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Dedicated to Academician of the Russian Academy of Sciences N.S.Zefirov on occasion of his 70th anniversary

Nitropyridines: III.* Synthesis of *meta*-Terphenyls by Recyclization of Nitropyridinium Salts

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Abstract—A two-component Hantzsch synthesis was applied to preparation of nitropyridines and their quaternary salts from various enamines and chalcones based on nitroacetophenone. The recyclization of nitropyridinium quaternary salts under the treatment with an aqueous-alcoholic alkali led to preparation of 5'-methylamino-2'-nitro-*m*-terphenyl derivatives.

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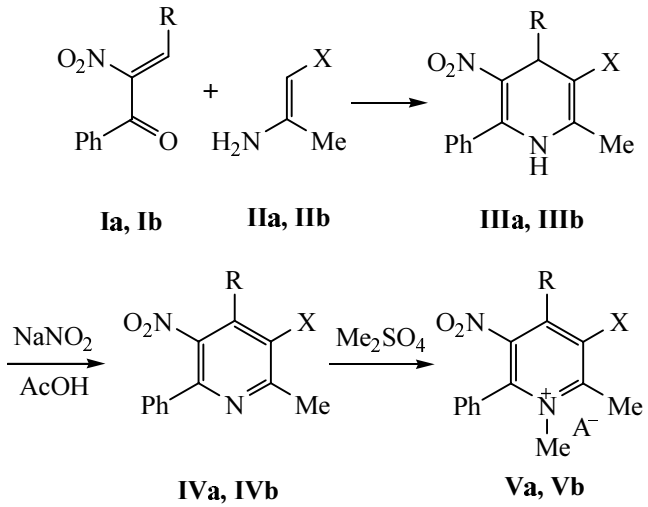
The most of the known *m*-terphenyls were prepared by building up the central benzene ring using the recyclization of pyrilium, thiopyrilium, and 5-nitropyrimidine salts under the treatment of C-nucleophiles [2–4], by the central ring closure from acyclic precursors followed by aromatization [5–8], by cross-coupling [9, 10] and 1,2-addition of Grignard reagents to 1,5-diaryl-3-oxocyclohexenes [11]. The *m*-terphenyls derivatives exhibit a wide range of biological activity [12–15].

Scheme 1.



Ia, Ib **IIa, IIb** **IIIa, IIIb**

IVa, IVb **Va, Vb**

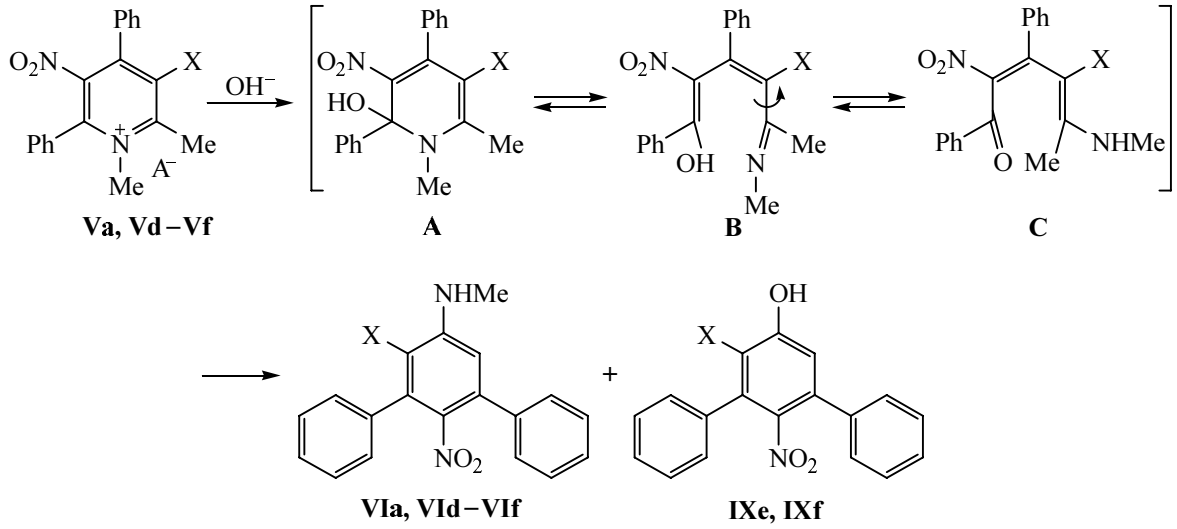
X = CONHPh, R = Ph, A = $MeSO_4$ (**a**); X = CO_2Et, R = 4-$MeOC_6H_4$, A = ClO_4 (**b**).

* For communication II, see [1].

The new approach to the *m*-terphenyls effected in this study was based on the recyclization of diphenylpyridinium salts **Va–Vf** by treating with an aqueous-alcoholic alkali. The key substances in this synthesis are unsymmetrical nitropyridines **IVa–IVf** obtained by a two-component Hantzsch synthesis from nitrochalcones **Ia** and **Ib** and various enamines with subsequent aromatization using sodium nitrite in the acetic acid. Pyridines **IVc–IVf** we described before [16], and compounds **IVa** and **IVb** were newly prepared in this study (Scheme 1). Both stages of the Hantzsch synthesis provided preparative yields. The alkylation of nitropyridinium salts was performed by prolonged heating of pyridines **IVa–IVf** with a triple excess of dimethyl sulfate.

The recyclization of diphenylpyridinium salts **Va, Vd–Vf** at the treatment with the aqueous-alcoholic alkali occurred by an attack of the hydroxy anion on the most electron-deficient position *6* of the pyridinium salt (in the *ortho*-position relative to the nitro group) followed by the isomerization of the pseudobase **A** into an open form **B**. The closure of the central benzene ring in terphenyls **VIa, VId–VIf** involved an intramolecular aldol-crotonic condensation of the arising benzoyl group with the methyl group in the open form **C** (Scheme 2). Yields of *m*-terphenyls amount to 46–70%. 5'-Methylaminoterphenyls **VIa** and **VId** are the only products of salts **Va** and **Vd** recyclization. The recyclization of salts **Ve** and **Vf** occurred with a partial hydrolysis of the enamine fragment of the open form **C** under the alkali action giving rise to 5'-hydroxyterphenyls **IXe** and **IXf,** but the contribution of the process is insignificant.

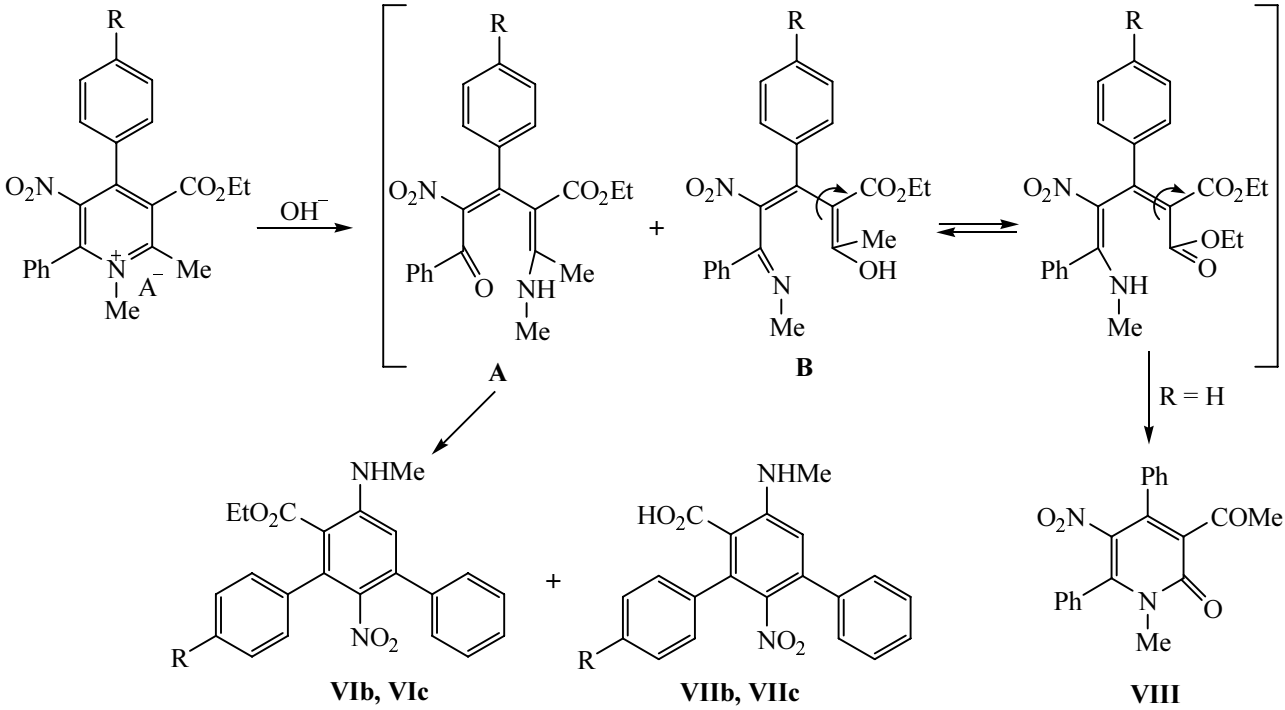
Scheme 2.



X = CONHPh (**a**), CN (**d**), Ac (**e**), Bz (**f**); **V**, A = $MeSO_4$ (**a, d**), ClO_4 (**e**), FSO_3 (**f**).

The recylization of quaternary salts **Vb** and **Vc** led mainly to the formation of 4'-ethoxycarbonyl-*m*-terphenyls **VIb** and **VIc** and 4'-carboxy-*m*-terphenyls **VIIb** and **VIIc** (Scheme 3). The heterocyclization process involving the ester group and giving pyridone **VIII** competes with the recyclization of salt **Vc**. The closure of pyridone **VIII** ring occurs via intramolecular acylation with the ester group of the enamine fragment of open form **B** arising as a result of an attack of the hydroxy anion on the position *2* of the pyridinium salt (into the *ortho*-position with respect to the ester group). It should be noted that for the nicotinic acid esters salts containing in the position *5* electron-withdrawing substituents less strong than the nitro group (CN, $COCH_3$, COPh, CONHPh) this reaction path

Scheme 3.



V–VII, R = OMe, A = ClO_4 (**b**); R = H, A = $MeSO_4$ (**c**).

of recyclization involving the ester group to form pyridones is the principal one [17].

In the ^{1}H NMR spectra of terphenyls **VIa–VIf**, **VIIb**, and **VIIc** the singlet from the aromatic proton of the central benzene ring appeared in the region δ 6.59–7.07 ppm, and the methyl attached to NH gave rise to a doublet at 2.57–2.93 ppm.

The composition and the structure of the newly synthesized compounds were proved by elemental analysis, by IR, ^{1}H NMR, and mass spectra.

It should be stated in conclusion that the recyclization of the available nitropyridines quaternary salts provided a possibility to obtain under mild conditions *m*-terphenyls with a versatile set of substituents in the central benzene ring.

EXPERIMENTAL

^{1}H NMR spectra were registered on a spectrometer Bruker AC-200 (operating frequency 200 MHz), internal referenceTMS. IR spectra were recorded on a spectrophotometer Specord 75IR from chloroform solutions, mass spectra were measured on Finnigan MAT-8200 instrument with direct admission of the sample into the ion source, ionizing electrons emergy 70eV. The reaction progress was monitored and the purity of compounds obtained was checked by TLC on Silufol UV-254 plates, eluents chloroform, chloroform–ethyl acetate, 9:1 or 1:1. Melting points were determined on a Boëtius heating block.

Compounds **Ia**, **IIIc–IIIf–Vc–Vf** we described in [16], *m*-terphenyl **VId**, in [18], **Ib** was synthesized by procedure [19], **IIa**, by that of [20].

2-Methyl-5-nitro-*N*,4,6-triphenyl-1,4-dihydropyridine-3-carboxamide (IIIa). A solution of 2.51 g (10 mmol) of reagent **Ia** and 1.76 g (10 mmol) of enamine **IIa** in 10 ml of glacial acetic acid was stirred for 12 h at room temperature. The precipitated crystals were filtered off and washed with ether to obtain 3.21 g (78%), mp 143–145°C (from aqueous ethenol). IR spectrum, cm^{-1}: 1310, 1520 (NO_2), 1670 (CO), 3380, 3420 (NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.06 s (3H, CH_3), 5.43 s (1H, H^4), 7.04–7.61 m (15H, 3Ph), 9.63 s (1H, NH Ht), 9.95 c (1H, NH_{amide}). Found, %: C 72.72; H 5.09. $C_{25}H_{21}N_3O_3$. Calculated, %: C 72.98; H 5.14.

Ethyl 2-methyl-4-(4-methoxyphenyl)-5-nitro-6-phenyl-1,4-dihydropyridine-3-carboxylate (IIIb) was similarly prepared from dihydropyridine **IIIa**. Yield 52%, mp 132–134°C (from ethanol). IR spectrum, cm^{-1}: 1320, 1500 (NO_2), 1690 (CO), 3440 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 t (3H, $CH_2\underline{CH}_3$, *J* 7.0 Hz), 2.35 s (3H, CH_3), 3.78 s (3H, OCH_3), 4.12 q (2H, $\underline{CH}_2CH_3$, *J* 7.0 Hz), 5.40 s (1H, H^4), 6.06 s (1H, NH), 6.83 d (2H, H$^{3''}$, H$^{5''}$, *J* 8.6 Hz), 7.30–7.44 m (7H, Ph, H$^{2''}$, H$^{6''}$). Found, %: C 67.38; H 5.76. $C_{22}H_{22}N_2O_5$. Calculated, %: C 66.99; H 5.62.

2-Methyl-5-nitro-N,4,6-triphenylnicotinamide (IVa). To a stirred suspension of 4.11 g (10 mmol) of dihydropyridine **IIIa** in 25 ml of glacial acetic acid was added at 60–70°C by portions 1.04 g (15 mmol) of sodium nitrite. On completion of addition of the oxidant the reaction mixture was stirred for 1 h more at the same temperature and then it was diluted with a three-fold volume of ice water and neutralized with ammonia solution. The precipitated crystals were filtered off and washed with water. Yield 2.43 g (80%), mp 201–203°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1540 (NO_2), 1680 (CO), 3420 (NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.69 s (3H, CH_3), 7.03–7.57 m (15H, 3 Ph), 10.58 s (1H, NH_{amide}). Found, %: C 73.65; H 4.72. $C_{25}H_{19}N_3O_3$. Calculated, %: C 73.34; H 4.68.

Ethyl 2-methyl-4-(4-methoxyphenyl)-5-nitro-6-phenylnicotinate (IVb) was prepared in the same way as dihydropyridine **IVa**. Yield 68%, mp 104–106°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1540 (NO_2), 1730 (CO). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.97 t (3H, $CH_2\underline{CH}_3$, *J* 7.1 Hz), 2.70 s (3H, CH_3), 3.79 C (3H, OCH_3), 4.06 q (2H, $\underline{CH}_2CH_3$, *J* 7.1 Hz), 6.89 d (2H, H$^{3''}$, H$^{5''}$, *J* 8.8 Hz), 7.24 d (2H, H$^{2''}$, H$^{6''}$, *J* 8.8 Hz), 7.39–7.65 m (5H, Ph). Found, %: C 67.80; H 5.29. $C_{22}H_{20}N_2O_5$. Calculated, %: C 67.34; H 5.14.

1,2-Dimethyl-5-nitro-N,4,6-triphenylnicotinamide methylsulfate (Va). A mixture of 2.05 g (5 mmol) of pyridine **IVa** and 1.4 ml (15 mmol) of dimethyl sulfate was heated for 10 h at 100°C, then cooled, washed with anhydrous ether, the precipitated crystals were filtered off. Yield 2.57 g (96%), mp 258–260°C (from ethanol, decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.99 s (3H, CH_3), 3.42 s (3H, $CH_3SO_4^-$), 4.00 s (3H, CH_3), 7.10–7.75 m (15H, 3 Ph), 10.80 s (1H, NH). Found, %: C 60.29; H 4.67. $C_{27}H_{25}N_3O_7S$. Calculated, %: C 60.55; H 4.71.

1,2-Dimethyl-4-(4-methoxyphenyl)-5-nitro-6-phenyl-3-ethoxycarbonylpyridinium perchlorate (Vb). A mixture of 1.96 g (5 mmol) of pyridine **IVb** and 1.4 ml (15 mmol) of dimethyl sulfate was heated for 20 h at 100°C, then cooled and washed with ether (3×10 ml). The oily residue was dissolved in 5 ml of water, and

a saturated water solution containing 0.64 g (5.3 mmol) of $NaClO_4$ was added. The separated crystals were filtered off and dried. Yield 1.59 g (63%), mp143–145°C (from ethanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.02 t (3H, $CH_2\underline{CH}_3$, *J* 7.1 Hz), 2.93 s (3H, CH_3), 3.83 s (3H, OCH_3), 3.93 s (3H, CH_3), 4.24 q (2H, $\underline{CH}_2CH_3$, *J* 7.1 Hz), 7.18 d (2H, H$^{3''}$, H$^{5''}$, *J* 8.8 Hz), 7.33 d (2H, H$^{2''}$, H$^{6''}$, *J* 8.8 Hz), 7.61–7.73 m (5H, Ph). Found, %: C 54.37; H 4.49. $C_{23}H_{23}ClN_2O_9$. Calculated, %: C 54.50; H 4.57.

Synthesis of *m*-terphenyls VIa–VIf, VIIb, VIIc, IXe, and IXf. To a suspension of 1 mmol of an appropriate pyridinium salt **Va–Vf** in 4 ml of ethanol was added 1.8 ml of 10% aqueous NaOH, and the mixture was stirred at room temperature for a time interval indicated further. Then the mixture was diluted with water, the precipitated crystals of terphenyls **VIa–VIf** were filtered off and washed with water. The filtrates after separating **VIe** and **VIf** were acidified with 50% aqueous AcOH, and the formed crystals of compounds **IXe** and **IXf** were filtered off and washed with water. Terphenyl **VIc** contained pyridone **VIII**, therefore the mixture was subjected to column chromatography on Silicagel L 60/100, eluent chloroform. Compounds **VIIb** and **VIIc** were isolated by acidifying with 10% HCl filtrates obtained after separation of compounds **VIb** and **VIc**.

5'-Methylamino-2'-nitro-*N*-phenyl-1,1':3'1''-terphenyl-4'-carboxamide (VIa). Yield 70% (reaction time 3 h), mp 193–194°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1520 (NO_2), 1670 (CO), 3420 (NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.84 d (3H, NH$\underline{CH}_3$, *J* 4.6 Hz), 6.12 br.s (1H, $\underline{NH}CH_3$), 6.60 s (1H, H$^{6'}$), 7.16–7.49 m (15H$_{arom}$), 10.12 s (1H, NH$_{amide}$). Mass spectrum, *m/z* (I_{rel}, %): 424 (29.9), 423 (100) [*M*]$^{+\cdot}$, 332 (19.2), 331 (88.8) [M–NHPh]$^+$, 315 (21.0), 314 (90.7) [*M*–NHPh–OH]$^{+\cdot}$, 298 (21.8), 297 (93.8), 285 (16.5)[*M*–NHPh–NO_2]$^{+\cdot}$, 269 (18.5), 256 (17.6) [*M*–NHPh–OH–NO–CO]$^+$, 93 (56.5). Found, %: C 73.91; H 5.12. $C_{26}H_{21}N_3O_3$. Calculated, %: C 73.74; H 5.00. *M* 423.47.

Ethyl-5'-methylamino-4''-methoxy-2'-nitro-1,1':3'1''-terphenyl-4'-carboxylate (VIb). Yield 17% (reaction time 30 min), mp 142–143°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1520 (NO_2), 1690 (CO), 3420 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.75 t (3H, $CH_2\underline{CH}_3$, *J* 7.1 Hz), 2.92 s (3H, NH$\underline{CH}_3$, *J* 5.1 Hz), 3.81 s (3H, OCH_3), 3.86 q (2H, $\underline{CH}_2CH_3$, *J* 7.1 Hz), 6.65 br.s (2H, $\underline{NH}CH_3$, H$^{6'}$), 6.88 d (2H, H$^{3'}$, H$^{5''}$, *J* 8.8 Hz), 7.19 d (2H, H$^{2''}$, H$^{6''}$, *J* 8.8 Hz), 7.35–7.52 m (5H, Ph). Found, %: C 67.69; H 5.32. $C_{23}H_{22}N_2O_5$. Calculated, %: C 67.97; H 5.46.

5'-Methylamino-4''-methoxy-2'-nitro-1,1':3'1''-terphenyl-4'-carboxylic acid (VIIb). Yield 60% (reaction time 30 min), mp 201–202°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1530 (NO_2), 1630 (CO), 3440 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.62 d (3H, NH$\underline{CH}_3$, *J* 4.9 Hz), 3.86 s (3H, OCH_3), 5.45 br.s (1H, $\underline{NH}CH_3$), 7.01 d (2H, H$^{3''}$, H$^{5''}$, *J* 8.8 Hz), 7.04 s (1H, H$^{6'}$), 7.30 d (2H, H$^{2''}$, H$^{6''}$, *J* 8.8 Hz), 7.34–7.50 m (5H, Ph), 13.14 s (1H, COOH). Found, %: C 66.78; H 4.81. $C_{21}H_{18}N_2O_5$. Calculated, %: C 66.66; H 4.79.

Ethyl-5'-methylamino-2'-nitro-1,1':3'1''-terphenyl-4'-carboxylate (VIc). Yield 42% (reaction time 3 h), mp 183–185°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1520 (NO_2), 1680 (CO), 3420 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.66 t (3H, $CH_2\underline{CH}_3$, *J* 7.2 Hz), 2.93 s (3H, NH$\underline{CH}_3$, *J* 5.0 Hz), 3.81 q (2H, $\underline{CH}_2CH_3$, *J* 7.2 Hz), 6.59 s (1H, H$^{6'}$), 6.69 br.s (1H, $\underline{NH}CH_3$), 7.24–7.41 m (10H$_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 377 (24.6), 376 (100) [*M*]$^{+\cdot}$, 330 (10.2) [*M*–NO_2]$^+$, 301 (10.5) [*M*–NO_2–C_2H_5]$^{+\cdot}$, 300 (16.9) [*M*–NO_2–$NHCH_3$]$^{+\cdot}$, 285 (9.6), 273 (10.2), 256 (14.1), 228 (11.1), 226 (9.4), 215 (10.6). Found, %: C 69.86; H 5.42. $C_{22}H_{20}N_2O_4$. Calculated, %: C 70.20; H 5.36. *M* 376.41.

5'-Methylamino-2'-nitro-1,1':3'1''-terphenyl-4'-carboxylic acid (VIIc). Yield 6% (reaction time 3 h), mp 243–245°C (from ethanol). IR spectrum, cm^{-1}: 1370, 1530 (NO_2), 1640 (CO), 3440 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.57 d (3H, NH$\underline{CH}_3$, *J* 4.8 Hz), 5.27 br.s (1H, $\underline{NH}CH_3$), 7.07 s (1H, H$^{6'}$), 7.36–7.53 m (10H$_{arom}$), 13.02 s (1H, COOH). Mass spectrum, *m/z* (I_{rel}, %): 349 (24.8), 348 (100) [*M*]$^{+\cdot}$, 347 (15.8) [*M*–H]$^+$, 318 (10.0) [*M*–$NHCH_3$]$^+$, 317 (31.6) [*M*–H–$NHCH_3$]$^{+\cdot}$, 300 (9.3), 272 (15.5) [*M*–NO_2–$NHCH_3$]$^{+\cdot}$, 244 (10.7), 231 (9.3), 216 (13.0), 215 (44.5), 213 (18.5), 204 (9.4), 202 (14.3), 189 (9.6), 129 (19.8). Found, %: C 69.30; H 4.77. $C_{20}H_{16}N_2O_4$. Calculated, %: C 68.96; H 4.63. *M* 348.46.

3-Acetyl-1-methyl-5-nitro-4,6-diphenylpyridin-2(1*H*)-one (VIII). Yield 9% (reaction time 3 h), mp 166–168°C (from ethanol). IR spectrum, cm^{-1} ($CHCl_3$): 1700, 1640 (CO), 1530, 1360 (NO_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.27 s (3H, $COCH_3$), 3.31 s (3H, NCH_3), 7.25–7.56 m (10H$_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 349 (14.3), 348 (66.95) [*M*]$^{+\cdot}$, 347 (37.6)[*M*–H]$^+$, 334 (9.0), 333 (40.6) [*M*–CH_3]$^+$, 331 (20.6)[*M*–OH]$^+$, 316 (12.4) [*M*–CH_3–OH]$^{+\cdot}$, 301 (26.8) [*M*–OH–NO]$^{+\cdot}$, 300 (100), 299 (13.0), 287 (11.1) [*M*–NO_2–CH_3]$^{+\cdot}$, 286

(43.8), 258 (12.4), 230 (9.5), 202 (11.9), 189 (11.2), 118 (76.6), 77 (43.7) $[Ph]^+$, 43 (18.3) $[COCH_3]^+$. Found, %: C 69.35; H 4.59. $C_{20}H_{16}N_2O_4$. Calculated, %: C 68.96; H 4.63. *M* 348.46.

1-(5'-Methylamino-2'-nitro-1,1':3'1''-terphenyl-4'-yl)ethanone (VIe). Yield 46% (reaction time 6 h), mp 158–160°C (from ethanol). IR spectrum, cm^{-1}: 1340, 1510 (NO_2), 1640 (CO), 3420 (NH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.65 s (3H, $COCH_3$), 2.90 d (3H, $NHCH_3$, *J* 5.0 Hz), 6.59 s (1H, $H^{6'}$), 6.87 br.s (1H, $NHCH_3$), 7.31–7.44 m ($10H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 347 (25.1), 346 (100) $[M]^{+\cdot}$, 332 (14.1), 331 (65.0) $[M - CH_3]^+$, 314 (12.0) $[M - CH_3 - OH]^{+\cdot}$, 297 (15.7), 286 (11.1), 285 (18.3) $[M - NO_2 - CH_3]^{+\cdot}$, 257 (10.7), 256 (11.9), 215 (10.5), 43 (15.2) $[COCH_3]^+$. Found, %: C 72.65; H 5.34. $C_{21}H_{18}N_2O_3$. Calculated, %: C 72.82; H 5.24. *M* 346.39.

(5'-Methylamino-2'-nitro-1,1':3'1''-terphenyl-4'-yl)phenylmethanone (VIf) was obtained by heating for 15 min at 70°C followed by stirring at room temperature for 2 h. Yield 52%, mp 180–181°C (from ethanol). IR spectrum, cm^{-1}: 1360, 1520 (NO_2), 1640 (CO), 3440 (NH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.87 d (3H, $NHCH_3$, *J* 4.6 Hz), 5.39 br.s (1H, $NHCH_3$), 6.65 s (1H, $H^{6'}$), 7.05–7.45 m ($15H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 409 (30.8), 408 (100) $[M]^{+\cdot}$, 407 (50.6) $[M-H]^+$, 391 (12.7) $[M-OH]^+$, 360 (10.9), 332 (10.0) $[M-NO_2 - NHCH_3]^{+\cdot}$, 331 (24.8) $[M-Ph]^+$, 105 (28.3) $[COPh]^+$, 77 (30.3) $[Ph]^+$. Found, %: C 76.23; H 4.87. $C_{26}H_{20}N_2O_3$. Calculated, %: C 76.45; H 4.94. *M* 408.46.

1-(5'-Hydroxy-2'-nitro-1,1':3'1''-terphenyl-4'-yl)-ethanone (IXe). Yield 8% (reaction time 6 h), mp 109–111°C (from ethanol). IR spectrum, cm^{-1}: 1370, 1530 (NO_2), 1640 (CO). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.76 s (3H, $COCH_3$), 7.08 s (1H, $H^{6'}$), 7.30–7.64 m ($10H_{arom}$), 12.24 s (1H, OH). Mass spectrum, *m/z* (I_{rel}, %): 334 (23.2), 333 (100) $[M]^{+\cdot}$, 316 (12.9) $[M-OH]^+$, 301 (11.1) $[M-OH-CH_3]^{+\cdot}$, 287 (16.0), 286 (63.6) $[M-OH-NO]^{+\cdot}$, 284 (24.1), 274 (12.0), 271 (12.8), 268 (11.1), 244 (14.1), 239 (11.6), 216 (13.1), 215 (49.5), 213 (17.6), 202 (15.6), 189 (11.8), 77 (13.4) $[Ph]^+$, 43 (69.7) $[COCH_3]^+$. Found, %: C 71.98; H 4.68. $C_{20}H_{15}NO_4$. Calculated, %: C 72.06; H 4.54. *M* 333.34.

(5'-Hydroxy-2'-nitro-1,1':3'1''-terphenyl-4'-yl)-phenylmethanone (IXf) was obtained by heating for 15 min at 70°C followed by stirring at room temperature for 2 h. Yield 22%, mp 118–120°C (from ethanol). IR spectrum, cm^{-1}: 1380, 1540 (NO_2), 1650 (CO). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 7.00–7.44 m ($16H_{arom}$), 13.04 s (1H, OH). Mass spectrum, *m/z* (I_{rel}, %): 396 (28.4), 395 (100) $[M]^{+\cdot}$, 394 (70.3) $[M-H]^+$, 347 (11.7), 215 (17.1), 105 (83.3) $[COPh]^+$, 77 (51.5) $[Ph]^+$. Found, %: C 76.09; H 4.47. $C_{25}H_{17}NO_4$. Calculated, %: C 75.94; H 4.33. *M* 395.41.

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