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# Reactivity of 4-Methylpyridinium Salts in a New Reaction of Ring Transformation of Pyridine and Isoquinoline Derivatives

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**Abstract**—Dependence of reactivity on substituents in the pyridine ring of the 4-methylpyridinium salts was studied in intermolecular reaction of ring transformation involving quaternary salts of pyridinium and isoquinolinium promoted by methylammonium sulfite.

The capability of azines and their quaternary salts to suffer ring transformation under the action of nucleophilic agents is well known. These processes are characteristic of electron-deficient heterocycles with two or more nitrogen atoms, and of their fused analogs [1].

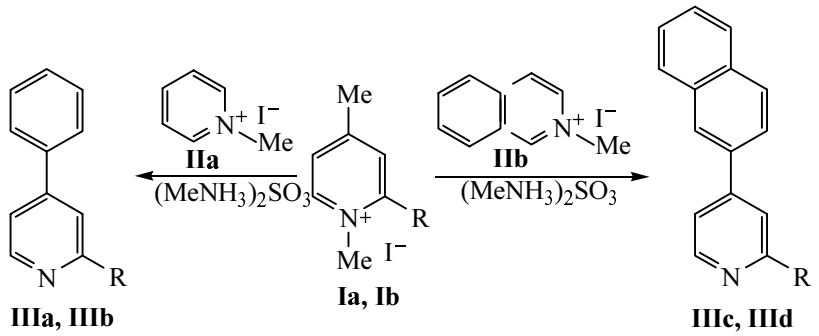
Pyridines are the most stable against ring opening among the six-membered electron-deficient heterocycles. The first studied process of this type was the Zincke–Koenig reaction [2], namely, the cleavage of a quaternizated pyridine ring effected by aromatic amines to afford dianils of glutaconic dialdehyde. Quite a number of the pyridine ring transformations under the action of nucleophiles was discovered lately [3, 4]. First of all a series of new rearrangements should be mentioned, like amidine rearrangement [5, 6], enamine rearrangement of pyridine derivatives [3, 7], and the unusual reaction we discovered of the ring transformation of nitropyridinium salts into indoles [8, 9].

Isoquinoline derivatives may be regarded both as a fused azine system which is characterized as a whole by a higher electron-deficiency with respect to nucleophiles [1, 9], and as pyridine derivatives that typically enter into the nucleophilic additions and ring transformation reactions [10–13].

We formerly established [14–17] that pyridinium and isoquinolinium salts and also pyridine an and isoquinoline proper reacted with 4-methylpyridinium salts under the action of methylammonium sulfite affording as a result of the pyridine ring transformation a 4-phenylpyridine and a 4-(2-naphthyl)pyridine in 57 and 62% yield respectively.

We report here on results of a study on the effect of substituents in the pyridine ring of 4-methylpyridinium salt on the reactivity in the ring transformation reaction of the pyridine and isoquinoline derivatives. As the study objects 4-methyl-2-phenylpyridinium and 4,4'-di-

**Scheme 1.**




**I, III**, R = **(a, c)**, **(b, d)**.

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methyl-2,2'-dipyridylium salts **Ia** and **Ib** were selected (Scheme 1).

The quaternary salts of the heterocyclic bases **Ia** and **Ib** required for this research were prepared in a 92% yield by heating respectively 4-methyl-2-phenylpyridine and 4,4'-dimethyl-2,2'-dipyridyl with methyl iodide in a sealed ampule.

At heating a mixture of 1-methylpyridinium iodide (**IIa**) or 2 methylisoquinolinium iodide (**IIb**) with 2-substituted 1,4-dimethylpyridinium iodide **Ia** and **Ib** in the presence of methylammonium sulfite we obtained 4-arylpyridines and 4-aryldipyridyls **IIIa–IIId** in 7–41% yields (Scheme 1).

In keeping with the previously suggested mechanism of this type reactions [15, 17] apparently the sulfite-ion addition to salt **IIa** and **IIb** occurs thus distorting the aromaticity of the pyridine ring followed by its opening. The non-cyclic intermediate thus formed underwent condensation promoted by a base with the active methyl group of salt **Ia** and **Ib** which in the form of anhydro base acts as a C-nucleophile in the process of the pyridine ring transformation. The subsequent electrocyclic closure leads to a benzene ring and to formation of a quaternary salt of 4 arylpyridinium and 4-aryldipyridylium. The last stage consists in the N demethylation of this quaternary salt under a direct attack of a nucleophile on the Me–N bond affording 4-aryl derivative **IIIa–IIId** (Scheme 2).

It was presumable that isoquinoline derivatives would prove to be more active than pyridine derivatives in the nucleophilic reactions of the pyridine ring transformation. Actually, in reactions with pyridinium salts **Ia** and **Ib** containing a substituent in the position *2* of the pyridine ring we observed this trend: The corresponding 4-naphthyl derivatives **IIIc** and **IIId** were obtained in considerably greater yields that the 4-phenyl derivatives **IIIa** and **IIIb**.

Apparently the presence of a bulky susbtituent in the position *2* of the pyridinium salt **Ia** and **Ib** favors the competing reaction of N-demethylation of the initial salt **Ia** and **Ib** leading to decreased yield of the target products. This statement was indirectly confirmed by detection in the reaction products with the use of TLC of 4,4'-dimethyl-2,2'-dipyridyl and 4 methyl-2-phenylpyridine which as we proved did not react with compounds **IIa** and **IIb** under the conditions of the reaction in question.

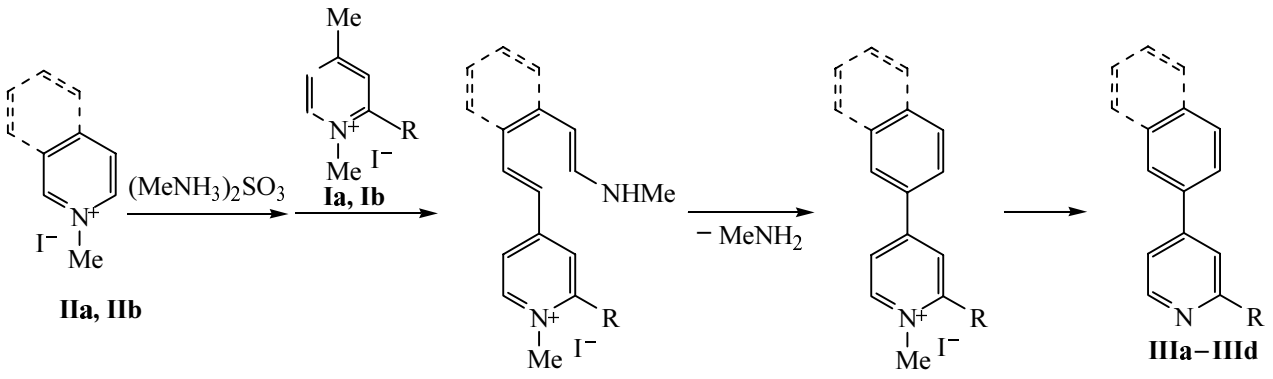
Prior to our studies no published evidence existed on the ability of unsubstituted isoquinoline to take part in reactions of pyridine ring transformation. In the preceding article [17] we described the first instance of the pyridine ring transformation in a non-quaternizated isoquinoline in the course of its reaction with 1,4-dimethyl-pyridinium iodide under the action of methylammonium sulfite yielding 4-naphthylpyridine. The driving force of the reaction is presumably the exchange of amine moiety for methyl-amine in the intermadiate non-cyclic structure (Scheme 3).

We found that at heating isoquinoline and salts **Ia** and **Ib** with the water solution of methylammonium sulfite 4-naphthylpyridine (**IIIc**) formed in a 3% yield, and 4 naphthyldipyridyl (**IIId**) in a 22% yield. Thus the introduction of a substituent into the position *2* of the 1,4-dimethylpyridinium salt reduced the yields of the target products **IIIc** and **IIId** compared to that of 4-naphthylpyridine in [17].

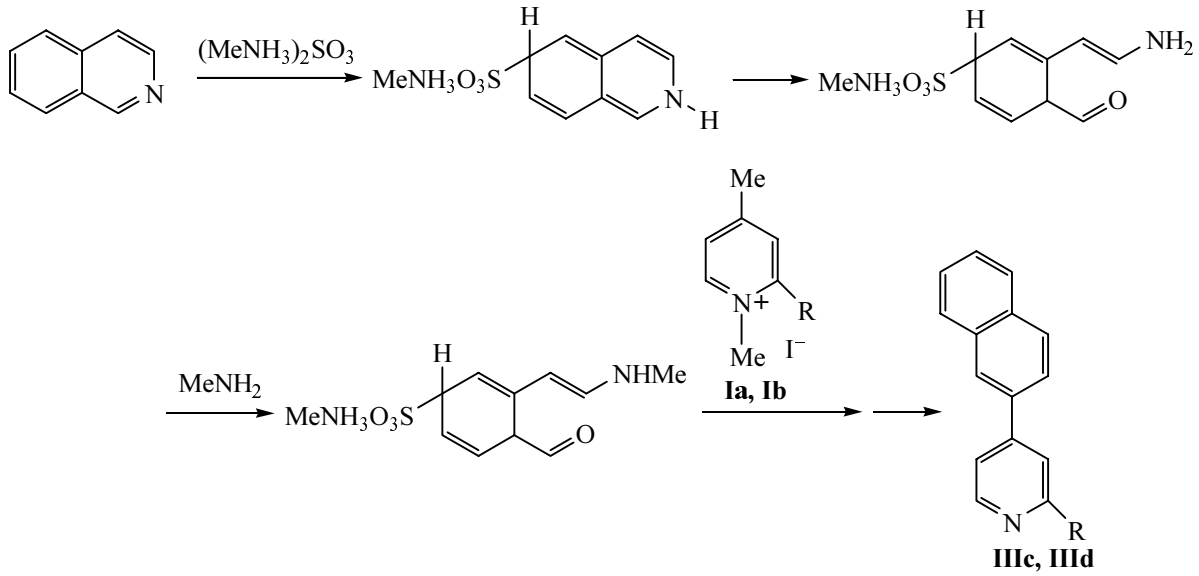
The structure of all compounds obtained was established with the use of $^{1}H$ and $^{13}C$ NMR spectra, in particular, those registered in the two-dimensional mode COSY and NOESY. The composition was supported by elemental analysis and mass spectra.

Dipyridyls and and arylpyridines constitute an important class of heterocyclic compounds whose derivatives are

**Scheme 2.**



**Scheme 3.**




applied as ligands in the complexes of transition metals, also as fluorescent and biologically active compounds, and in preparation of liquid crystals [18–20]. Nowadays the main approaches to the synthesis of 2,2'-dipyridyls and their 4-aryl derivatives consist in various versions of coupling of simple pyridines, Hantzsch sythesis, and Michael reaction [21–23]. In its turn the main procedures for the synthesis of 4-arylpyridines are alternative versions of Suzuki reaction, of free-radical arylation, and regioselective addition of Grignard reagents. The application of Chichibabin reaction was described only for the industrial production of 4 phenylpyridine. However in most cases the 4 arylpyridines and unsymmetrical 4-aryl-2,2'-dipyridyls are difficultly accessible for the listed procedures are relatively expensive, toxic, and originate from difficultly available initial compounds; the target products must be isolated from a mixture with hard-to-separate side reaction products [24–29].

Thus we established that introducing in position *2* of 4-methylpyridinium salt of aryl or hetaryl groups did not prevent the formation of 4-arylpyridines and 4-aryldipyridyls. Simultaneously a simple and convenient method was developed for preparation from available compounds of 4 naphthyl derivatives of 2,2'-dipyridyl and pyridine in good yield and of high purity. The naphthyl derivatives of dipyridyl and pyridine are promising for testing as fluorescent ligands and luminophors.

## EXPERIMENTAL

$^1$H and $^{13}$C NMR spectra were registered on a spectrometer Bruker DRX-500 at operating frequencies 500.13 and 125.76 MHz respectively at 25–30°C from solutions in $CDCl_3$ and DMSO $d_6$ using solvent signals for internal reference. 2D homonuclear $^1$H–$^1$H COSY, NOESY and heteronuclear $^1$H–$^{13}$C COSY (HSQC, HMBC) spectra served for assignment of proton and carbon signals. Chemical shifts were measured with an accuracy of 0.01ppm, coupling constants were good to 0.1Hz. Mass spectra were taken on Varian MAT-311A instrument, high-resolution mass spectra were measured on Finnigan MAT 8430 instrument using perfluorokerosene as standard, ionizing electrons energy 70 eV, a direct admission of the sample into the ion source. The reaction progress was monitored by TLC on DC Alufolen Kieselgel 60 $F_{254}$ (Merck) plates. The column chromatography was carried out using silica gel Kieselgel 60 (0.063–0.100 mm) (Merck). 4,4'-Dimethyl-2,2'-dipyridyl was purchased from Aldrich. 4-Methyl-2-phenylpyridine was obtained as described in [30].

**1,4-Dimethyl-2-phenylpyridinium iodide (Ia).** To 0.338 g (2 mmol) of 4-methyl-2 phenylpyridine was added 5 ml (80 mmol) of methyl iodide. The mixture was heated in a sealed ampule on an oil bath at 100°C for 10 h. Then the reaction mixture was evaporated, the residue was boiled with benzene, and benzene was on cooling decanted, the residue was dried in a vacuum. Yield 0.570 g (92%), mp 130–132°C. $^1$H NMR spectrum (DMSO $d_6$, 30°C), δ, ppm: 2.63 s (3, Me), 4.04 s (3 H, NMe), 7.65 m (5H, Ph), 7.95 s (1H, H$^3$ of pyridine), 7.97 br.d (1H, H$^5$ of pyridine, *J* 6.4 Hz), 8.95 d (1H, H$^6$ of pyridine, *J* 6.4 Hz). Found, %: C 50.08; H 4.51; N 4.35. $C_{13}H_{14}IN$. Calculated, %: C 50.18; H 4.53; N 4.50.

**1,4,4'-Trimethyl-2,2'-dipyridylium iodide (Ib).** To 0.370 g (2 mmol) of 4,4'-dimethyl-2,2'-dipyridyl was added 5 ml (80 mmol) of methyl iodide. The mixture was heated in a sealed ampule on an oil bath at 100°C for 20 h. The separated precipitate was filtered off and recrystallized from a mixture benzene–methanol, 1:1. Yield 0.597 g (92%), mp 190–191°C (from benzene–methanol, 1:1). $^1$H NMR spectrum ($CDCl_3$, 25°C), δ, ppm: 2.53 s (3H, MeC$^4$ of methylpyridyl), 2.73 s (3 H, MeC$^4$ of pyridine), 4.43 s (3 H, NMe of pyridine), 7.35 m (1 H, H$^5$ of methylpyridyl), 7.82 br.s (1 H, H$^3$ methylpyridyl), 7.90 br.s (1H, H$^3$ of pyridine), 7.95 m (1H, H$^5$ of pyridine), 8.62 d (1H, H$^6$ of methylpyridyl, $J$ 4.8 Hz), 9.38 d (1 H, H$^6$ of pyridine, $J$ 6.3 Hz). Found, %: C 47.97; H 4.48; N 8.61. $C_{13}H_{15}IN_2$. Calculated, %: C 47.87; H 4.64; N 8.59.

**2,4-Diphenylpyridine (IIIa).** To 0.105 g (5 mmol) of 1-methylpyridinium iodide (**IIa**) and 0.312 g (1 mmol) of compound **Ia** dissolved in 0.2 ml of water was added 10.8 ml of 40% water solution of $MeNH_2$ and 5.2 ml of 68% solution of $MeNH_3HSO_3$. The reaction mixture in a sealed ampule placed in a metal pressure reactor was heated for 60 h to 230°C on a bath with Wood's alloy. On opening the ampule the reaction mixture was diluted with water and extracted with benzene. The extract was dried over $MgSO_4$ and evaporated in a vacuum. The residue was purified by column chromatography on $SiO_2$ eluting first with hexane, then with a mixture hexane–ethyl acetate, 2:1. Yield 16 mg (7%), mp 69–70°C (from hexane) [23]. $^1$H NMR spectrum ($CDCl_3$, 25°C), δ, ppm: 7.49 m (5, H$^3$, H$^5$ of phenyls, H$^3$ of pyridine), 7.53 m (1, H$^5$ of pyridine), 7.60 m (2 H, H$^4$ of phenyls), 7.72 m (1, H$^6$ of pyridine), 7.82 m (4, H$^2$, H$^6$ of phenyls).

**4'-Methyl-4-phenyl-2,2'-dipyridyl (IIIb).** To 0.663 g (3 mmol) of 1-methylpyridinium iodide (**IIa**) and 0.326 g (1 mmol) of compound **Ib** dissolved in 1.5 ml of water was added 1.5 ml of 40% water solution of $MeNH_2$ and 1.3 ml of 68% solution of $MeNH_3HSO_3$. The reaction mixture in a sealed ampule placed in a metal pressure reactor was heated for 60 h to 230°C on a bath with Wood's alloy. On opening the ampule the reaction mixture was diluted with water and extracted with benzene. The extract was dried over $Na_2SO_4$ and evaporated in a vacuum. The residue was purified by column chromatography on $SiO_2$. As eluent was first used a mixture ethanol–acetic acid, 4:1, then ethanol. The obtained 4-phenyldipyridyl **IIIb** was washed with a water solution of $Na_2CO_3$ and extracted into benzene. The extract was evaporated in a vacuum, and the residue was for the second time subjected to chromatography on $SiO_2$, eluent benzene–ethyl acetate, 2:1. Yield 30 mg (12%), mp 72–74°C. $^1$H NMR spectrum ($CDCl_3$, 30°C), δ, ppm: 2.46 s (3 H, Me), 7.16 d (1 H, H$^5$ of methylpyridyl, $J$ 4.7 Hz), 7.43–7.46 m (1 H, H$^4$ of phenyl), 7.48–7.51 m (2H, H$^3$, H$^5$ of phenyl), 7.54 d.d (1 H, H$^5$ of pyridine, $J$ 5.1, $J$ 1.4 Hz), 7.78 d (2 H, H$^2$, H$^6$ of phenyl, $J$ 7.5 Hz), 8.30 br.s (1 H, H$^3$ of methylpyridyl), 8.57 d (1 H, H$^6$ of methylpyridyl, $J$ 4.7 Hz), 8.68 br.s (1 H, H$^3$ of pyridine), 8.73 d (1 H, H$^6$ of pyridine, $J$ 5.1 Hz). $^{13}$C NMR spectrum ($CDCl_3$, 30°C), δ, ppm: 21.38 (Me), 119.43 (C$^3$ of pyridine), 121.74 (C$^3$ of methylpyridyl), 122.32 (C$^5$ of pyridine), 124.99 (C$^5$ of methylpyridyl), 127.36 (C$^2$, C$^6$ of phenyl), 129.20 and 129.22 (C$^3$, C$^5$, C$^4$ of phenyl), 138.51 (C$^1$ of phenyl), 148.39 (C$^4$ of methylpyridyl), 149.17 (C$^6$ of methylpyridyl), 149.59 (C$^4$ of pyridine), 149.74 (C$^6$ of pyridine), 156.09 (C$^2$ of methylpyridyl), 156.97 (C$^2$ of pyridine). Mass spectrum, $m/z$ ($I_{rel}$, %): 246 (100) $[M]^+$, 247 (19), 245 (37), 219 (4), 218 (11), 217 (3), 154 (7), 127 (4), 123 (4), 65 (2). Found, %: C 82.87; H 5.76; N 11.12. $C_{17}H_{14}N_2$. Calculated, %: C 82.90; H 5.73; N 11.37.

**4-(2-Naphthyl)-2-phenylpyridine (IIIc).** To 1.355 g (5 mmol) of 2 methylisoquinolinium iodide (**IIb**) and 0.311 g (1 mmol) of compound **Ia** dissolved in 0.2 ml of water was added 5.4 ml of 40% water solution of $MeNH_2$ and 2.6 ml of 68% solution of $MeNH_3HSO_3$. The reaction mixture in a sealed ampule placed in a metal pressure reactor was heated for 40 h to 190°C on a bath with silicone oil. On opening the ampule the reaction mixture was diluted with water and extracted with benzene. The extract was dried over $MgSO_4$ and evaporated in a vacuum. The residue was purified by column chromatography on $SiO_2$. Elution was performed first with benzene, then with a mixture benzene–ethyl acetate, 20:1. Then the chromatography on $SiO_2$ was repeated eluting with a mixture hexane–pyridine, 15:1. Yield 0.101 g (39%), mp 62–63°C. $^1$H NMR spectrum ($CDCl_3$, 28°C), δ, ppm: 7.48 m (1, H$^4$ of phenyl), 7.54–7.57 m (4 H, H$^5$, H$^3$ of phenyl, H$^6$, H$^7$ of naphthyl), 7.63 m (1 H, H$^5$ of pyridine), 7.83 d (1 H, H$^3$ of naphthyl, $J$8.5 Hz), 7.92 m (1, H$^5$ of naphthyl), 7.97 m (1 H, H$^8$ of naphthyl), 8.00 d (1, H$^4$ of naphthyl, $J$ 8.5 Hz), 8.09 br.s (1H, H$^3$ of pyridine), 8.10 m (2, H$^2$, H$^6$ of phenyl), 8.20 br.s (1, H$^1$ of naphthyl), 8.81 d (1 H, H$^6$ of pyridine, $J$4.9 Hz). $^{13}$C NMR spectrum ($CDCl_3$, 30°C), δ, ppm: 118.96 (C$^3$ of pyridine), 120.41 (C$^5$ of pyridine), 124.55 (C$^3$ of naphthyl), 126.48 (C$^1$ of naphthyl), 126.67 (C$^7$ of naphthyl), 126.83 (C$^6$, C$^9$ of naphthyl), 127.09 (C$^2$, C$^6$ of phenyl), 127.70 (C$^5$ of naphthyl), 128.42 (C$^8$ of naphthyl), 128.78 (C$^3$, C$^5$ of phenyl), 128.93 (C$^4$ of naphthyl), 129.15

($C^4$ of phenyl), 134.45 ($C^{10}$ of naphthyl), 135.58 ($C^2$ of naphthyl), 139.13 ($C^1$ of phenyl), 149.46 ($C^4$ of pyridine), 149.79 ($C^6$ of pyridine), 157.91($C^2$ of pyridine). Mass spectrum, *m/z* ($I_{rel}$, %): 281 (100) $[M]^+$, 280 (52), 278 (6), 252 (7), 204 (7), 176 (3), 152 (3), 149 (8), 140 (5), 139 (5). High resolution mass spectrum, $[M]^+$, *m/z*: found 281.1201. $C_{21}H_{15}N$. Calculated *M* 281.1204.

**4-Methyl-4'-(2-naphthyl)-2,2'-dipyridyl (IIId)**. To 0.273 g (1 mmol) of 2 methylisoquinolinium iodide (**IIb**) and 0.328 g (1 mmol) of compound **Ib** dissolved in 0.2 ml of water was added 5.4 ml of 40% water solution of $MeNH_2$ and 2.6 ml of 68% solution of $MeNH_3HSO_3$. The reaction mixture in a sealed ampule placed in a metal pressure reactor was heated for 40 h to 190°C on a bath with silicone oil. On opening the ampule the reaction mixture was diluted with water and extracted with benzene. The extract was dried over $MgSO_4$ and evaporated in a vacuum. The residue was purified by column chromatography on $SiO_2$. The product was eluted first with a mixture hexane–pyridine, 15:1, then hexane–pyridine, 10:1. Yield 0.121 g (41%), mp 120–121°C (from hexane). $^1$H NMR spectrum ($CDCl_3$, 25°C), δ, ppm: 2.46 s (3 H, Me), 7.16 d (1 H, $H^5$ methylpyridyl, *J* 4.5 Hz), 7.54 m (2 H, $H^6$, $H^7$ of naphthyl), 7.65 m (1H, $H^5$ of pyridine, *J* 5 Hz), 7.88 and 7.95 2 m (4H, $H^3$, $H^8$, $H^4$, $H^5$ of naphthyl), 8.26 s (1 H, $H^1$ of naphthyl), 8.33 C (1H, $H^3$ of methylpyridyl), 8.60 d (1 H, $H^6$ of methylpyridyl, *J* 4.5 Hz), 8.76 d (1H, $H^6$ of pyridine, *J* 5 Hz), 8.81 C (1H, $H^3$ of pyridine). $^{13}$C NMR spectrum ($CDCl_3$, 25°C), δ, ppm: 21.15 (Me), 119.21 ($C^3$ of pyridine), 121.67 ($C^5$ of pyridine), 122.08 ($C^3$ of methylpyridyl), 124.65 ($C^3$ of naphthyl), 124.77 ($C^5$ of methylpyridyl), 126.52 ($C^7$, $C^1$ of naphthyl), 126.68 ($C^6$ of naphthyl), 127.63 ($C^5$ of naphthyl), 128.45 and 128.74 ($C^4$, $C^8$ of naphthyl), 133.42 ($C^9$, $C^{10}$ of naphthyl), 135.43 ($C^2$ of naphthyl), 148.12 ($C^4$ of methylpyridyl), 148.95 ($C^6$ of methylpyridyl), 149.15 ($C^4$ of pyridine), 149.56 ($C^6$ of pyridine), 155.86 ($C^2$ of methylpyridyl), 156.77 ($C^2$ of pyridine). Mass spectrum, *m/z* ($I_{rel}$, %): 296 (100) $[M]^+$, 295 (62), 294 (8), 268 (15), 204 (18), 176 (6), 148 (12), 147 (5), 65(5), 58 (40). Found, %: C 85.10; H 5.42; N 9.53. $C_{21}H_{16}N_2$. Calculated, %: C 85.11; H5.44; N 9.45.

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