ORIENTATION IN THE BROMINATION OF 2-ARYL(AND ALKYL)-IMIDAZO[1,2-a]PYRIDINES*

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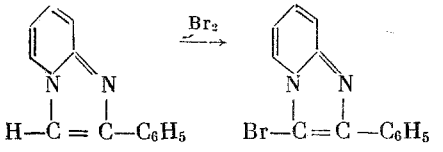
In 1936 by the bromination of 2-phenylimidazo[1,2-a]pyridine (I) Matveev [1] obtained a monobromo derivative, to which he attributed the structure (II); this conclusion, however, was not confirmed experimentally.



(I) (II)

As we were intending to synthesize a number of new compounds from such halogen derivatives, it was necessary to make a closer study of the bromination of 2-substituted imidazo[1,2-a]pyridines to clarify the position regarding orientation in this process. In the present paper we give data which made the position clear. The first task was to determine which of the nuclei—imidazole or pyridine—is attacked by bromine, and we attempted to carry this out by oxidizing the product (II) of the bromination of 2-phenylimidazo[1,2-a]pyridine. In the mixture obtained by treatment of the latter with chromic anhydride in sulfuric acid we found benzoic acid and 2-amino-5-bromopyridine. This result could be interpreted as indicating that bromine attacked the pyridine ring, and not the imidazole or benzene ring of 2-phenylamidazo[1,2-a]pyridine. However, closer investigation showed that the aminobromopyridine was the product of a secondary reaction. This view is supported by the fact that the substituted

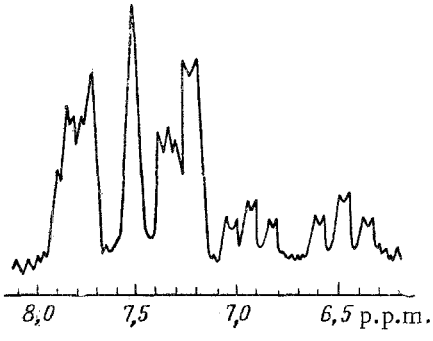


Fig. 1. NMR spectrum of 2-phenylimidazo-[1,2-a]pyridine in CCl_4.

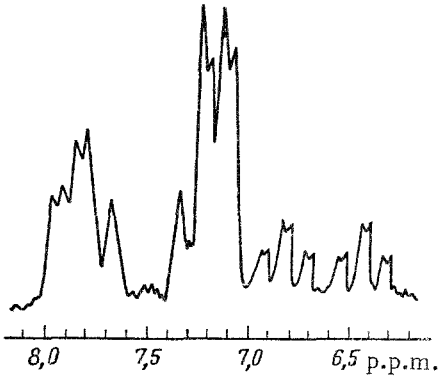


Fig. 2. NMR spectrum of 3-bromo-2-phenyl-imidazo[1,2-a]pyridine in CCl_4.

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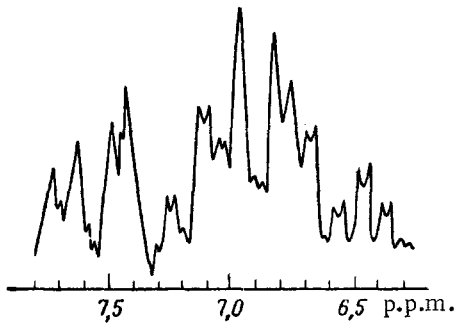
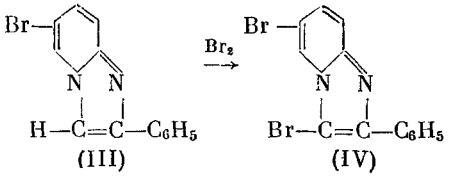


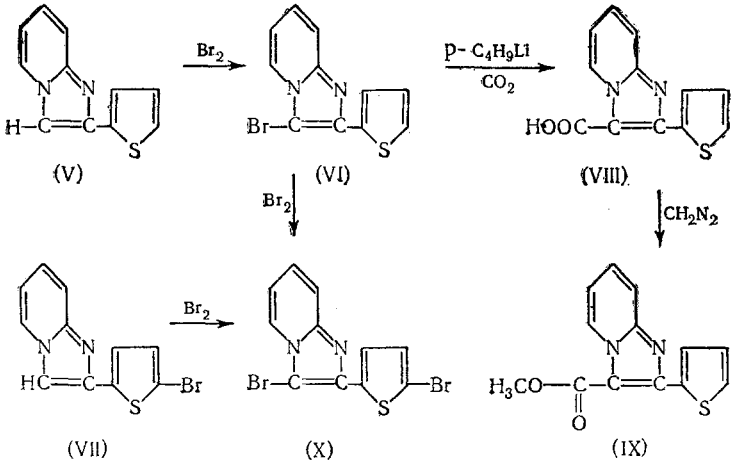
Fig. 3. NMR spectrum of 3-bromo-2-(2-thienyl)imidazo[1,2-a]pyridine in CCl_4.

imidazo[1,2-a]pyridine (III) which we obtained by the condensation of 2-amino-5-bromopyridine with 2-bromoacetophenone was greatly different in its properties from the bromo derivative described by Matveev. It was therefore possible to maintain that this bromo derivative did indeed have the structure (II).

The results of a study of NMR spectra confirmed this conclusion: the signal which may be attributed to the proton in the 3-position of the 2-phenylimidazo[1,2-a]pyridine molecule is a single peak (Fig. 1, at 7.5 p.p.m.). Such a signal is absent in the spectrum of the product of the bromination of 2-phenylimidazo[1,2-a]pyridine (Fig. 2). A priori the presence of the extremely reactive (toward electrophilic reagents) imidazole ring in the 2-phenylimidazo[1,2-a]pyridine molecule does not exclude the possibility of the entry of bromine, when it is in excess, into the pyridine ring (into the 6-position of the bicyclic ring system). However, experiment showed that under the action of bromine in CCl_4 3-bromo-2-phenylimidazo[1,2-a]pyridine does not brominate further. On the other hand, 6-bromo-2-phenylimidazo[1,2-a]pyridine (III) under similar conditions conditions gives a dibromo derivative, probably (IV).



In view of these data it will be understood that 2-methylimidazo[1,2-a]pyridine also brominates in the 3-position with formation of a bromo derivative which differs in properties from the already described 6-bromo-2-methylimidazo[1,2-a]pyridine [2]. A new situation arises when the benzene ring in 2-phenylimidazo[1,2-a]-pyridine is replaced by thiophene, for the new molecule contains two extremely reactive places for the attack of an electrophilic reagent—the 3-position of the bicyclic system and the α-position of the thiophene ring. We found that the bromination product, m.p. 108-111°, obtained in this case was, as shown by thin-layer chromatography on unbound alumina [3], a mixture, but not of two bromo derivatives, but of unchanged 2-(2-thienyl)imidazo[1,2-a]-pyridine (V) and 3-bromo-2-(2-thienyl) imidazo[1,2-a]pyridine (VI). After repeated crystallization or chromatography on a column of alumina this substituted imidazo[1,2-a]pyridine (VI) melted at 113-115°.



The structure of the product of the bromination of 2-(2-thienyl)imidazo[1,2-a]pyridine as 3-bromo-2-(2-thienyl) imidazo[1,2-a]pyridine (VI) followed from a number of facts: 1) in properties it differed from the previously described 2-(5-bromo-2-thienyl)imidazo[1,2-a]pyridine (VII) [4] and from 6-bromo-2-(2-thienyl)imidazo[1,2-a]-

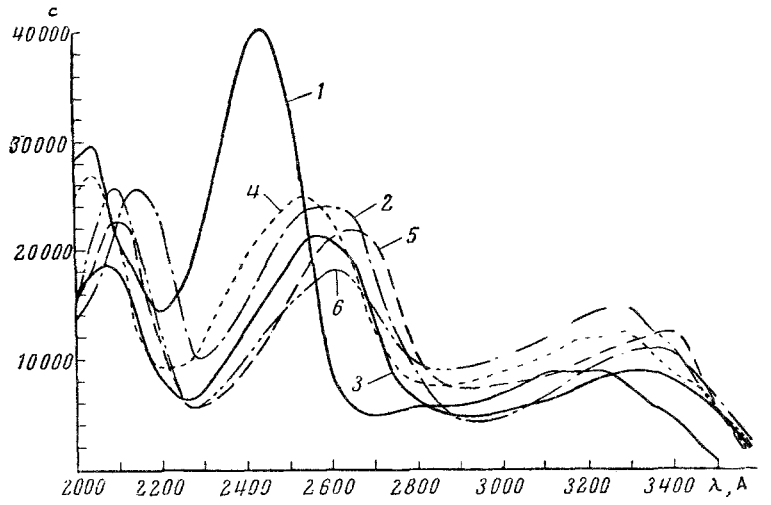
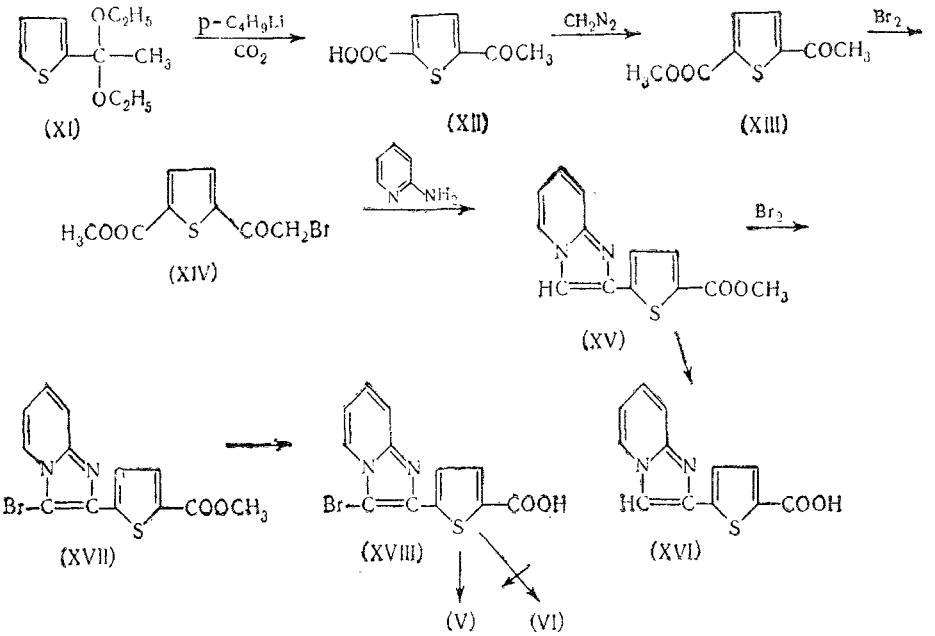


Fig. 4. Ultraviolet spectra: 1) 2-phenyl-; 2) 6-bromo-2-(2-thienyl)-; 3) 3-bromo-2-(2-thienyl)-; 4) 2-(2-thienyl)-; 5) 3-bromo-2(5-bromo-2-thienyl)-; 6) 2-(5-bromo-2-thienyl)-imidazo[1,2-a]pyridines in alcohol.

pyridine, synthesized by us from 2-amino-5-bromopyridine and 2-(bromoacetyl)thiophene (see table); 2) products of the replacement of bromine in the new bromo derivative (VI) differed from the analogous compounds containing the same groupings in the thiophene ring. Hence, as in the case of 2-phenylimidazo[1,2-a]pyridine, in the action of bromine on the imidazo[1,2-a]pyridine (V) the 3-position is the first attacked. The reaction conditions for the replacement of halogen in the 3-position of the imidazo[1,2-a]pyridine system were investigated for the case of 3-bromo-2-phenylimidazo[1,2-a]pyridine, from which via the organolithium compound we prepared 2-phenylimidazo[1,2-a]pyridine-3-carboxaldehyde. Our investigation of the replacement of halogen in (VI) resulted from an attempt which we made to prepare it in the following way, which would establish its structure unequivocally:



We realized all stages of this process except the last. By the method of acetal protection [5-7] through the stage of metalation we obtained 5-acetyl-2-thiophenecarboxylic acid (XII), and by the further action of diazomethane we obtained (XIII), the bromination of which gave methyl 5-(bromoacetyl)-2-thiophenecarboxylate (XIV). Condensation of the latter with 2-aminopyridine gave methyl 5-(imidazo[1,2-a]pyridine-2-yl)-2-thiophenecarboxylate (XV), which gave the corresponding acid (XVI) on hydrolysis. By the action of bromine on the ester (XV) we

obtained methyl 5-(3-bromoimidazo[1,2-a]pyridin-2-yl)-2-thiophenecarboxylate (XVII), which was converted into the acid (XVIII). Unfortunately, we did not succeed in effecting the last stage—decarboxylation in the required direction—and instead of 3-bromo-2-(2-thienyl)imidazo[1,2-a]pyridine (VI), 2-(2-thienyl)imidazo[1,2-a]pyridine (V) was formed. As already stated, the imidazo[1,2-a]pyridine derivatives (XV) and (XVI) differed in properties from the isomeric compounds (IX) and (VIII), prepared from the product (VI) of the bromination of 2-(2-thienyl)-imidazo[1,2-a]pyridine. It was stated above that under our conditions a second bromine atom cannot be introduced into the molecule of (II). On the contrary, 3-bromo-2-(2-thienyl)imidazo[1,2-a]pyridine (VI) is readily brominated. As would be expected, the dibromo derivative (X) then formed is identical in properties with the product of the bromination of 2-(5-bromo-2-thienyl)imidazo[1,2-a]pyridine (VII).

The NMR spectrum of 3-bromo-2-(2-thienyl)imidazo[1,2-a]pyridine (VI) (Fig. 3), like that of its benzene analog (II), contains no signal corresponding to a proton in the 3-position. In this case the situation is complicated by the fact that the signal from the α-proton of the thiophene nucleus falls in the same region (7.5-7.8 p.p.m.). Investigation of the optical properties of the bromine-substituted imidazo[1,2-a]pyridine compounds (Fig. 4) showed that the introduction of halogen in any place in the molecule brings about a shift in the ultraviolet absorption band toward longer waves; the same sort of shift is observed also when a second bromine atom is introduced into the molecule of a monobromo derivative.

EXPERIMENTAL SECTION

2-Amino-5-bromopyridine was prepared by the directions given in [8].

2-Methylimidazo[1,2-a]pyridine was prepared by Chichibabin [9] in 10-12% yield. By changing the isolation conditions we were able to obtain it as a fraction of b.p. 144-150° (20 mm) in 38.7% yield: the reaction mixture was neutralized with sodium ethoxide, ethanol was driven off, and the residue was vacuum-fractionated.

2-Phenylimidazo[1,2-a]pyridine [10] and 2-(2-Thienyl)imidazo[1,2-a]pyridine [4, 11] were prepared by the condensations of 2-aminopyridine with 2-bromoacetophenone and with 2-(bromoacetyl)thiophene in an aqueous medium in presence of sodium bicarbonate. The yield of the first of these compounds was 91%, and the yield of the second was 44%.

a) 2-Aryl(and Alkyl)-3-bromoimidazo[1,2-a]pyridines. To a solution of the 2-aryl(or alkyl)imidazo[1,2-a]-pyridine in $CHCl_3$ or CCl_4 we added an equimolecular amount of bromine in the same solvent dropwise, and stirring was continued at 20° for 3-4 h. The precipitate of the hydrobromide was filtered off. The base was isolated by the method described below (c). A further amount of the product was isolated from the mother solution.

b) 2-Aryl(and Alkyl)-6-bromoimidazo[1,2-a]pyridines. An ethanolic solution of an equimolecular mixture of 2-amino-5-bromopyridine and the appropriate α-bromo ketone was boiled for 10-15 h in a water bath. Solvent was vacuum-distilled off, the residue was dissolved in 10% hydrochloric acid, and the solution was extracted with ether to remove nonbasic impurity. The base was isolated from the resulting solution by the method given below (c).

c) Isolation of the Base. The hydrobromide of the base was dissolved or suspended in water, and the mixture was made alkaline with 20% sodium hydroxide solution. The base was filtered off, washed free from alkali on the filter with water, dried in air, and purified by crystallization from a suitable solvent. The data obtained are summarized in the table.

2-Phenylimidazo[1,2-a]pyridine-3-carboxaldehyde. In a stream of nitrogen 35 ml of ethereal butyllithium solution was added to a solution of 10.9 g of 3-bromo-2-phenylimidazo[1,2-a]pyridine in 80 ml of dry benzene at at −6°. Stirring was then continued at 0° for five hours. To the resulting mixture 3.3 g of dimethyl formamide was added at −10°, and stirring was again continued at 20° for four hours. The mixture was left overnight and then treated with 150 ml of 14% hydrochloric acid. The aqueous layer was separated, made alkaline with sodium hydroxide solution, and extracted with ether. The extract was dried over magnesium sulfate, ether was driven off, and the residue was recrystallized from ethanol. We obtained 4.1 g (45.6%) of product, m.p. 147-148°. Found: C 75.26; H 4.53%. $C_{14}H_{10}N_2O$. Calculated: C 75.66; H 4.43%. The sulfate of the 2,4-dinitrophenylhydrazone of this product formed bright-orange crystals, decomp. temp. 282°. Found: N 16.62; S 6.35%. $C_{20}H_{14}N_6O_4 \cdot H_2SO_4$. Calculated: N 16.79; S 6.41%.

2-(2-Thienyl)imidazo[1,2-a]pyridine-3-carboxylic Acid (VIII). In a stream of nitrogen 26.4 ml of ethereal butyllithium solution was added dropwise to a solution of 5.1 g of 3-bromo-2-(2-thienyl) imidazo[1,2-a]pyridine in 40 ml of dry benzene and 20 ml of dry ether at −20°. The mixture was stirred at −20° for two hours. It was then added to ether mixed with finely divided solid carbon dioxide. On the next day the mixture was treated with 50 ml of water, the organic layer was separated and extracted with 50 ml of water, and the aqueous extracts were combined and acidified with hydrochloric acid to pH 4-3. The precipitate formed was filtered off and dried in a desiccator over calcium chloride. We obtained 3.2 g of a substance with decomp. temp. 114-116°. Extraction of the acidic aqueous solutions with chloroform gave a further 0.4 g of the substance. It is sparingly soluble in acetone, ethanol, and water, and it sublimes in a vacuum when heated in a boiling water bath. By the crystallization of the sublimed substance from acetone we obtained crystals with decomp. temp. 141-143°. Found C 58.77; H 3.24; S 12.97%; neutralization equiv. 245.6; 239.6. $C_{11}H_8N_2O_2S$. Calculated: C 59.00; H 3.30; S 13.13%; neutralization equiv. 244.3.

Methyl 2-(2-Thienyl)imidazo[1,2-a]pyridine-3-carboxylate (IX). A solution containing 2.4 g of diazomethane in 50 ml of ether was added dropwise with stirring to 0.4 g of the acid (VIII) in 150 ml of dry ether at 20-22°. The substance gradually dissolved; stirring was continued further for eight hours. The solution was filtered to remove polymer derived from diazomethane, and solvent was removed in a vacuum. The residue was recrystallized from heptane. We obtained 0.4 g (92.3%) of the ester (IX), m.p. 75.5-77°. Found: C 60.11; H 4.01; S 12.37%. $C_{13}H_{10}N_2O_2S$. Calculated: C 60.45; H 3.9; S 12.42%.

2-Acetylthiophene Diethyl Acetal (XI) was prepared by the method given in [5, 10]; b. p. 82-84° (6 mm); n_D^{20} 1.4862; yield 65.7%. The literature [12] gives: b. p. 84-85.5° (8 mm); n_D^{20} 1.4850.

5-Acetyl-2-thiophenecarboxylic Acid (XII). In a stream of nitrogen a solution of 49.4 g of 2-acetylthiophene diethyl acetal in 200 ml of dry ether was added to an ethereal solution containing 23.4 ml of ethereal butyllithium at −35°. Stirring was then continued for one hour, during which the temperature rose to 5°. In a stream of nitrogen the mixture, cooled to −10°, was added in small portions to ether mixed with finely divided solid carbon dioxide. After four hours the mixture was treated with 100 ml of water, and the ethereal layer was separated and extracted with 100 ml of water. The combined aqueous extracts were acidified with hydrochloric acid. The precipitate formed was filtered off, washed with water, and dried in a vacuum desiccator over calcium chloride. We obtained 31.3 g (74.1%) of a substance of m.p. 204-205° (from water). A mixture of this substance with previously described 5-acetyl-2-thiophenecarboxylic acid [5] melted without depression at 204-205°. The literature gives: m.p. 203.5-204.5° (from water) [13]; m.p. 205.5-206° [5].

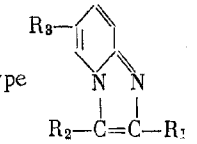
Methyl 5-Acetyl-2-thiophenecarboxylate (XIII). 60 ml of an ethereal solution containing 6.3 g of diazomethane was added dropwise with stirring to 8.0 g of the acid (XII) in 150 ml of dry ether at 20°, and the mixture was left overnight. Ether was driven off, and the residue was recrystallized from octane. We obtained 8.0 g of the ester (XIII), m.p. 137-138°. Found: C 52.25; H 4.74; S 17.56%. $C_8H_8O_3S$. Calculated: C 52.16; H 4.38; S 17.41%.

A mixture of 17.7 g of 5-acetyl-2-thiophenecarboxylic acid, 200 ml of methanol, and 3 ml of sulfuric acid was refluxed for seven hours. The crystals which precipitated on cooling were filtered off and dried in air. We obtained 16 g of the ester (XIII), m.p. 133-135°.

Methyl 5-(Bromoacetyl)-2-thiophenecarboxylate (XIV). Bromine was added dropwise with stirring to a solution of 6.4 g of the ester (XIII) in 600 ml of ether containing 0.3 g of anhydrous aluminum chloride until the ethereal solution acquired a permanent coloration (11.2 g of bromine). Stirring was then continued at 20° for six hours. Ether was driven off in a vacuum, and the residue was filtered off, washed on the filter with cold water, dried in a desiccator over calcium chloride, and recrystallized from heptane. We obtained 8.26 g (90.5%) of colorless needles, m.p. 122-123°. Found: C 36.55; H 2.65; S 12.61; Br 30.06%. $C_8H_7BrO_3S$. Calculated: C 36.52; H 2.68; S 12.19; Br 30.37%.

Methyl 5-(Imidazo[1,2-a]pyridin-2-yl)-2-thiophenecarboxylate (XV). A mixture of 2.1 g of 2-aminopyridine and 6.0 g of methyl 5-(bromoacetyl)-2-thiophenecarboxylate (XIV) in 20 ml of absolute ethanol was boiled for two hours. The mixture was treated by method (b), and the base was isolated by method (c) and recrystallized from absolute ethanol. We obtained 4.8 g (81%) of (XV), m.p. 187-188°. Found: C 60.20; H 4.08; N 10.96; S 12.39%. $C_{13}H_{10}N_2O_2S$. Calculated: C 60.45; H 3.9; N 10.85; S 12.42%.

By the removal of ether from the ether extract, dried over magnesium sulfate, we obtained 1 g of unchanged ester (XIV), m.p. 120-123°.

Bromine-Substituted Imidazo[1,2-a]pyridines of the Type 

No.	R_1	R_2	R_3	Imidazo[1,2-a]pyridine	Molecular formula	M.p., °C (solvent)	Found, %			Calculated, %			Yield, %	Ref.
							C	H	Br	C	H	Br		
1	C_6H_5	Br	H	3-Bromo-2-phenyl-	$C_{13}H_9BrN_2$	88-90 (octane)	–	–	–	–	–	–	90.2	[1]
2	CH_3	Br	H	3-Bromo-2-methyl-*	$C_8H_7BrN_2$	73-74 (heptane)	45.91	3.37	37.79	45.52	3.34	37.86	72.6	
3	C_4H_3S	Br	H	3-Bromo-2-(2-thienyl)-†	$C_{11}H_7BrN_2S$	113.5-115 (octane)	47.16	2.42	28.56	47.32	2.53	28.63	73	
4	C_6H_5	H	Br	6-Bromo-2-phenyl	$C_{13}H_9BrN_2$	201-202 (ethanol)	57.43	3.36	29.48	57.16	3.32	29.26	70.4	
5	CH_3	H	Br	6-Bromo-2-methyl-	$C_8H_7BrN_2$	101-101.5 (petroleum ether)	45.37	3.4	38.38	45.52	3.34	37.86	40	[2]
6	C_4H_3S	H	Br	6-Bromo-2-(2-thienyl)-	$C_{11}H_7BrN_2S$	218-219 (ethanol)	47.38	2.74	28.48	47.32	2.53	28.63	46	
7	C_6H_5	Br	Br	3,6-Dibromo-2-phenyl-	$C_{13}H_8Br_2N_2$	156-157 (ethanol)	44.6	2.38	45.10	44.35	2.29	45.40	84.6	
8	C_4H_3S	Br	Br	3,6-Dibromo-2-(2-thienyl)-‡	$C_{11}H_6Br_2N_2S$	145-146 (octane)	36.99	1.72	–	36.89	1.69	–	85.5	
9	C_4H_2BrS	H	H	2-(6-Bromo-2-thienyl)-	$C_{11}H_7BrN_2S$	143-144 (methanol)	–	–	–	–	–	–	72	[4]
10	C_4H_2BrS	Br	H	3-Bromo-2-(6-bromo-2-thienyl)-	$C_{11}H_6Br_2N_2S$	132-134.5 (heptane)	36.94	1.7	44.12	36.89	1.69	44.64	88.3 [from (3)] 74 [from (9)]	

*B.p. 150-153° (14 mm).

†3-Bromo-2-(2-thienyl)imidazo[1,2-a]pyridine, m.p. 108-111°: after isolation from the reaction mixture it was purified by repeated crystallization from octane or by chromatography on a column of alumina (0.9 g of product per 40 g of alumina of activity II; elution was with a mixture of equal volumes of petroleum ether and diethyl ether).

‡The bromination of 6-bromo-2-(2-thienyl)imidazo[1,2-a]pyridine was conducted in glacial acetic acid at 20°.

5-(Imidazo[1,2-a]pyridin-2-yl)-2-thiophenecarboxylic Acid (XVI). A mixture of 2 g of methyl 5-(imidazo-[1,2-a]pyridin-2-yl)-2-thiophenecarboxylate and 50 ml of 10% hydrochloric acid was boiled for 30 min. The precipitate with formed on cooling was filtered off, washed with water, and dried. We obtained 1.7 g (90%) of the acid (XVI), which after recrystallization from acetone melted with decomposition at 256-258°. Found: C 58.75; H 3.23; S 13.03%; neutralization equiv. 250.0; 244.7. $C_{12}H_8N_2O_2S$. Calculated: C 59.00; H 3.3; S 13.13%; neutralization equiv. 244.3.

Methyl 5-(3-Bromoimidazo[1,2-a]pyridin-2-yl)-2-thiophenecarboxylate (XVII). A solution of 4.9 g of bromine in 30 ml of chloroform was added dropwise with stirring to a solution of 7.9 g of the ester (XV) in 200 ml of chloroform. Stirring was continued further for two hours at 20°. Chloroform was distilled off. The product was isolated from the residue as indicated in method (c). We obtained 9 g (88%) of a substance of m.p. 139-142°, which after crystallization from nonane melted at 140-142°. Found: C 46.18; H 2.83; Br 23.19%. $C_{13}H_9BrN_2O_2S$. Calculated: C 46.30; H 2.69; Br 23.7%.

5-(3-Bromoimidazo[1,2-a]pyridin-2-yl)-2-thiophenecarboxylic Acid (XVIII). A mixture of 4 g of the ester (XVII) and 60 ml of 5% hydrochloric acid was boiled for two hours. The precipitate formed was filtered off, washed with water, and dried in a desiccator over calcium chloride. We obtained 2.9 g of a substance which, after recrystallization from acetone, melted with decomposition at 239-241°. Found C 44.60; H 2.31; S 9.77%; neutralization equiv. 322.9. $C_{12}H_7BrN_2O_2S$. Calculated: C 44.60; H 2.18, S 9.92%; neutralization equiv. 323.2.

Decarboxylation of 5-(3-Bromoimidazo[1,2-a]pyridin-2-yl)-2-thiophenecarboxylic Acid. 2.1 g of the acid was boiled in 30 ml of hexadecane for eight hours. Appreciable resinification occurred. The mixture was extracted with 14% hydrochloric acid. The acid extracts were combined, and the resulting solution was made alkaline with strong sodium hydroxide solution and extracted with ether. The extract was dried over magnesium sulfate, ether was driven off, and we isolated a crystalline substance which was free from halogen. Recrystallization from octane gave 0.3 g of a substance of m.p. 135-136.5°. A mixture of this with 2-(2-thienyl)imidazo[1,2-a]pyridine melted at 135-137°.

NMR spectra were determined in the Physicochemical Investigation Laboratory of the Institute for the Chemistry of Natural Products with an INM C-60 spectrometer (60 MHz) by V. M. Sheichenko, and the absorption spectra were measured in the Optical Laboratory of the Institute of Organic Chemistry by V. A. Petukhov; to these workers the authors express their sincere thanks.

CONCLUSIONS

1. In the bromination of 2-methyl- and 2-phenyl-imidazo[1,2-a]pyridines the hydrogen in the 3-position is replaced.

2. In the case of 2-(2-thienyl)imidazo[1,2-a]pyridine, despite the presence in the molecule of the α-hydrogen atom of the thiophene ring which is highly reactive toward electrophilic reagents, the orientation in the bromination process remains the same.

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