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Practical Synthesis of 1,8-Naphthyridine-2,7-dialdehydes Syntone

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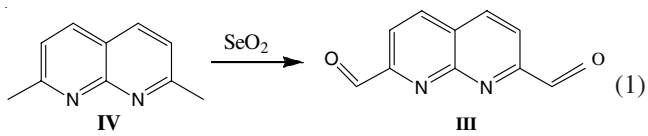
Several synthetic approaches for synthesizing 1,8-naphthyridines were tested. Reliable protocols for synthesis of some new 1,8-naphthyridine-2,7-dialdehydes were developed starting from 2-amino-6-methylpyridine and acetoacetate. The dialdehyde were found to react with pyrrole to form either a polymer or a macrocycle depending on conditions.

Keywords: 1,9-Naphthyridine, 2-Amino-6-methylpyridine, Dialdehyde.

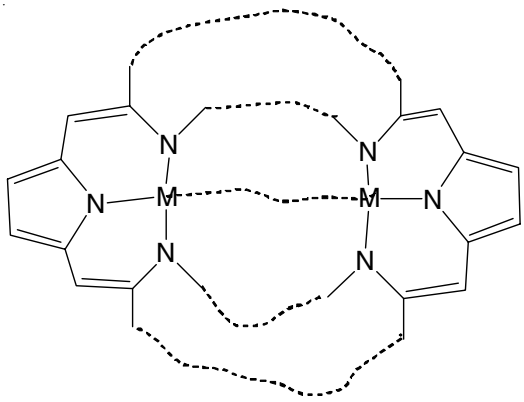
INTRODUCTION

Synthesis of conductive and semi-conductive polymers requires molecules with extended system of conjugation of π-electrons [1]. Their synthesis can be easily arranged by a condensation reaction of dialdehydes of different heterocycles with nucleophiles like pyrroles or thiophenes. Besides synthesis of conductive materials, 1,8-naphthyridine 2,7-dialdehydes, potentially, can be used for synthesis of multi-aromatic polynuclear chelates of general formula **I** [2]. In particular, we are interested in simple yet effective synthesis of 1,8-naphthyridine-2,7-dialdehydes to obtain one of such multi-aromatic chelate (**II**). Chelate **II** was planned to synthesize by reaction of 1,8-naphthyridine dialdehyde (**III**) with pyrrole.

Several authors obtained dialdehyde **III** by oxidation of corresponding dimethyl precursors IV with SeO_2 (eqn. 1) [3-9].

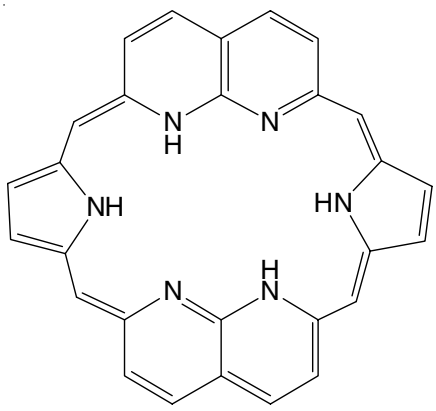


IV **III** (1)

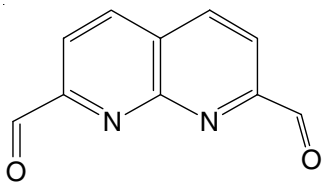


M = Transition metal

I

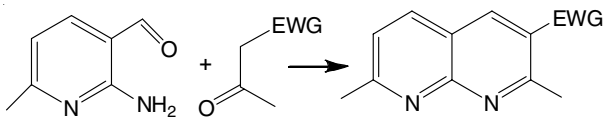


II



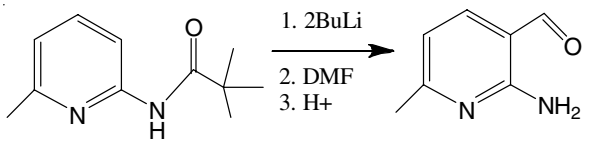
III

Hence, compound **IV** could be the key intermediate in synthesis of dialdehyde **III**. From literature, several reaction pathways were tested in order to synthesize naphthyridine **IV**. However, a Fridlander synthesis reported for the synthesis of aminonicotinaldehyde (eqn. 2) looked the most attractive [10,11].



(2)

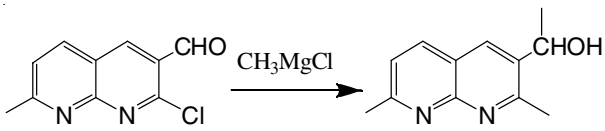
However, corresponding aminoaldehyde (methylaminonicotene aldehyde) turned out to be a problem. Synthesis of this compound using lithiation (eqn 3) was not successful as only the starting aminomethylpyridine was recovered with traces of aldehyde derivative [12].



(3)

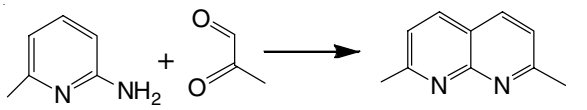
Synthesis of 1,8-naphthyridine [13] is carried out by using the following reaction (eqn 4) which is followed by replacing of chlorine atom with methyl group (eqn 5), however these reactions did not work either. Synthesis of naphthyridine using eqn 4 is failed in our lab. Several products were detected in the reaction mixture but none of them consist of aldehyde as confirmed by NMR analysis.

(4)



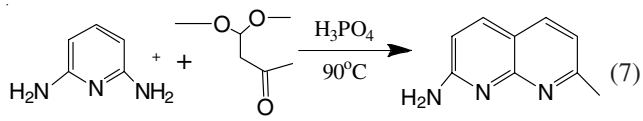
(5)

However, an attempt is made for synthesis of compound **IV** (Skroup synthesis) according to Fox *et al*. [6] using pyridine and crotonaldehyde gave substantial amount of black sticky gum which was very difficult to separate from the desired naphthyridine (eqn 6).



(6)

Brown [14] successfully applied dimethylal of crotonaldehyde to synthesize 2-amino-7-methyl-1,8-naphthyridine (eqn 7). Our attempt to apply this approach to synthesize 2,7-dimethyl-1,8-naphthyridine resulted in recovering of starting pyridine. Above reaction (eqn 7) was also attempted at different temperatures (90, 150 and 250 ºC). Presumably, cyclization failed because of less electron density on aminomethylpyridine as compared with diaminopyridine.



(7)

The only fessible route that gave appropriate results in present experiments was the one based on condensation of 2-aminopicoline and acetoacetic aster [5,7]. However, pursuing this route, several difficulties was occured, so the optimization of literature synthetic procedures is obviously required.

EXPERIMENTAL

All solvents were distilled and stored in air-tight bottles. Solid (Acros) reagents were used as received. NMR spectra were recorded in $CDCl_3$ using Bruker Avance spectrometer with 500 MHz working frequency. Molecular weights were determined by HPLC chromtograph "Waters" in THF solutions at 35 ºC with UV-detector and styrogel 103 and 105 Å columns calibrated with polystyrene standards. High resolution mass spectra (HRMS) were registered on a Bruker Daltonics micrOTOF-Q II instrument using electrospray ionization (ESI). The measurements were done in positive ion mode. Interface capillary voltage: 4500 V; mass range from *m/z* 50 to 3000; external calibration (electrospray calibrant solution, Fluka); nebulizer pressure: 0.4 bar; flow rate: 3 µL/min; dry gas: nitrogen (6 L/min); interface temperature: 180 ºC. Samples were injected into the mass spectrometer chamber from the Agilent 1260 HPLC system equipped with Agilent Poroshell 120 EC-C_{18} column (3.0 × 50 mm; 2.7 µm) and an identically packed security guard using an autosampler. The samples were injected from 50 % acetonitrile (LC-MS grade) in water (MilliQ ultrapure water, Merck Millipore KGaA, Germany) solution. The column temperature was 30 ºC and 5 µL of sample solution was injected. The column was eluted in a gradient of concentrations of A (acetonitrile) in B (water) with the flow rate of 400 µL/min in the following gradient parameters: 0-15 % A for 6.0 min, 15 %-85 % A for 1.5 min, 85 %-0 % A for 0.1 min, 0 % A for 2.4 min.

2,6-dimethyl-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (V): In 1 L flask, 190 g of 115 % polyphosphoric acid is placed and the flask is kept at 60 ºC for 20 min. A solution of 32.4 g (0.3 mol) of 2-aminopicoline in 41 g (0.031 mol) of ethyl acetoacetate is added by small portions with intensive mixing. Formed homogeneous mixture with some foam is left for 0.5 h. Mixing is stopped and temperature raised to 100 ºC and the flask is opened to air letting vapours of ethanol out. After 1 h vine mixture is chilled and compound **V** is isolated by either of the two following methods.

Method A: The reaction mixture is neutralized with 5 M NaOH to pH 4-5 upon chilling with water bath. Colour of the solution is changed from vine to yellow and some oil is separated. The overnight crystallization at room temperature gave yellow crystals. The crystals were filtered under pressure and dried in air. Reasonably dry crystals were dissolved in chloroform or methylene chloride, filtered and the solution was evaporated.

Method B: The reaction mixture was neutralized with 5 M solution of KOH till pH 6-7 extracted with methylene chloride and the solution is evaporated under vacuum at 90 ºC. Brown viscous liquid was allowed to stand overnight at room temper-

ature to form a mixture of crystals and brown oil. The mixture is placed onto sufficient amount of filter paper and kept until all oil was absorbed by filter paper.

Yield: about 13 g (25 %) of dark yellow crystals of 2,6-dimethyl-4*H*-pyrido[1,2-*a*]pyrimidin-4-one pure enough for the next step. Analytical pure compound **V** can be obtained by crystallization from hot heptane as transparent needles. ^{1}H NMR (500 MHz, $CHCl_3$) δ ppm: 2.14 (s, 3H, CH_3), 2.82 (s, 3H, CH_3), 5.91 (s, 1H, CH, Ar), 6.41 (d, 1H, CH, Ar, *J* = 8.3 Hz), 7.09 (d, 1H, CH, Ar, J= 8,3Hz), 7.20 (t, 1H, CH, Ar, *J* = 8.3 Hz).

2,7-Dimethyl-1,8-naphthyridin-4-ol (VI): About 25 mL of diphenyl ether and 26 g of 2,6-dimethyl-4*H*-pyrido[1,2-*a*]-pyrimidin-4-one in 100 mL flask with short Vigreux column without a stopper (to let ethanol vapour out) and without a stirrer are placed into preheated to about 360 ºC at aluminum block. After 4 h of refluxing, the fask content was chilled down and diluted with 30 mL of methylene chloride. The solid product was filtered off and dried in air. Yield: 10.3 g (40 %), poorly soluble in acetone, but soluble in DMSO and DMF.

4-Chloro-2,7-dimethyl-1,8-naphthyridine (VII): To a 100 mL flask containing 21 g (0.11 mol) of dry compound **VI**, about 65-70 mL of $POCl_3$ was added and a short Vigreux column with a stopper is put into the flask and warmed the reaction contents upto ~70 ºC. Then the flask was kept for 1 h at 90 ºC, cooled to room temperature and then dark reaction solution was poured by portions into ~100 g of sodium carbonate in 500 mL of ice-water. Water bath can be used instead of ice to maintain temperature below ~45 ºC. The solution is adjusted to neutral with sodium carbonate and extracted three times with methylene chloride or chloroform. Combined organic solution was dried with sodium sulfate over night, evaporated to dryness and the product was extracted twice with hot heptane. Crystallization of 4-chloro-2,7-dimethyl-1,8-naphthyridine is slow and can take 12 h and even more hours. Precipitated white-orange or white with black coating needles can be recrystallized from hot heptane to yield colourless crystals. Yield: 15.2 g (72 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm: 2.76 (s, 3H, CH_3), 2.80 (s, 3H, CH_3), 7.40 (s, 1H, CH, Ar), 7.39 (d, 1H, CH, Ar, *J* = 8.3 Hz), 8.39 (d, 1H, CH, Ar, J = 8.3 Hz).

4-(4-(*tert*-Butyl)phenoxy)-2,7-dimethyl-1,8-naphthyridine (VIIIa): About 4 g of powdered KOH was placed into a 250 mL flask and 22 g of 4-(*tert*-butyl)phenol was put with it. The mixture was kept at 160 ºC for 2 h periodically shaking. To this hot solution, 7.8 g (0.04 mol) of compound **VII** was added. After 2 h, the mixture was cooled down forming a glass at room temperature. Magnetic stirrer is placed onto the glassy mass, then 40 mL of chloroform and 30 mL of water were added. Stirring this mixture results the dissolution of glassy mass. Then water layer was separated together with white precipitation of poorly soluble in water of sodium *tert*-butylphenolate. The washing repeated several times with new portions of 5 M NaOH solution until no white precipitation formed. Chloroform layer finally evaporated to dryness and pure compound **VIIIa** was obtained by crystallization from hot heptane as beige wax. Yield: 10.6 g (84 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm: 1.39 (s, 9H, *t*-Bu), 2.63 (s, 3H, CH_3), 2.80 (s, 3H, CH_3), 6.47 (s, 1H, CH, Ar), 7.11 (d, 2H, CH, Ar, *J* = 8.3 Hz), 7.33 (d, 1H, CH, Ar, *J* = 8.3 Hz), 7.49 (d, 2H, CH, Ar, *J* = 8.3 Hz), 8.53 (d, 1H, CH, Ar, *J* = 8.3 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 25.00 (CH_3), 25.47 (CH_3), 31.02 (CH_3, *t*-Bu), 34.04 (Cq, *t*-Bu), 104.07(CH, Ar), 111.69 (Cq, Ar), 119.83 (2CH, Ph), 120.77 (CH, Ph), 126.60 (2CH, Ph), 130.73 (CH, Ar), 148.24 (Cq, Ar), 151.26 (Cq, Ar), 156.10 (Cq, Ar), 161.83 (Cq, Ar), 162.54 (Cq, Ar), 162.83 (Cq, Ar). HRMS (ESI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{20}H_{23}N_2O^+$ 307.1810, found 307.1821.

4-Butoxy-2,7-dimethyl-1,8-naphthyridine (VIIIb): To a 5.5 g (0.0285mol) of compound **VII**, 70 mL of 0.7 M NaOBu in butanol was added and the solution was kept over night at room temperature. Most of the butanole was removed under vacuum, 5 mL of water was added and all volatiles were removed under high vacuum at 70-80 ºC. The product **VIIIb** was extracted three times with hot heptane. After left for overnight, light brown crystals were collected and dried in air. Yield: 4.8 g (73 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm: 1.03 (t, 3H, CH_3, *J* = 7.4 Hz), 1.58 (m, 2H, CH_2), 1.91 (m, 2H, CH_2), 2.71 (s, 3H, CH_3), 2.74 (s, 3H, CH_3), 4.17 (t, 2H, CH_2, *J* = 6.4 Hz), 6.62 (s, 1H, CH, Ar), 7.23 (d, 1H, CH, Ar, *J* = 8.3 Hz), 8.36 (d, 1H, CH, Ar, *J* = 8.3 Hz). ^{13}C NMR (100 MHz, CDCl3) δ ppm: 13.27 (CH_3, Bu), 18.75 (CH_2, Bu), 24.90 (CH_3, Ar), 25.65 (CH_3, Ar), 30.37 (CH_2, Bu), 67.78 (OCH_2, Bu), 110.71 (CH, Ar), 111.55 (Cq, Ar), 120.22 (CH, Ar), 130.79 (CH, Ar), 156.18 (Cq, Ar), 161.57 (Cq, Ar), 161.94 (Cq, Ar), 162.89 (Cq, Ar). HRMS (ESI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{20}H_{19}N_2O^+$ 335.1395, found 335.1409.

4-(4-(*tert*-Butyl)phenoxy)-1,8-naphthyridine-2,7-dicarbaldehyde (IXa): In 100 mL flask, 5 g (0.016 mol) of compound **VIIIa**, 5 g (0.05 mol) of SeO_2 and 60 mL of dioxane treated with solid NaOH were added. The mixture was refluxed for 90 min. Dark brown suspension was centrifuged and clear solution was evaporated to dryness. The solid mass refluxed with 15 mL of chloroform for 20 min, then 100 mL of hot heptane was added and after 15 min, refluxed hot mixture was decanted. The extraction was repeated 2 more times. Combined heptane solution stayed over night and precipitated solid recrystallized from hot heptane. Overage yield: 3 g (64 %) of light brown 4-(4-(*tert*-butyl)phenoxy)-1,8-naphthyridine-2,7-dicarbaldehyde. ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm: 1.42 (s, 9H, *t*-Bu), 7.15 (d, 2H, CH, phenyl, *J* = 8.7 Hz), 7.35 (s, 1H, CH, Ar), 7.55 (d, 2H, CH, phenyl, *J* = 8.7 Hz), 8.28 (d, 1H, CH, Ar, *J* = 8.5 Hz), 9.03 (d, 1H, CH, Ar, *J* = 8.5 Hz), 10.24 (s, 1H, CHO), 10.38 (s, 1H, CHO). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 30.89 ($3CH_3$, *t*-Bu), 34.18 (Cq, *t*-Bu), 101.13 (CH, Ar), 111.69 (Cq, Ar), 119.74 (2CH, phenyl), 120.77 (CH, Ar), 127.20 (2CH, phenyl), 130.73 (CH, Ar), 149.50 (Cq, Ar), 150.21 (Cq, Ar), 155.54 (Cq, Ar), 156.20 (Cq, Ar), 156.46 (Cq, Ar), 163.63 (Cq, Ar), 192.56 (CHO), 192.58 (CHO). HRMS (ESI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{20}H_{19}N_2O_3^+$ 335.1396, found 335.1412.

4-Butoxy-1,8-naphthyridine-2,7-dicarbaldehyde (IXb): The procedure is essentially the same as for compound **IXa**. ^{1}H NMR (500 MHz, $CHCl_3$) δ ppm: 1.06 (t, 3H, CH_3, *J* = 7.4 Hz), 1.63 (m, 2H, CH_2), 1.98 (m, 2H, CH_2), 4.37 (t, 2H, CH_2, *J* = 6.4 Hz), 7.53 (s, 1H, CH, Ar), 8.19 (d, 1H, CH, Ar, *J* = 8.3 Hz), 8.83 (d, 1H, CH, Ar, *J* = 8.3 Hz), 10.27 (s, 1H, CHO), 10.34 (q, 1H, CHO). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 13.21 (CH_3, Bu), 18.768 (CH_2, Bu), 30.14 (CH_2, Bu), 69.38 (OCH_2, Bu), 98.30 (CH, Ar), 118.33, (Cq, Ar), 120.25 (CH, Ar), 133.56

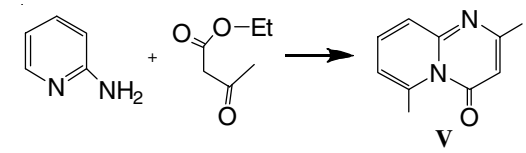
(CH, Ar), 155.17 (Cq, Ar), 155.95 (Cq, Ar), 156.57 (Cq, Ar), 163.20 (Cq, Ar), 192.70 (CHO), 192.91 (CHO). HRMS (ESI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{14}H_{15}N_2O_3^+$ 259.1083, found 259.1106.

Polymer composition made from compound IX: To solution of 0.258 g (1 mmol) of compound **IXb** in 2 mL of chloroform 0.06 g (1 mmol) of pyrrole in 1 mL of chloroform was added. The solution was kept three days at room temperature. Evaporated solution was found to be a polymer with pick molecular weight of 1340 Daltons.

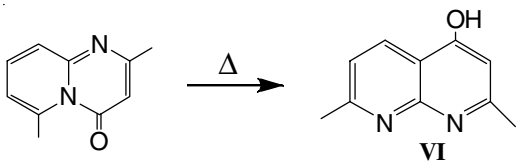
Macrocyclic compound Xb: A 0.002 M solutions of pyrrole and dialdehyde **IXb** are prepared separately in 20 mL of methylene chloride, then 1 mL of each solution were added into nitrogen-flashed flask. After 5 such additions amount of aliquots was increased to 2 mL. When about 1/2 of the solutions have been mixed, addition continued the same way with 4 mL of aliquots. The final mixture allowed to stay 4 more days and evaporated in high vacuum at room temperature to form transparent colourless waxy solid. The solid consist mostly of compound **Xb** with traces of higher oligomers. Yield: 72 % (HPLC). HRMS (ESI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{36}H_{39}N_6O_6^+$ 651.2931, found 651.2902.

RESULTS AND DISCUSSION

According to the literature reaction schemes [5,7], pyrido-[1,2-*a*]pyrimidine (**V**) was obtained by the following reaction (eqn 8) followed by isomerization of compound **V** into compound **VI** at 350 ºC (eqn 9).

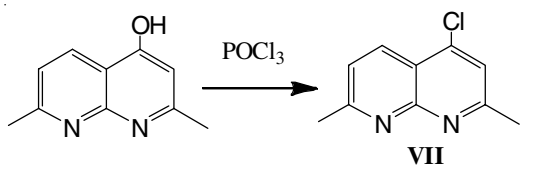


V (8)

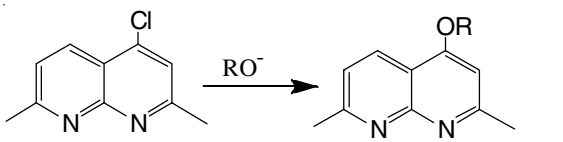


VI (9)

Poorly soluble compound **VI**, then was converted into corresponding ethers, first, by converting into 4-chloro derivative **VII** (eqn 10), followed by the reaction of compound **VII** with alcoholates to produce compound **VIII** (eqn 11).



VII (10)



(11)

VIIIa - R=4-(*tert*-butyl)phenyl
VIIIb - R=butyl

Finally, corresponding dialdehydes **IX** were obtained by oxidation with selenium dioxide. All new naphthyridines, **VIII** and **IX**, showed excellent solubility in solvents of intermediate polarity, like acetone or methylene chloride at room temperature and in non-polar solvent (heptane) at ~100 ºC.

Reproduction of literature reactions (eqns 8-11) gave less yields in several cases. Also, pursuing this route, several difficulties were faced so optimization of literature synthetic procedures was obviously required.

For better results of reaction (eqn 8), 115 % polyphosphoric acid should be used. Less concentrated, *e.g*. 105 % polyphosphoric acid provided poor yields. Temperature of mixing of all reagents is important. Best results were obtained at 60 ºC *vs.* 90 ºC. Based on this observation, temperature less than 60 ºC might be even better but crystallinity and/or high viscosity of polyphosphoric acid at such temperatures prevent mixing to reach complete homogeneity which is important for good results.During the reaction substantial foaming may occur, so the volume of reaction mixture may increase 3-5 times. To prevent foaming and runaway of the reaction solution, mixing is recommended to be stopped at 100 ºC. Major byproduct in eqn 2, is a non-crystallize liquid that does not seem to effect next reaction so that high purity compound **V** is not necessary to synthesize.

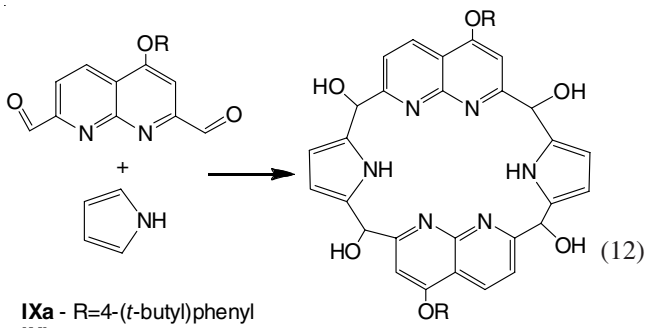
For the reaction (eqn 9), thermal isomerization of compound **V** as recommended in literature 350 ºC requires very high dilution (6 % solution) of compound **V** in paraffin oil. An increase of compound **V** concentration in paraffin results in fast sublimation of compound **V**. We found that isomerization can be conducted at 300 ºC but sublimation is still substantial at this temperature. Reaction (eqn 3) can be conducted in refluxing diphenyl ether at 275 (beginning of the process)-265 ºC (end of the process). These temperatures require more time for completion but prevent sublimation completely. Also, byproducts of reaction (eqn 3) are not soluble in hexane so that the isolation of compound **VI** by dilution of thermal isomerization reaction mixture with methylene chloride is recommended.

For reaction (eqn 10), the replacement of hydroxyl in compound **VI** with chlorine undergoes easier than in many other hydroxypyridines, so that reaction temperature at 90 ºC provides less byproducts as compared with high temperature (100-110 ºC). In the reaction (eqn. 11) of compound **VII** with 4-*tert*-butylphenol is complicated by the formation of strong complex **VII** with excess of phenol. The only method to remove phenol is to do the repeated washing using chloroform solution of reaction products with aqueous KOH or NaOH. Potassium and especially, sodium phenolates are very poorly soluble in water and forms white powder on boundary between chloroform and aqueous phase. Removal of *tert*-butylphenol by sublimation did not provide pure compound **VIII**. In this connection, aliphatic alcohols seem to be more convenient.

Oxidation reaction of methyl groups of compound **VIII** depends on quality of SeO_2 (eqn 1). Different samples of SeO_2 may provide different results. Because of that we prepared selenium dioxide ourselves. Too much of SeO_2 results in formation of carboxylic acid, while insufficient amount of selenium dioxide causes formation of hydroxymethyl groups. The latter is more difficult to separate from dialdehyde **IX** than from acid impurity. The problem of separation of colloidal selenium from

dialdehydes **IX** was achieved by extraction of dialdehydes **IX** with hot heptane.

Naphthyridines **IX** reacted with pyrrole without any acidic catalysts at room temperature to form polyaddition compounds (eqn 12). Cyclic derivative of this reaction (eqn 6) was obtained applying low concentrations of both reagents, however, the reaction was not optimized. Cycles **X** are formed as two regio isomers because of non-symmetrical substitution in naphthyridine **IX**. Besides, presence of oligomers and four chiral centers in **X** (naphthyridine-C*H(OH)-pyrrole) make NMR spectra very broad. All together, it complicates not only isolation of **X** but also their next derivatives. However, there are no doubts that cyclic compound **X** was synthesized under conditions indicated in the experimental sections as HRMS spectra indicate.



(12)

IXa - R=4-(*t*-butyl)phenyl
IXb - R=butyl

Xa - R=4-(*t*-butyl)phenyl
Xb - R=butyl

Conclusion

Synthetic procedures for making 1,8-naphthyridine-2,7-diladehyles were optimized. Several new naphthyridines were synthesized with high solubility in non-polar solvents. It was showed that 1,8-naphthyridine-2,7-diladehyles reacts readily with pyrrole with formation of polymer or macrocyclic product under mild conditions.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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