ISSN 1070-3632, Russian Journal of General Chemistry, 2011, Vol. 81, No. 3, pp. 535–541. © Pleiades Publishing, Ltd., 2011.
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Reaction of Acetyl- and 1,1'-Diacetylferrocene with Isatin (Pfitzinger Reaction)

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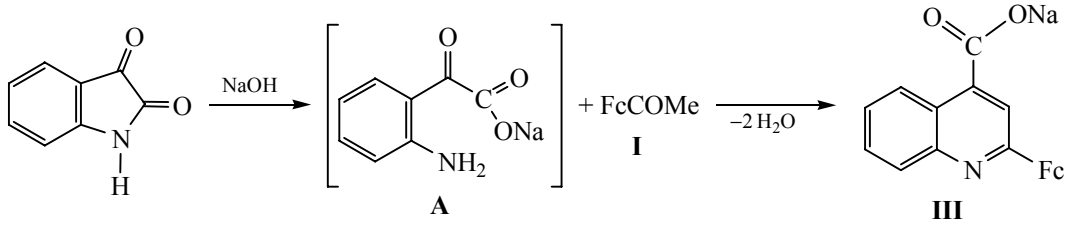
Received March 23, 2010

Abstract—An efficient method for the synthesis of 2-ferrocenyl-substituted quinoline-4-carboxylic acids via the reaction of acetyl- and 1,1'-diacetylferrocene with isatin under the conditions of the Pfitzinger reaction was developed. Starting from the obtained acids methyl esters, amides, *N*-methyl-*N*-methoxyamides, and oximes (at one of the free acetyl groups) of some of these compounds were synthesized.

DOI: 10.1134/S1070363211030157

It is well known [1] that the Pfitzinger reaction [2] consisting in the interaction of a hydrogen-containing ketones with isatin in strongly alkaline medium is a convenient method of preparation of 2(3)-substituted quinoline-4-carboxylic acids, which are of scientific [3] and practical interest, for example, as biologically active compounds [4, 5]. In continuation of the studies of ferrocene heterocyclic compounds [6, 7] with a broad spectrum of biological activity [8, 9], we examined for the first time the reaction of acetyl- **I** and 1,1'-diacetylferrocene **II** with isatin under the Pfitzinger reaction conditions.

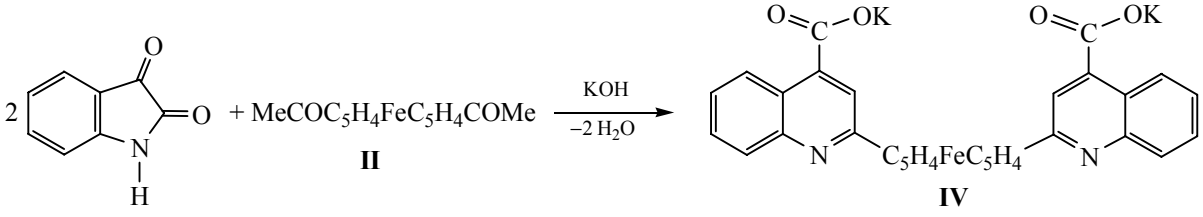
We found that this reaction successfully proceeded at boiling the equimolar amounts of ketone **I** with isatin in the presence of 30-fold excess of NaOH in a dioxane–water mixture (1:1) over 6 h. At the same time, according to the published data [10], initially isatin is hydrolyzed to isatic acid, whose sodium salt **A** then condenses with ketone **I** to give sodium 2-ferrocenylquinoline-4-carboxylate **III** in a quantitative yield.



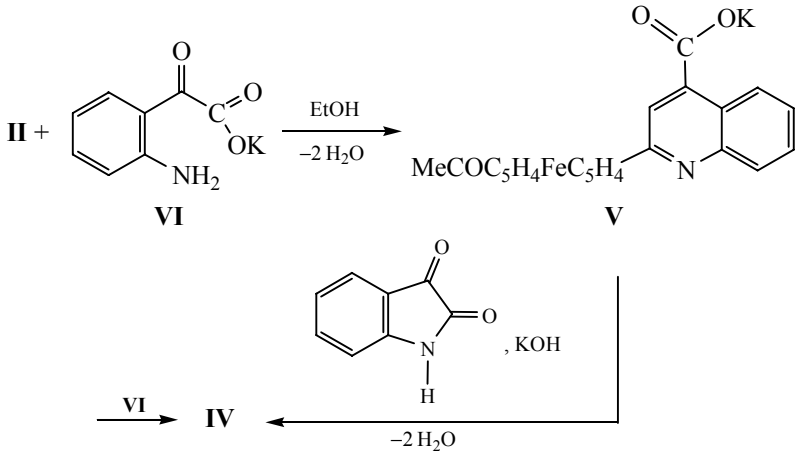
Fc = $C_5H_5FeC_5H_5$.

Under similar conditions the reaction of diketone **II** and isatin at the molar ratio 1:2 in the presence of twenty-fold excess of KOH produces dipotassium salt of 1,1'-di(4-carboxylatoquinolin-2-yl)ferrocenylene **IV** in a quantitative yield.

Using the molar ratio of reagents 1:1, in the same reaction conditions we obtained (according to GC–MS data) only a mixture of compounds **IV** and **V** (~1:2), which could not be separated. However, when in the reaction with diketone **II** was used the specifically

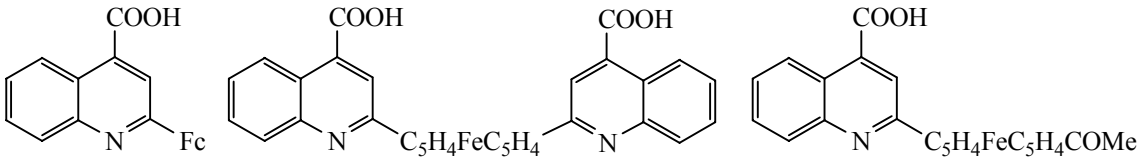


synthesized by procedure [11] potassium salt of isatic acid **VI,** we succeeded in obtaining the product **V** of condensation only by one acetyl group at reflux of equimolar amounts of reactants in anhydrous ethanol, in 96% yield. Further condensation of this compound with salt **VI** excess or with isatin under the Pfitzinger reaction conditions affords dipotassium salt **IV** in a quantitative yield.

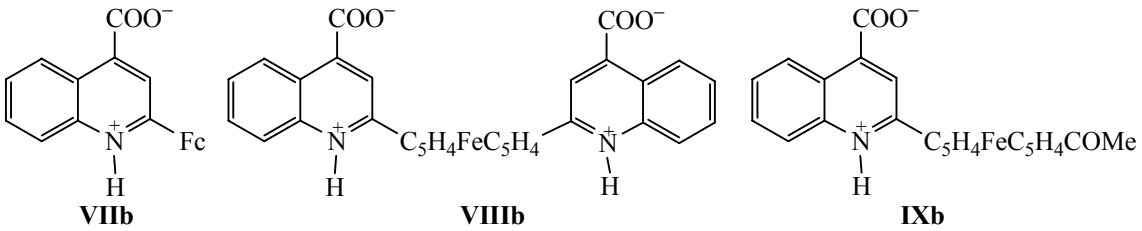


The composition and structure of salts **III–V** are confirmed by elemental analysis, gas chromatography–mass spectrometry (Table 1), IR and ^{1}H NMR spectral data (Table 2), as well as by chemical transformations.

Thus, when the red-orange solutions of a salts **III–V** in methanol were acidified with glacial acetic acid to pH 5–6, the color of the solution at the equivalence point changes quickly to blue-purple due to the formation of the corresponding acids **VIIa–IXa**.



The UV spectra of solutions of these acids, unlike those of the original salts **III–V**, possess a long-wave absorption band at 556–580 nm characteristic of the ferrocene compounds [7, 12, 13] containing the cationic center conjugated with an electronic system of the ferrocenyl skeleton. Apparently, the intra- or intermolecular protonation of the quinoline core in the acids **VIIa–IXa** leads to the correspond-ing ammonium salt **VIIb**, **IXb** where the cationic center is conjugated with the ferrocenyl fragment.



After distilling off the solvent and washing the residue with minimal amount of cold water, the acids **VII–IX** were isolated in an individual state as dark violet solids. They are well soluble in polar organic solvents, poorly soluble in water, and insoluble in ether and hydrocarbons.

Table 1. Yields, melting points, elemental analysis and chromato-mass–spectral data of compounds **III–V**, **VII–XXI**

Comp. no.	Yield, %	mp, °C	Mass-spectrum, *m*/*z* $[M+1]^+$	Found, %				Formula	Calculated, %				*M*
				C	H	Fe	N		C	H	Fe	N	
III	99.5	>250 (decomp.)	358 ($[M+2]^+$)	63.21	3.62	14.68	3.76	$C_{20}H_{14}FeNNaO_2$	63.35	3.70	14.74	3.70	356[a]
V	99.7	>280 (decomp.)	529 ($[M+3]^+$)	59.44	2.83	9.61	4.56	$C_{30}H_{18}FeK_2N_2O_4$	59.62	2.98	9.25	4.64	526[a]
V	96.0	>250 (decomp.)	400 ($[M+2]^+$)	60.18	3.48	12.54	3.37	$C_{22}H_{16}FeKNO_3$	60.43	3.66	12.78	3.20	398[a]
VII	82.9	182–184 (decomp.)	358	67.36	4.17	15.42	3.88	$C_{20}H_{15}FeNO_2$	67.26	4.20	15.65	3.92	357
VIII	84.2	205–207 (decomp.)	529	68.12	3.69	10.35	5.43	$C_{30}H_{20}FeN_2O_4$	68.20	3.79	10.58	5.30	528
IX	78.5	176–178 (decomp.)	400	65.94	4.13	13.85	3.42	$C_{22}H_{17}FeNO_3$	66.19	4.26	14.00	3.51	399
X	70.0	84–85	371	68.03	4.54	15.23	3.68	$C_{21}H_{17}FeNO_2$	68.14	4.60	15.10	3.79	370
XI	69.4	112–113	555	69.41	4.28	9.96	4.84	$C_{32}H_{24}FeN_2O_4$	69.33	4.33	10.08	5.06	554
XII	64.3	96–97	413	66.84	4.56	13.40	3.28	$C_{23}H_{19}FeNO_3$	67.01	4.61	13.56	3.40	412
XIII	54.5	172–173	357	67.33	4.38	15.51	7.64	$C_{20}H_{16}FeN_2O$	67.44	4.50	15.69	7.87	356
XIV	63.2	196–197	527	68.24	4.05	10.34	10.71	$C_{30}H_{22}FeN_4O_2$	68.46	4.18	10.62	10.65	526
XV	51.3	175–176	399	66.28	4.37	13.96	7.16	$C_{22}H_{18}FeN_2O_2$	66.36	4.52	14.04	7.04	398
XVI	64.6	108–109	401	65.84	4.92	14.11	6.93	$C_{22}H_{20}FeN_2O_2$	66.02	5.00	13.97	7.00	400
XVII	68.7	138–139	615	66.30	4.72	8.92	8.85	$C_{34}H_{30}FeN_4O_4$	66.47	4.89	9.10	9.12	614
XVIII	60.8	101–102	443	65.11	4.85	12.61	6.19	$C_{24}H_{22}FeN_2O_3$	65.18	4.98	12.64	6.34	442
XIX	73.8	145–146	429	64.37	4.52	12.90	6.43	$C_{23}H_{20}FeN_2O_3$	64.51	4.67	13.05	6.54	428
XX	78.5	210–212	414	63.87	4.58	13.40	10.09	$C_{22}H_{19}FeN_3O_2$	63.95	4.60	13.53	10.17	413
XXI	74.3	153–154	460	62.48	5.31	12.23	9.25	$C_{24}H_{25}FeN_3O_3$	62.77	5.45	12.17	9.15	459

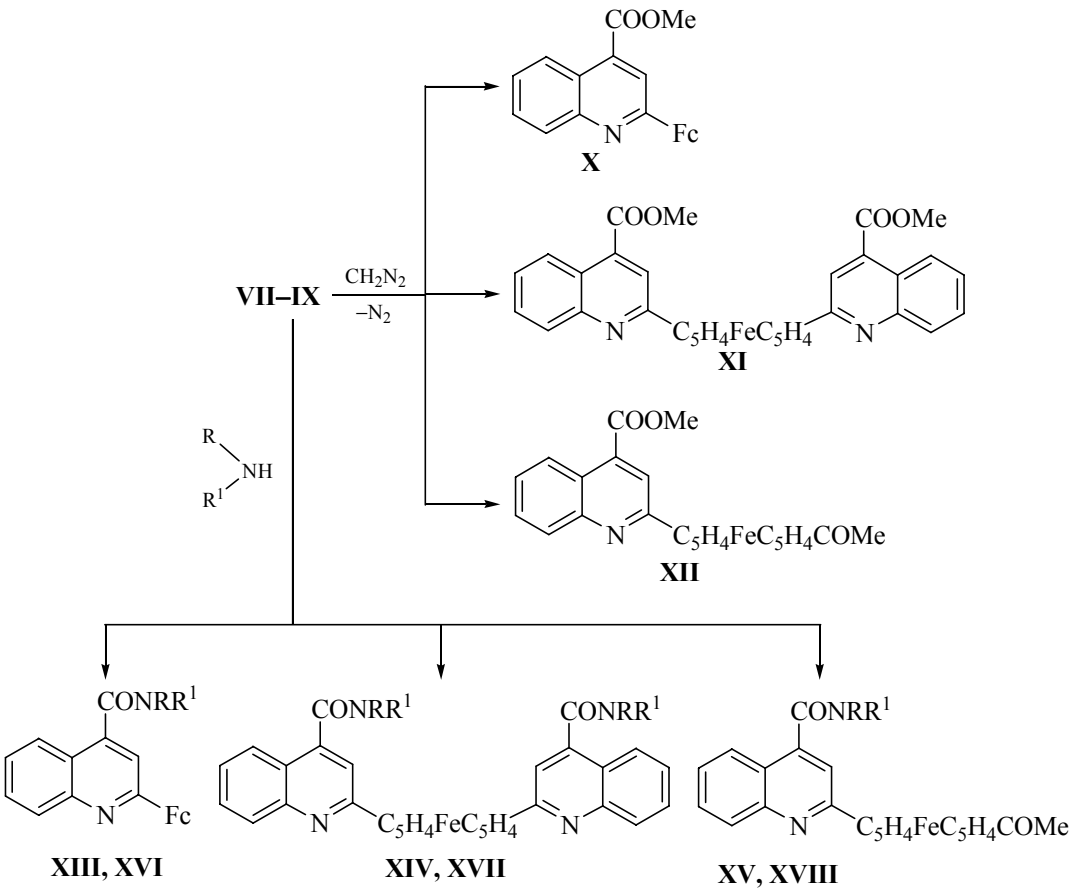
[a] Molecular mass of anion.

Table 2. IR and ^{1}H NMR spectral data for compounds **III–V, VII– XXI**

Comp. no.	IR spectrum, ν, cm^{-1}			^{1}H NMR spectrum, δ, ppm
	Ferrocene core	C=O (C=N)	N–H (O–H)	
III	3021, 1415, 1101, 1002, 834, 487	1686		4.05 s (5H, C_5H_5), 4.47 s (2H, C_5H_2), 5.06 s (2H, C_5H_2), 7.38 t (1H, H^6 quinoline), 7.56 t (1H, H^7), 7.71 s (1H, H^3), 7.85 d (1H, H^5), 8.66 d (1H, H^8)
IV	3005, 1418, 1162, 980, 841, 458	1682, 1675		4.21 s (4H, C_5H_4), 4.93 s (4H, C_5H_4), 7.48 s (2H, $2H^6$ quinoline), 7.53 s (2H, $2H^7$), 7.72 s (2H, $2H^3$), 7.84 s (2H, $2H^5$), 8.68 s (2H, $2H^8$)
V	3000, 1417, 1161, 978, 843, 469	1691, 1675		2.04 s (3H, CH_3), 4.24 m (4H, C_5H_4), 4.87 m (2H, C_5H_2), 5.08 m (2H, C_5H_2), 7.41 t (1H, H^6 quinoline), 7.59 t (1H, H^7), 7.87 s (1H, H^3), 7.98 d (1H, H^5), 8.74 d (1H, H^8)
VII	3020, 1415, 1102, 1000, 835, 487	1703	(3452)	4.08 s (5H, C_5H_5), 4.52 s (2H, C_5H_2), 5.08 s (2H, C_5H_2), 7.38 t (1H, H^6 quinoline), 7.58 t (1H, H^7), 7.78 s (1H, H^3), 7.88 d (1H, H^5), 8.72 d (1H, H^8), 13.01 br.s (1H, COOH)
VIII	3002, 1420, 1163, 980, 842, 463	1708, 1696	(3458, 3428)	4.26 s (4H, C_5H_4), 5.05 s (4H, C_5H_4), 7.52 s (2H, $2H^6$ quinoline), 7.56 s (2H, $2H^7$), 7.78 s (2H, $2H^3$), 7.91 s (2H, $2H^5$), 8.73 s (2H, $2H^8$), 12.98 br.s (2H, 2 COOH)

Table 2. (Contd.)

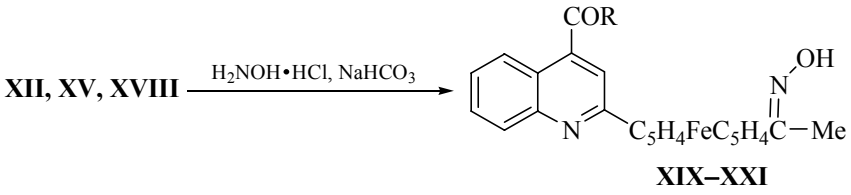
Comp. no.	IR spectrum, ν, cm^{-1}			^1H NMR spectrum, δ, ppm
	Ferrocene core	C=O (C=N)	N–H (O–H)	
IX	3000, 1418, 1170, 978, 842, 468	1705, 1672	(3468)	2.07 s (3H, CH_3), 4.28 m (4H, C_5H_4), 4.91 m (2H, C_5H_2), 5.12 m (2H, C_5H_2), 7.46 t (1H, H^6 quinoline), 7.64 t (1H, H^7), 7.94 s (1H, H^3), 7.99 d (1H, H^5), 8.83 d (1H, H^8), 13.05 br.s (1H, COOH)
X	3015, 1415, 1102, 1001, 835, 491		1701	3.93 s (3H, OCH_3), 4.06 s (5H, C_5H_5), 4.38 m (2H, C_5H_2), 5.01 m (2H, C_5H_2), 7.32 t (1H, H^6 quinoline), 7.52 t (1H, H^7), 7.68 s (1H, H^3), 7.85 d (1H, H^5), 8.62 d (1H, H^8)
XI	3008, 1418, 1170, 983, 842, 457	1704, 1698	3287, 3198	3.95 s (6H, $2OCH_3$), 4.20 s (4H, C_5H_4), 4.90 m (4H, C_5H_4), 7.42 s (2H, $2H^6$ quinoline), 7.49 s (2H, $2H^7$), 7.70 s (2H, $2H^3$), 7.82 s (2H, $2H^5$), 8.61 s (2H, $2H^8$)
XII	3004, 1418, 1168, 982, 845, 460	1705, 1682	3305, 3294, 3208	2.03 s (3H, CH_3), 4.01 s (3H, OCH_3), 4.22 m (4H, C_5H_4), 4.85 m (2H, C_5H_2), 5.06 m (2H, C_5H_2), 7.38 t (1H, H^6 quinoline), 7.54 t (1H, H^7), 7.83 s (1H, H^3), 7.96 d (1H, H^5), 8.71 d (1H, H^8)
XIII	3008, 1416, 1100, 1003, 838, 489	1673	3308, 3205	4.01 s (5H, C_5H_5), 4.39 s (2H, C_5H_2), 4.96 s (2H, C_5H_2), 7.37 t (1H, H^6 quinoline), 7.56 t (1H, H^7), 7.72 s (1H, H^3), 7.84 d (1H, H^5), 8.66 d (1H, H^8), 9.34 s (2H, H_2NCO)
XIV	3000, 1420, 1165, 980, 843, 459	1685, 1671		4.20 s (4H, C_5H_4), 4.90 m (4H, C_5H_4), 7.42 s (2H, $2H^6$ quinoline), 7.53 s (2H, $2H^7$), 7.70 s (2H, $2H^3$), 7.82 s (2H, $2H^5$), 8.64 s (2H, $2H^8$), 9.28–9.36 m (4H, $2H_2NCO$)
XV	3001, 1420, 1165, 980, 844, 468	1675, 1664		2.01 s (3H, CH_3), 4.22 m (4H, C_5H_4), 4.82 m (2H, C_5H_2), 5.03 m (2H, C_5H_2), 7.40 t (1H, H^6 quinoline), 7.59 t (1H, H^7), 7.84 s (1H, H^3), 7.96 d (1H, H^5), 8.72 d (1H, H^8), 9.42 s (2H, H_2NCO)
XVI	3006, 1418, 110, 1001, 842, 481	1680		3.21 s (3H, NCH_3), 3.68 s (3H, OCH_3), 4.04 s (5H, C_5H_5), 4.47 s (2H, C_5H_2), 5.04 s (2H, C_5H_2), 7.37 t (1H, H^6 quinoline), 7.54 t (1H, H^7), 7.72 s (1H, H^3), 7.83 d (1H, H^5), 8.67 d (1H, H^8)
XVII	3000, 1418, 1170, 982, 840, 458	1692, 1673	(3345)	3.24 s (6H, $2NCH_3$), 3.71 s (6H, $2OCH_3$), 4.22 s (4H, C_5H_4), 4.92 s (4H, C_5H_4), 7.47 s (2H, $2H^6$ quinoline), 7.56 s (2H, $2H^7$), 7.70 s (2H, $2H^3$), 7.84 s (2H, $2H^5$), 8.69 s (2H, $2H^8$)
XVIII	2998, 1422, 1168, 978, 845, 464	1688, 1667	(3360)	2.02 s (3H, CH_3), 3.22 s (3H, NCH_3), 3.64 s (3H, OCH_3), 4.22 m (4H, C_5H_4), 4.85 m (2H, C_5H_2), 5.04 m (2H, C_5H_2), 7.41 t (1H, H^6 quinoline), 7.58 t (1H, H^7), 7.84 s (1H, H^3), 7.93 d (1H, H^5), 8.72 d (1H, H^8)
XIX	3014, 1416, 1161, 983, 838, 492	1700 (1638)	(3328)	1.94 m (3H, CH_3), 4.01 s (3H, OCH_3), 4.21 m (4H, C_5H_4), 4.86 m (2H, C_5H_2), 5.04 m (2H, C_5H_2), 7.36 t (1H, H^6 quinoline), 7.52 t (1H, H^7), 7.82 s (1H, H^3), 7.94 d (1H, H^5), 8.72 d (1H, H^8), 10.06 m (1H, =NOH)
XX	3005, 1418, 1169, 980, 841, 460	1690 (1633)		1.93 m (3H, CH_3), 4.23 m (4H, C_5H_4), 4.79 m (2H, C_5H_2), 5.01 m (2H, C_5H_2), 7.38 t (1H, H^6 quinoline), 7.59 t (1H, H^7), 7.85 s (1H, H^3), 7.94 d (1H, H^5), 8.70 d (1H, H^8), 9.42 s (2H, H_2NCO), 10.07 m (1H, =NOH)
XXI	3000, 1421, 1170, 978, 846, 468	1692 (1635)		1.95 m (3H, CH_3), 3.22 s (3H, NCH_3), 3.65 s (3H, OCH_3), 4.22 m (4H, C_5H_4), 4.84 m (2H, C_5H_2), 5.03 m (2H, C_5H_2), 7.42 t (1H, H^6 quinoline), 7.59 t (1H, H^7), 7.85 s (1H, H^3), 7.92 d (1H, H^5), 8.72 d (1H, H^8), 10.03 m (1H, =NOH)



R = R^1 = H (**XIII–XV**), R = Me, R^1 = OMe (**XVI–XVIII**).

Reacting with diazomethane, acids **VII–IX** are readily converted into the corresponding methyl esters **X–XII.** Their reaction with amines (ammonia, *N*-methyl-*N*-methoxyamine) in the presence of dehydrating agent benzotriazol-1-yloxytris(dimethylamino)phosphonio-hexafluorophosphate gives primary **XIII–XV** and tertiary **XVI–XVIII** amides, respectively. The latter are of preparative interest as synthons for obtaining new ferrocene derivatives of quinoline series.

Compounds **XII**, **XV**, **XVIII** containing acetyl group react with hydroxylamine in aqueous methanol to form the corresponding oximes **XIX–XXI**.



R = OMe (**XIX**), NH_2 (**XX**), N(Me)OMe (**XXI**).

Analytical and spectral characteristics of the synthesized compounds **VII–XXI,** confirming unambiguously their structure, are listed in Tables 1 and 2.

Thus, we developed effective methods of preparating ferrocene derivatives of quinoline-4-carboxylic acid based on the Pfitzinger reaction, starting

from the accessible acetyl- and 1,1'-diacetylferrocene. The compounds obtained are of interest as potentially biologically active substances.

EXPERIMENTAL

The IR spectra were recorded on a Specord 75 IR instrument from KBr pellets. The ^{1}H NMR spectra were registered on a Varian Mercury Plus-400 spectrometer (400 MHz) in DMSO-d_6 with internal reference HMDS. The GC–MS spectra were recorded on a Surveyor MSQ instrument (Thermo Finnigan, USA) by the method of chemical ionization at atmospheric pressure. The UV spectra were recorded on a Specord UV-Vis spectrophotometer from 1×10^{-3} M solutions in methanol. Identity and purity of the compounds obtained was monitored by TLC on Silufol UV-254 plates, eluting with hexane–ethyl acetate, 2:1, and detecting with UV irradiation.

Sodium 2-ferrocenylquinoline-4-carboxylate (III). A mixture of 1.47 g of isatin and 11.2 g of NaOH in 20 ml of 50% aqueous dioxane was stirred for 20 min, then mixed with 2.28 g acetylferrocene **I** and refluxed for 6 h with stirring. Then to the cooled mixture was added 100 ml of water. The organic layer was extracted with ethyl acetate (3×80 ml), dried over anhydrous sodium sulfate, and concentrated. The residue was washed in succession with diethyl ether and hexane and dried in a vacuum. Yield 3.77 g (99.5%).

1,1'-Di(4-carboxylatoquinolin-2-yl)ferrocenylene-dipotassium salt (IV). A mixture of 2.94 g of isatin and 11.2 g of KOH in 20 ml of 50% aqueous dioxane was stirred for 20 min, mixed with 2.7 g of 1,1'-diacetylferrocene **II**, and refluxed for 2 h while stirring. The red-brown precipitate formed. The mixture was cooled for 12 h at 6–7°C. The precipitate was filtered off, washed with 10 ml of cold water, with diethyl ether, hexane, and dried in a vacuum at 70°C to constant mass. Yield 6.02 g (99.7%).

1-Acetyl-1'-(4-carboxylatoquinilin-2-yl)ferrocenyl potassium salt (V). A mixture of 2.03 g of potassium salt of isatic acid **VI** [11] and 2.70 g of diketone **II** in 50 ml of anhydrous ethanol was refluxed for 6 h while stirring (monitoring with TLC). The solvent was removed in a vacuum. The solid red-orange residue was washed with ether, hexane and dried to the constant mass. Yield 4.20 g (96%).

2-Ferrocenylquinoline-4-carboxylic acid (VII). To a solution of 3.79 g of sodium salt **III** in 30 ml of methanol was gradually added 0.65 g of glacial acetic acid at stirring. The color of the solution changes to blue-violet. The mixture was stirred at 50°C for 2 h and concentrated. The residue was washed in succession with 5 ml of cold water, ether and hexane and dried in vacuum. Yield 2.96 g (82.9%).

Compounds **VIII** and **IX** were prepared similarly.

Methyl 2-ferrocenylquiniline-4-carboxylate (X). To a solution of 5 mmol of acid **VII** in 15 ml of methanol was added ether solution of diazomethane, prepared from 1.03 g of *N*-nitrosomethylurea by procedure [14]. Nitrogen was liberated, and color of the solution changed from blue-violet to yellow-orange. The mixture was stirred for 2 h (monitoring with TLC). Diazomethane excess was decomposed with acetic acid. The solvent was removed. The residue was subjected to chromatography eluting with ethyl acetate–hexane mixture (1:2).Yield 1.25 g (70%).

Compounds **XI** and **XII** were prepared similarly.

2-Ferrocenylquinoline-4-carboxylic acid amide (XIII). To a mixture of 1.79 g of acid **VII**, 2.66 g of benzotriazol-1-yloxytris(dimethylamino)phosphonio-hexafluorophosphate in 20 ml of DMSO was added 0.8 ml of triethylamine. The mixture was stirred for 1 h at 50°C. Then 1.8 g of ammonium carbonate was added, and the reaction mixture was stirred at room temperature for 70 h (monitoring with TLC). Then the mixture was poured into 100 ml of water, extracted with ethyl acetate (3×60 ml), washed twice with water, dried over anhydrous sodium sulfate, and concentrated. The residue was recrystallized from a mixture hexane–ethanol (2:1). Yield 0.97 g (54.5%).

Compounds **XIV–XVIII** were prepared similarly using *N*-methyl-*N*-methoxyamine hydrochloride and equimolar amount of triethylamine as HCl acceptor.

1-Acetyl-1'-(4-methylcarboxyquinolin-2-yl)ferrocenylene oxime (XIX). To a solution of 5 mmol of compound **XII** in 20 ml of methanol was added 10 ml of aqueous solution of hydroxylamine obtained from 5.5 mmol of $NH_2OH\cdot HCl$ and 5.5 mmol of $NaHCO_3$. The mixture was refluxed for 2 h (monitoring with TLC), cooled, mixed with 50 ml of water, and extracted with ethyl acetate (3×40 ml). The organic layer was washed with water, dried over anhydrous sodium sulfate and concentrated. The residue was recrystallized from aqueous ethanol, 1:4. Yield 1.58 g (73.8%).

Compounds **XX** and **XXI** were prepared similarly.

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