	76.3
4	395	92.4	$C_{27}H_{41}NO$	82.03 (82.21)	10.38 (9.94)	3.54 (3.43)	—	84.7
5	423	89.4	$C_{29}H_{45}NO$	82.27 (82.50)	10.64 (10.78)	3.31 (3.79)	—	89.7
6	697	65.7	$C_{45}H_{80}NO_2P$	77.47 (77.71)	11.48 (11.26)	2.01 (2.11)	4.45 (4.37)	oil
7	725	71.4	$C_{47}H_{84}NO_2P$	77.79 (77.52)	11.59 (11.73)	1.93 (2.17)	4.28 (4.09)	oil
8	753	82.3	$C_{49}H_{88}NO_2P$	78.09 (78.38)	11.69 (11.55)	1.86 (2.12)	4.12 (3.93)	oil
9	781	57.4	$C_{51}H_{92}NO_2P$	78.36 (78.02)	11.78 (12.01)	1.73 (2.06)	3.97 (4.23)	oil
10	809	77.5	$C_{53}H_{96}NO_2P$	78.62 (78.99)	11.87 (11.33)	1.73 (1.57)	3.83 (3.66)	103

Table 2. Antimicrobial activity (growth inhibition zone, mm) of compounds (1–10) (c = 50 μg/0.1 mL).

Compound	*Staphylococcus aureus*	*Escherichia coli*	*Bacillus cereus*	*Pseudomonas aeruginosa*	*Candida albicans*
1	23 ± 0.5	—	17 ± 0.6	13 ± 0.3	21 ± 0.75
2	25 ± 0.3	33 ± 0.5	36 ± 0.7	47 ± 0.5	30 ± 0.5
3	25 ± 0.4	26 ± 0.7	30 ± 0.5	28 ± 0.7	31 ± 0.7
4	25 ± 0.3	18 ± 0.3	26 ± 0.4	20 ± 0.35	29 ± 0.25
5	37 ± 0.2	28 ± 0.15	36 ± 0.3	40 ± 0.8	30 ± 0.5
6	15 ± 0.5	—	11 ± 0.5	15 ± 0.2	9 ± 0.3
7	12 ± 0.4	12 ± 0.2	17 ± 0.25	12 ± 0.3	8 ± 0.2
8	13 ± 0.35	11 ± 0.2	7 ± 0.2	10 ± 0.25	15 ± 0.5
9	14 ± 0.4	—	11 ± 0.5	14 ± 0.3	17 ± 0.3
10	17 ± 0.3	8 ± 0.1	15 ± 0.5	14 ± 0.4	18 ± 0.2
Chlorohexidine	16 ± 0.5	15 ± 0.2	9 ± 0.2	7 ± 0.1	17 ± 0.3
Griseofulvin	0	0	0	0	18 ± 0.2



n = 10 (**1,6**); 12 (**2,7**); 14 (**3,8**); 16 (**4,9**); 18 (**5,10**)

Azomethines and α-aminophosphonates were synthesized in good yield and were fully characterized by physical data in Table 1.

Biological evaluations

The antibacterial and antifungal activity of a series of azomethines (1–5) and us α-aminophosphonates (6–10) were investigated *in vitro* against several pathogenic representative Gram-negative bacteria (*Pseudomonas aeruginoza* ATCC 27853 and Escherichia coli ATCC 25922), Gram-positive bacteria (*Staphylococcus aureus* ATCC 29213 and *Bacillus cereus* 11778), and pathogenic fungi *Candida albicans* ATCC 885-653. The results were summarized in Table 2.

Cup-plate Agar method was used for evaluation of antibacterial activity. The nutrient agar medium is used. The medium with bacteria was poured into sterilized Petri dishes under aseptic conditions. Standard drugs were Chlorhexidine (50 μg/0.1 mL) and test compounds at concentration of 50 μg/0.1 mL. Solvent used was mixture of water and isopropanol at different rations (1:10). Plates were incubated at 37 °C for 24 hours. The antifungal activity was carried out by using cup-plate method using Sabouraud's agar medium. The standard drug used was Griseofulvin (50 μg/0.1 mL) and the test compounds at concentration of 50 μg/0.1 mL by using of the mixture of solvents – ethanol-water at different ratios (1:10). After incubation the average of inhibition was recorded in mm.

All tests were performed in triplicate. Zone of inhibition 22 to 33: highly significant, between 13 to 21 mm: less significant, below 12 mm: poor active.

Conclusions

In conclusion, new azomethines and us α-aminophosphonates were synthesized and their structures were determined by NMR, TG-DSC and elemental analyses. All obtained derivatives with long alkyl aliphatic chains of various lengths were synthesized in good yield, characterized by different spectral studies, and their antimicrobial activity has been evaluated. So, it may be concluded from our results that the synthesized compounds (2–5) are potent antimicrobial agents against pathogenic bacteria and fungi.

Experimental

Chemistry

All chemicals purchased from Sigma-Aldrich were reagent grade and used without purification. IR spectra were taken on a spectrophotometer Spectrum Two PERKIN ELMER in the range 400–3700 cm^{-1}. ^{1}H NMR (D_2O) and ^{31}P NMR (DMSO-d_6) spectra were determined on a Bruker Avance digital spectrometer 400.13 MHz. Chemical shifts were determined with respect to external reference, 85% H_3PO_4. The purity and thermal stability of crustal compounds were determined by simultaneous TG-DSC analysis on a NETZSCH STA 449C instrument (temperature range 40–400 °C, heating rate 10 degrees/min, argon atmosphere).

Materials and methods

General procedure for the synthesis of 1-((alkylamino)methyl)naphthalen-2-ol and its various derivatives 1–5

A mixture of equimolar quantities of alkyl-1-amine (0.01 mol) and appropriate 2-hydroxy-1-naphthaldehyde (0.01 mol) was refluxed in dry ethanol (50 ml) for 1 h. Excess of solvent was removed under reduced pressure. The resulting azomethines were obtained as yellow crystals and purified by diethyl ether from starting reagents.

1-((decylimino)methyl)naphthalen-2-ol 1. Mw.: 311.5. Yield: 82%. M.p.: 75.0 °C. FTIR (ATR, cm^{-1}): 3110 (OH), 2817, 2849 (aromatic C-H), 1643 (C = N). 1H-NMR (400 MHz, $CDCl_3$, δ, ppm): 0.90 (3H, t, CH_3), 1.27 (12H, m, CH_2), 1.48 (2H, p, CH_2), 1.79 (2H, p, CH_2), 3,60 (2H, t, CH_2), 6.97 (1H, d, naph-H), 7.23 (1H, t, naph-H), 7.51 (1H, t, naph-H), 7.63 (1H, d, naph-H), 7.69 (1H, d, naph-H), 7.81 (1H, d, naph-H), 8.65 (1H, d, CH), 14.43 (1H, s, OH).

1-((dodecylimino)methyl)naphthalen-2-ol 2. Mw.: 339. Yield: 85%. M.p.: 79.5 °C. FTIR (ATR, cm^{-1}): 3100 (OH), 2815, 2850 (aromatic C-H), 1647 (C = N). 1H-NMR (400 MHz, $CDCl_3$, δ, ppm): 0.93 (3H, t, CH_3), 1.23 (16H, m, CH_2), 1.49 (2H, p, CH_2), 1.77 (2H, p, CH_2), 3,62 (2H, t, CH_2), 6.90 (1H, d, naph-H), 7.21 (1H, t, naph-H), 7.54 (1H, t, naph-H), 7.60 (1H, d, naph-H), 7.70 (1H, d, naph-H), 7.84 (1H, d, naph-H), 8.63 (1H, d, CH), 14.41 (1H, s, OH).

1-((tetradecylimino)methyl)naphthalen-2-ol 3. Mw.: 367. Yield: 91%. M.p.: 76.3 °C. FTIR (ATR, cm^{-1}): 3087 (OH), 2811, 2849 (aromatic C-H), 1649 (C = N). 1H-NMR (400 MHz, $CDCl_3$, δ, ppm): 0.97 (3H, t, CH_3), 1.28 (20H, m, CH_2), 1.45 (2H, p, CH_2), 1.76 (2H, p, CH_2), 3,66 (2H, t, CH_2), 6.93 (1H, d, naph-H), 7.27 (1H, t, naph-H), 7.49 (1H, t, naph-H), 7.57 (1H, d, naph-H), 7.63 (1H, d, naph-H), 7.87 (1H, d, naph-H), 8.62 (1H, d, CH), 14.57 (1H, s, OH).

1-((hexadecylimino)methyl)naphthalen-2-ol 4. Mw.: 395,62. Yield: 90%. M.p.: 87.2 °C. FTIR (ATR, cm^{-1}): 3091 (OH), 2813, 2855 (aromatic C-H), 1655 (C = N). 1H-NMR (400 MHz, $CDCl_3$, δ, ppm): 0.91 (3H, t, CH_3), 1.25 (24H, m, CH_2), 1.43 (2H, p, CH_2), 1.77 (2H, p, CH_2), 3,62 (2H, t, CH_2), 6.90 (1H, d, naph-H), 7.33 (1H, t, naph-H), 7.44 (1H, t, naph-H), 7.61 (1H, d, naph-H), 7.67 (1H, d, naph-H), 7.92 (1H, d, naph-H), 8.67 (1H, d, CH), 14.39 (1H, s, OH).

1-((octadecylimino)methyl)naphthalen-2-ol 5. Mw.: 423,7. Yield: 89%. M.p.: 86.7 °C. FTIR (ATR, cm^{-1}): 3111 (OH), 2817, 2852 (aromatic C-H), 1661 (C = N). 1H-NMR (400 MHz, $CDCl_3$, δ, ppm): 0.93 (3H, t, CH_3), 1.26 (28H, m, CH_2), 1.45 (2H, p, CH_2), 1.81 (2H, p, CH_2), 3,67 (2H, t, CH_2), 6.91 (1H, d, naph-H), 7.28 (1H, t, naph-H), 7.41 (1H, t, naph-H), 7.69 (1H, d, naph-H), 7.61 (1H, d, naph-H), 7.85 (1H, d, naph-H), 8.68 (1H, d, CH), 14.47 (1H, s, OH).

General procedure for the synthesis of 1-((dialkylphosphoryl)alkylamino)naphthalen-2-ol 6-10

A mixture of equimolar quantities of azomethines **1-5** (0.01 mol) and appropriate didodecylphosphine oxide (0.01 mol) was refluxed in dry ethanol (100 ml) for 25 h. The reaction mixture was left overnight at room temperature, wherein the oil of the product was obtained. Excess of solvent was removed under reduced pressure. The resulting compounds were obtained as yellow oils and purified by ethanol and diethyl ether from starting reagents.

((Decylimino)(2-hydroxynaphthalen-1-y)methyl)didodecylphosphine oxide 6. Mw.: 697. Yield: 62%. Yellow oil. FTIR (ATR, cm^{-1}): 3400-3300 (NH), 1126 (P = O), 776 (P-C). ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 0.87 (9H, m, CH_3), 1.04 (6H, t, CH_2), 1.22 (52H, m, CH_2), 1.63 (2H, s, CH_2), 4,44 (2H, m, CH_2), 7.50 (2H, dd, naph-H) [7.69 (1H, d, naph-H), 8.45 (1H, d, naph-H)], 7.63 (1H, t, naph-H), 7.72 (1H, d, naph-H), 7.81 (1H, d, naph-H), 8.16 (1H, t, naph-H), 8.3 (1H, s, OH), 8.50 (1H, s, CH). ^{31}P-NMR (400 MHz, $CDCl_3$, δ, ppm): 55.36.

((Dodecylimino)(2-hydroxynaphthalen-1-y)methyl)didodecylphosphine oxide 7. Mw.: 725. Yield: 65%. Yellow oil. FTIR (ATR, cm^{-1}): 3400-3300 (NH), 1129 (P = O), 775 (P-C). ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 0.84 (9H, m, CH_3), 1.07 (6H, t, CH_2), 1.28 (56H, m, CH_2), 1.65 (2H, s, CH_2), 4,43 (2H, m, CH_2), 7.53 (2H, dd, naph-H) [7.69 (1H, d, naph-H), 8.45 (1H, d, naph-H)], 7.63 (1H, t, naph-H), 7.72 (1H, d, naph-H), 7.81 (1H, d, naph-H), 8.16 (1H, t, naph-H), 8.3 (1H, s, OH), 8.50 (1H, s, CH).^{31}P-NMR (400 MHz, $CDCl_3$, δ, ppm): 57.13.

((Tetradecylimino)(2-hydroxynaphthalen-1-y)methyl)didodecylphosphine oxide 8. Mw.: 753. Yield: 73%. Yellow oil. FTIR (ATR, cm^{-1}): 3410-3300 (NH), 1130 (P = O), 777 (P-C). ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 0.90 (9H, m, CH_3), 1.10 (6H, t, CH_2), 1.32 (60H, m, CH_2), 1.69 (2H, s, CH_2), 4,41 (2H, m, CH_2), 7.55 (2H, dd, naph-H) [7.69 (1H, d, naph-H), 8.45 (1H, d, naph-H)], 7.63 (1H, t, naph-H), 7.72 (1H, d, naph-H), 7.81 (1H, d, naph-H), 8.16 (1H, t, naph-H), 8.3 (1H, s, OH), 8.50 (1H, s, CH). ^{31}P-NMR (400 MHz, $CDCl_3$, δ, ppm): 53.66.

((Hexadecylimino)(2-hydroxynaphthalen-1-y)methyl)didodecylphosphine oxide 9. Mw.: 781. Yield: 52%. Yellow oil. FTIR (ATR, cm^{-1}): 3400-3300 (NH), 1129 (P = O), 774 (P-C). ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 0.86 (9H, m, CH_3), 1.09 (6H, t, CH_2), 1.29 (64H, m, CH_2), 1.6371 (2H, s, CH_2), 4,40 (2H, m, CH_2), 7.57 (2H, dd, naph-H) [7.69 (1H, d, naph-H), 8.45 (1H, d, naph-H)], 7.63 (1H, t, naph-H), 7.72 (1H, d, naph-H), 7.81 (1H, d, naph-H), 8.16 (1H, t,

naph-H), 8.3 (1H, s, OH), 8.50 (1H, s, CH). ^{31}P-NMR (400 MHz, $CDCl_3$, δ, ppm): 56.90.

((Octadecylimino)(2-hydroxynaphthalen-1-y)methyl)didodecylphosphine oxide 10. Mw.: 809. Yield: 56%. Yellow oil. FTIR (ATR, cm^{-1}): 3400-3300 (NH), 1127 (P = O), 777 (P-C). ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 0.87 (9H, m, CH_3), 1.00 (6H, t, CH_2), 1.27 (68H, m, CH_2), 1.68 (2H, s, CH_2), 4,47 (2H, m, CH_2), 7.60 (2H, dd, naph-H) [7.69 (1H, d, naph-H), 8.45 (1H, d, naph-H)], 7.63 (1H, t, naph-H), 7.72 (1H, d, naph-H), 7.81 (1H, d, naph-H), 8.16 (1H, t, naph-H), 8.3 (1H, s, OH), 8.50 (1H, s, CH). ^{31}P-NMR (400 MHz, $CDCl_3$, δ, ppm): 57.37.

Disclosure statement

No potential conflict of interest was reported by the authors.

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