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SEQUENTIAL THREE-COMPONENT SYNTHESIS OF 1,4-BIS[TRIAZOLO[4,5-*d*]PYRIMIDIN-7(6*H*)-ONE]PIPERAZINES

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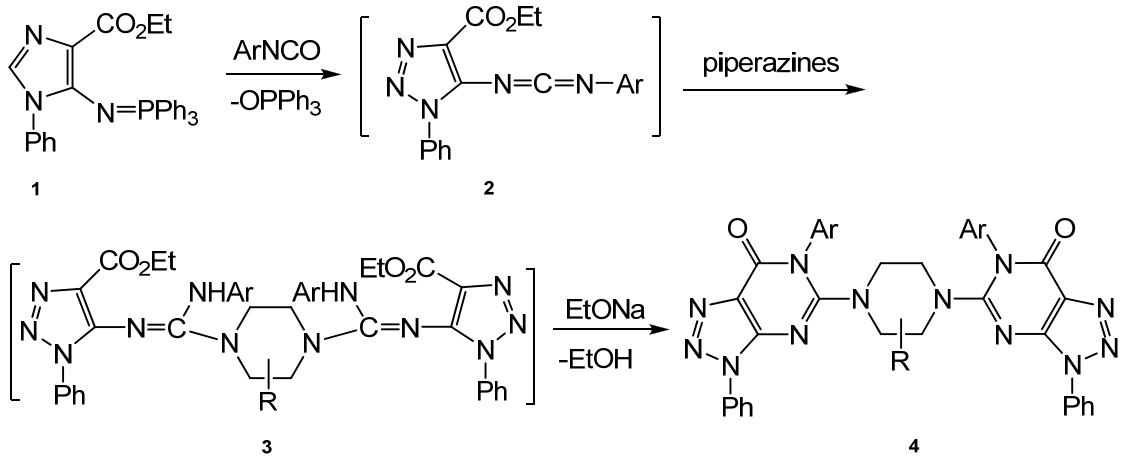
Abstract – A simple three-component reaction and efficient method is described for the synthesis of 1,4-bis[triazolo[4,5-*d*]pyrimidin-7(6*H*)-one]piperazines by a sequential three-component process involving an aza-Wittig reaction/heterocyclization in the presence of sodium ethoxide as catalyst. The iminophosphorane **1** reacted with aromatic isocyanate gave carbodiimides **2**, followed by addition of piperazine derivatives to give the corresponding guanidine intermediates **3**. The guanidine intermediates were cyclized in the presence of catalytic amount of sodium ethoxide to give 1,4-bis[triazolo[4,5-*d*]-pyrimidin-7(6*H*)-one]piperazines **4** in good yields.

In recent years, multicomponent reactions (MCRs) have become important tools in modern preparative synthetic chemistry because these reactions increase efficiency by combining several operational steps without any isolation of intermediates or changes in the conditions.[1-4] This principle, therefore, is highly efficient in terms of time as well as resources. Among the known multicomponent reactions to date, the most valuable reactions are those based on isocyanides. In the development of strategies for the preparation of heterocycles, the aza-Wittig reaction has proved to be a powerful tool for the synthesis of fused nitrogen heterocycles.[5,6] Triazolo[4,5-*d*]pyrimidines are widely recognized as pharmaceutically and biologically useful heterocycles because of their structural similarities to purine bases.[7,8] An important synthetic route for triazolo[4,5-*d*]pyrimidines in previous reports is the nitrosative cyclization of 5,6-diamino-2-phenylpyrimidin-4(3*H*)-ones with nitrous acid.[9] However, this method is characterized as a long reaction time and low yield. Recently, we have been interested in the synthesis of fused pyrimidinones via aza-Wittig reaction of β-ethoxycarbonyliminophosphorane with aromatic isocyanate and subsequent reaction with various nucleophiles under mild conditions. As a continuation of our research for new biologically active heterocycles,[10,11] here we wish to report an efficient synthesis of

1,4-bis[triazolo[4,5-*d*]pyrimidin-7(6*H*)-one]piperazines, a series of compounds which have not been reported before.

The key iminophosphorane **1** (synthesized according to the literature[12]) reacted with aromatic isocyanate to give carbodiimides **2**. The reaction proceeded smoothly in THF at room temperature. Then, reaction of carbodiimides **2** with piperazine derivatives at room temperature gave intermediate guanidines **3** via initial double nucleophilic addition of piperazine derivatives to the carbodiimide. Even in refluxing toluene, **3** did not cyclize. However, in dry ethylene chloride and in the presence of a catalytic amount of EtONa, compounds **3** were converted smoothly to the 1,4-bis[triazolo[4,5-*d*]pyrimidin-7(6*H*)-one]piperazines **4** in satisfactory yields at room temperature (Scheme 1). We found that heterocyclization occurred via nucleophilic displacement of the neighboring ester ethoxide group to provide the target compounds **4** by intramolecular hetero conjugate addition annulations. Irrespective of the fact whether substituted piperazines or unsubstantiated piperazine, the cyclization proceeded very smoothly in the same regioselectivity. No matter the substituents on the benzene are electron-withdrawing or electron-donating groups, the cyclization can be completed easily under mild conditions.



Scheme 1

The results are listed in Table 1.

Table 1. Preparation of 1,4-Bis[triazolo[4,5-*d*]pyrimidin-7(6*H*)-one]piperazines

Compd.	*Ar*	*R*	*Yield */%*
4a	Ph	H	87
4b	Ph	2-Me	85
4c	Ph	2,5-diMe	79
4d	Ph	2,6-diMe	74
4e	Ph	2-Et	83

4f	4-Me-C_6H_4	H	82
4g	4-Me-C_6H_4	2-Me	80
4h	4-Me-C_6H_4	2,5-diMe	74
4i	4-Me-C_6H_4	2,6-diMe	75
4j	4-Me-C_6H_4	2-Et	78
4k	4-Cl-C_6H_4	H	93
4l	4-Cl-C_6H_4	2-Me	87
4m	4-Cl-C_6H_4	2,5-diMe	82
4n	4-Cl-C_6H_4	2,6-diMe	78
4o	4-Cl-C_6H_4	2-Et	81

*isolated yields based on iminophosphorane **1**

In conclusion, we have demonstrated that 1,4-bis[triazolo[4,5-*d*]pyrimidin-7(6*H*)-one]piperazines can be produced via aza-Wittig reaction/heterocyclization in the presence of sodium ethoxide as catalyst. As the synthetic method reported in this paper starts with readily available compounds and is operationally simple, it may find some value in organic synthesis.

EXPERIMENTAL

Melting points were determined on an *X-4* model digital micro-melting point hot stage apparatus and were uncorrected. IR spectra were recorded on a *Nicolet 7500 NXR* infrared spectrometer as KBr pellets with absorption values given in wave numbers (ν, cm^{-1}). ^1H NMR spectra were measured in $CDCl_3$ on a *Varian Mercury Plus 300* (300Hz) spectrometer and chemical shift values were expressed in δ values (ppm) relative to Me_4Si (TMS) as an internal reference ($\delta = 0$). ^{13}C NMR spectra were determined on the same *Varian Mercury Plus 300* spectrometer at 75 MHz. Mass spectral (MS) data were obtained on a *Finnigan LCQ Advantage MAX* mass spectrometer. Elementary analyses were taken on a *Perkin-Elmer CHN 2400* elemental analysis instrument. Progress of the reactions was monitored by analytical thin-layer chromatography (TLC) on glass sheets precoated with UV fluorescent silica gel.

General preparation of 1,4-Bis[triazolo[4,5-*d*]pyrimidin-7(6*H*)-one]piperazines 4

Aromatic isocyanate was added (0.006 mol) to the solution of iminophosphorane **1** (0.006 mol) in THF (10 mL) at 0-5 °C. When the resulting iminophosphorane **1** reacted with aromatic isocyanate, triphenylphosphine oxide was formed. After the reaction mixture was left unstirred for 5-6 h at 0-5 °C, the solvent was removed off under reduced pressure and Et_2O/petroleum ether (1:2, 12 mL) was added to

precipitate triphenylphosphine oxide. Removal of the solvent gave carbodiimides **2**, which were used directly without further purification. Piperazine derivatives (0.003 mol) were added to the solution of **3** prepared above in CH_2Cl_2 (10 mL). After the reaction mixture was left unstirred for 2-3 h, the solvent was removed and EtONa in anhydrous EtOH (8 mL, 10%) was added. The mixture was stirred for 3-5 h at room temperature. The solution was condensed and the residue was recrystallized from EtOH to give the target compound **4**.

1,4-Bis[4-oxo-1,5-diphenyl-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]piperazine (4a): white crystals; mp 258-259 °C; IR 3341, 2953, 1718 (C=O), 1527, 1423, 1368 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.62 (t, *J* = 7.0 Hz, 8H), 6.87-7.54 (m, 20H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 44.58, 118.05, 124.40, 127.91, 128.50, 128.87, 129.06, 129.45, 129.87, 133.51, 149.38, 155.64, 161.38; ESI-MS m/z 661.25 (M^+ + H). Anal. Calcd for $C_{36}H_{28}N_{12}O_2$: C, 65.44; H, 4.27; N, 25.44. Found: C, 65.29; H, 4.18; N, 25.53.

1,4-Bis[4-oxo-1,5-diphenyl-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2-methylpiperazine (4b): white crystals; mp 273-274 °C; IR 3340, 2932, 1709 (C=O), 1521, 1436, 1359 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.15 (d, *J* = 6.8 Hz, 3 H), 2.46 (d, *J* = 7.0 Hz, 2H), 2.56 (t, *J* = 7.2 Hz, 4H), 2.98 (m, 1 H), 6.86-7.45 (m, 20 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.32, 42.84, 44.15, 45.55, 46.62, 118.05, 124.40, 127.91, 128.50, 128.87, 129.06, 129.45, 130.96, 133.51, 149.38, 155.64, 161.38; ESI-MS *m/z* 675.36 (M^+ + H). Anal. Calcd for $C_{37}H_{30}N_{12}O_2$: C, 65.86; H, 4.48; N, 24.91. Found: C, 65.95; H, 4.39; N, 25.01.

1,4-Bis[4-oxo-1,5-diphenyl-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2,5-dimethypiperazine (4c): white crystals; mp 281-282 °C; IR 3341, 2942, 1717 (C=O), 1548, 1443, 1371 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 1.18 (d, *J* = 7.0 Hz, 6 H), 2.55 (d, *J* = 7.2 Hz, 4H), 3.01 (t, *J* = 6.2 Hz, 2 H), 6.76-7.35 (m, 20 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.32, 41.58, 46.96, 118.05, 124.40, 127.91, 128.50, 128.87, 129.06, 129.42, 130.32, 132.34, 149.45, 155.65, 161.37; ESI-MS *m/z* 689.46 (M^+ + H). Anal. Calcd for $C_{38}H_{32}N_{12}O_2$: C, 66.27; H, 4.68; N, 24.40. Found: C, 66.21; H, 4.73; N, 24.34.

1,4-Bis[4-oxo-1,5-diphenyl-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2,6-dimethylpiperazine (4d): white crystals; mp 268-269 °C; IR 3312, 2924, 1718 (C=O), 1524, 1436, 1340 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.17 (d, *J* = 7.0 Hz, 6H), 2.54 (d, *J* = 7.2 Hz, 4H), 3.02 (t, *J* = 6.8 Hz, 2 H), 6.76-7.41 (m, 20 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.60, 44.08, 44.46, 118.21, 124.40, 127.91, 128.50, 128.87, 129.06, 129.42, 130.21, 132.62, 149.33, 155.62, 161.64; ESI-MS *m/z* 689.43 (M^+ + H). Anal. Calcd for $C_{38}H_{32}N_{12}O_2$: C, 66.27; H, 4.68; N, 24.40. Found: C, 66.23; H, 4.77; N, 24.37.

1,4-Bis[4-oxo-1,5-diphenyl-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2-ethylpiperazine (4e): white crystals; mp＞300 °C; IR 3318, 2936, 1715 (C=O), 1548, 1450, 1342 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.01 (t, *J* = 7.2 Hz, 3H), 1.37 (m, 2H), 2.54 (d, *J* = 7.0 Hz, 2 H), 2.57 (t, *J* = 6.8 Hz, 4H), 6.54-7.33 (m, 20H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 9.06, 22.97, 41.62, 43.09, 45.56, 53.56, 118.05, 124.40, 127.91,

128.50, 128.87, 129.04, 129.23, 139.97, 132.33, 149.63, 155.65, 161.32; ESI-MS *m/z* 689.45 (M^+ + H). Anal. Calcd for $C_{38}H_{32}N_{12}O_2$: C, 66.27; H, 4.68; N, 24.40. Found: C, 66.28; H, 4.75; N, 24.36.

1,4-Bis[4-oxo-1-phenyl-5-(4-methylphenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]piperazine (4f): white crystals; mp 276-277 °C; IR 3321, 2940, 1708 (C=O), 1551, 1436, 1335 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.35 (s, 6H), 2.55 (t, *J* = 7.0 Hz, 8H), 6.62-7.25 (m, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.56, 45.33, 121.12, 127.91, 128.50, 128.87, 129.06, 129.34, 129.63, 129.91, 132.94, 146.32, 158.36, 164.42; ESI-MS *m/z* 689.43 (M^+ + H). Anal. Calcd for $C_{38}H_{32}N_{12}O_2$: C, 66.27; H, 4.68; N, 24.40. Found: C, 66.19; H, 4.55; N, 24.50.

1,4-Bis[4-oxo-1-phenyl-5-(4-methylphenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2-methylpiperazine (4g): white crystals; mp 261-262 °C; IR 3332, 2943, 1716 (C=O), 1553, 1435, 1354 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.15 (d, *J* = 6.8 Hz, 3 H), 2.35 (s, 6H), 2.42 (d, *J* = 7.2 Hz, 2H), 2.51 (t, *J* = 7.2 Hz, 4H), 2.98 (m, 1 H), 6.66-7.36 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.32, 24.56, 42.84, 44.15, 45.55, 46.62, 121.01, 127.91, 128.50, 128.86, 129.06, 129.21, 129.47, 130.96, 133.57, 149.35, 155.63, 161.38; ESI-MS *m/z* 703.36 (M^+ + H). Anal. Calcd for $C_{39}H_{34}N_{12}O_2$: C, 66.65; H, 4.88; N, 23.92. Found: C, 66.57; H, 4.71; N, 24.06.

1,4-Bis[4-oxo-1-phenyl-5-(4-methylphenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2,5-dimethylpiperazine (4h): white crystals; mp 287-288 °C; IR 3342, 2963, 1719 (C=O), 1552, 1452, 1336 cm^{-1}; ^1H NMR (300 MHz , $CDCl_3$) δ 1.13 (d, *J* = 7.0 Hz, 6 H), 2.35 (s, 6H), 2.55 (d, *J* = 7.2 Hz, 4 H), 3.02 (t, *J* = 6.8 Hz, 2 H), 6.49-7.37 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.32, 24.56, 42.51, 46.95, 118.07, 127.91, 128.50, 128.87, 129.06, 129.12, 129.42, 130.01, 134.57, 149.45, 155.66, 161.35; ESI-MS *m/z* 717.51 (M^+ + H). Anal. Calcd for $C_{40}H_{36}N_{12}O_2$: C, 67.02; H, 5.06; N, 23.45. Found: C, 66.93; H, 4.97; N, 23.36.

1,4-Bis[4-oxo-1-phenyl-5-(4-methylphenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2,6-dimethylpiperazine (4i): white crystals; mp＞300 °C; IR 3332, 2919, 1717 (C=O), 1548, 1466, 1323 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.15 (d, *J* = 7.0 Hz, 6H), 2.35 (s, 6H), 2.51 (d, *J* = 7.2 Hz, 4H), 3.04 (t, *J* = 6.8 Hz, 2 H), 6.61-7.34 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.60, 24.56, 44.08, 44.46, 118.21, 127.91, 128.50, 128.87, 129.06, 129.12, 129.45, 130.20, 133.62, 149.33, 155.62, 161.64; ESI-MS *m/z* 717.48 (M^+ + H). Anal. Calcd for $C_{40}H_{36}N_{12}O_2$: C, 67.02; H, 5.06; N, 23.45. Found: C, 66.95; H, 4.97; N, 23.33.

1,4-Bis[4-oxo-1-phenyl-5-(4-methylphenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2-ethylpiperazine (4j): white crystals; mp＞300 °C; IR 3332, 2934, 1715 (C=O), 1563, 1442, 1338 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.02 (t, *J* = 6.8 Hz, 3H), 1.37 (m, 2H), 2.35 (s, 6H), 2.53 (d, *J* = 7.2 Hz, 2H), 2.56 (t, *J* = 6.8 Hz, 4H), 6.54-7.33 (m, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 9.06, 22.97, 24.56, 41.62, 43.09, 45.56, 53.56, 119.35, 127.91, 128.50, 128.87, 129.06, 129.23, 129.49, 139.97, 133.87, 149.62,

155.61, 161.37; ESI-MS *m/z* 717.39 (M^+ + H). Anal. Calcd for $C_{40}H_{36}N_{12}O_2$: C, 67.02; H, 5.06; N, 23.45. Found: C, 66.97; H, 4.98; N, 23.39.

1,4-Bis[4-oxo-1-phenyl-5-(4-chlorophenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]piperazine (4k): white crystals; mp＞300 °C; IR 3322, 2912, 1718 (C=O), 1541, 1462, 1328 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.65 (t, *J* = 7.0 Hz, 8H), 6.62-7.25 (m, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 45.33, 123.15, 127.91, 128.44, 128.85, 129.03, 129.24, 129.81, 130.47, 131.34, 151.64, 157.37, 165.47; ESI-MS *m/z* 729.51 (M^++H). Anal. Calcd for $C_{36}H_{26}N_{12}O_2Cl_2$: C, 59.27; H, 3.59; N, 23.04. Found: C, 59.34; H, 3.53; N, 23.11.

1,4-Bis[4-oxo-1-phenyl-5-(4-chlorophenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2-methyl-piperazine (4l): white crystals; mp＞300 °C; IR 3318, 2931, 1716 (C=O), 1523, 1454, 1365 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.11 (d, *J* = 6.8 Hz, 3H), 2.54 (d, *J* = 7.0 Hz, 2H), 2.51 (t, *J* = 7.2 Hz, 4H), 2.98 (m, 1 H) , 6.43-7.25 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.32, 42.84, 44.15, 45.55, 46.62, 123.05, 127.93, 128.50, 128.88, 129.06, 129.25, 129.85, 130.46, 131.51, 151.38, 157.64, 165.32; ESI-MS *m/z* 743.26 (M^++H). Anal. Calcd for $C_{37}H_{28}N_{12}O_2Cl_2$: C, 59.76; H, 3.80; N, 22.60. Found: C, 59.85; H, 3.77; N, 22.52.

1,4-Bis[4-oxo-1-phenyl-5-(4-chlorophenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2,5-dimethyl-piperazine (4m): white crystals; mp＞300 °C; IR 3312, 2923, 1719 (C=O), 1525, 1449, 1361 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.11 (d, *J* = 7.0 Hz, 6 H), 2.54 (d, *J* = 7.2 Hz, 4H), 3.02 (t, *J* = 6.2 Hz, 2 H), 6.41-7.28 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.32, 41.58, 46.96, 118.05, 127.91, 128.50, 128.87, 129.06, 129.25, 129.88, 130.47, 131.46, 149.38, 155.64, 161.38; ESI-MS *m/z* 757.29 (M^++H). Anal. Calcd for $C_{38}H_{30}N_{12}O_2Cl_2$: C, 60.24; H, 3.99; N, 22.19. Found: C, 60.31; H, 3.92; N, 22.12.

1,4-Bis[4-oxo-1-phenyl-5-(4-chlorophenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2,6-dimethyl-piperazine (4n): white crystals; mp＞300 °C; IR 3331, 2917, 1717 (C=O), 1547, 1561, 1383 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.12 (d, *J* = 7.0 Hz, 6H), 2.52 (d, *J* = 7.2 Hz, 4H), 3.01 (t, *J* = 6.8 Hz, 2 H), 6.46-7.27 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.60, 44.08, 44.46, 118.05, 127.91, 128.50, 128.87, 129.06, 129.45, 129.95, 130.11, 131.51, 149.34, 155.61, 161.32; ESI-MS *m/z* 757.32 (M^++H). Anal. Calcd for $C_{38}H_{30}N_{12}O_2Cl_2$: C, 60.24; H, 3.99; N, 22.19. Found: C, 60.27; H, 3.94; N, 22.10.

1,4-Bis[4-oxo-1-phenyl-5-(4-chlorophenyl)-1,2,3-triazolo[4,5-*d*]pyrimidin-2(3*H*)-yl]-2-ethyl-piperazine (4o): white crystals; mp＞300 °C; IR 3332, 2941, 1715 (C=O), 1533, 1447, 1341 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.0 Hz, 3 H), 1.38 (m, 2 H), 2.53 (d, *J* = 7.2 Hz, 2 H), 2.56 (t, *J* = 6.8 Hz, 4H), 6.54-7.36 (m, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 9.06, 22.97, 41.62, 43.09, 45.56, 53.56, 118.05, 127.91, 128.50, 128.87, 129.06, 129.45, 130.07, 130.96, 131.51, 149.37, 155.64, 161.47; ESI-MS

m/z 757.34 (M^++H). Anal. Calcd for $C_{38}H_{30}N_{12}O_2Cl_2$: C, 60.24; H, 3.99; N, 22.19. Found: C, 60.27; H, 3.94; N, 22.15.

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