

Nitropyridines: VI.* Synthesis of 2-Aryl(hetaryl)- and 2,3-Polymethylene-5-nitropyridines

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Abstract—Previously unknown 2-aryl-, 2-hetaryl-, and 2-cyclopropyl-5-nitropyridines and 2,3-polymethylene-5-nitropyridines were synthesized by reactions of 1-methyl-3,5-dinitropyridin-2(1*H*)-one with various cyclic and acyclic ketones in the presence of ammonia.

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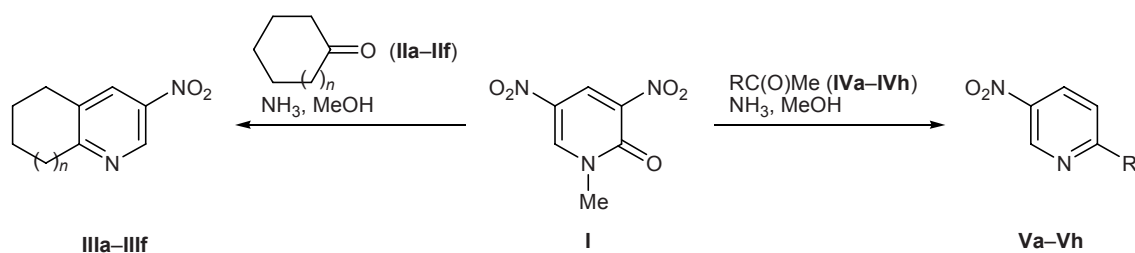
We previously proposed a new synthetic approach to metacyclophanes based on recyclization of quaternary 3-nitro-5,6,7,8,9,10,11,12,13,14-decahydrocyclo-dodeca[*b*]pyridinium salt [2]. Further extension of this novel procedure required nitropyridines **IIIa–IIIf** having cycloalkane rings of different sizes. While searching for convenient methods for the preparation of 2,3-polymethylene-5-nitropyridines, we found a simple procedure [3] for the synthesis of substituted 3-nitropyridines by reaction of 1-methyl-3,5-dinitropyridin-2(1*H*)-one (**I**) with various ketones in the presence of ammonia. 1-Methyl-3,5-dinitropyridin-2(1*H*)-one (**I**) was considered by the authors to be a latent form of nitromalonaldehyde. The reaction of compound **I** with cyclohexanone gave 83% of 3-nitro-5,6,7,8-tetrahydroquinoline (**IIIa**) [3] (Scheme 1).

We extended the series of cyclic ketones for the above reaction by including those containing 8- to 12-membered rings with a view to obtain the corre-

sponding nitropyridines **IIIb–IIIf**. The yield of **IIIb–IIIf** decreased from 65 to 45% as the size of the cycloalkane ring in the initial ketone increased. This procedure for the synthesis of polymethylene-bridged nitropyridines may be regarded as very convenient [4], taking into account accessibility of dinitropyridinone **I** and cyclic ketones and experimental simplicity.

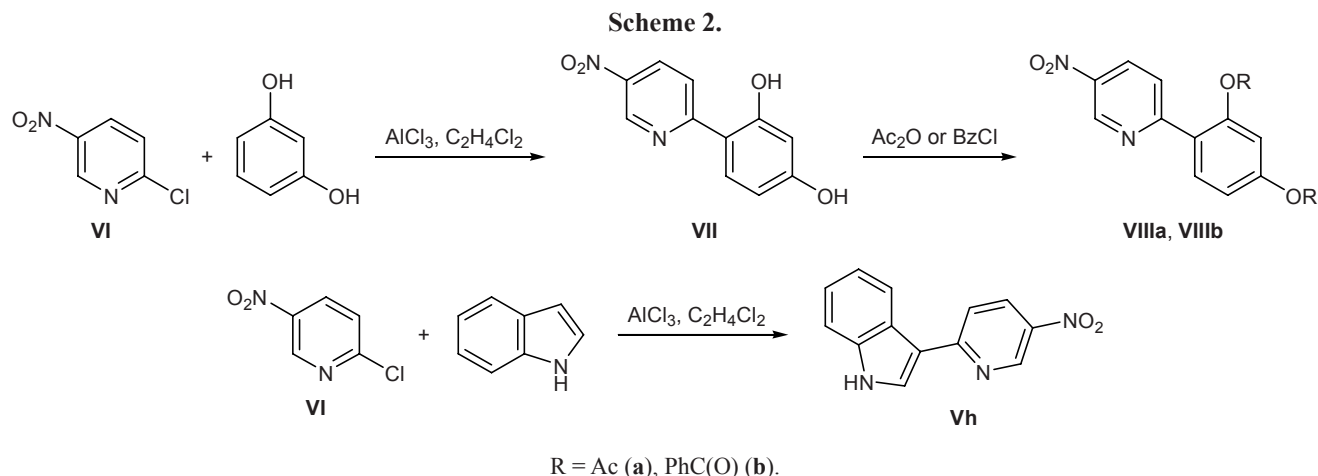
3,5-Dinitropyridin-2(1*H*)-one (**I**) reacted with cyclopropyl methyl ketone (**IVa**) [5] in the presence of ammonia to give 2-cyclopropyl-5-nitropyridine (**Va**) in 85% yield. Presumably, this reaction provides the simplest way of introducing a cyclopropyl substituent into the pyridine ring; the resulting structure attracts interest from the viewpoint of its synthetic potential [6–8]. In the reaction of compound **I** with 3-hydroxyimino-butan-2-one, the yield of 2-acetyl-5-nitropyridine oxime (**Vb**) was as poor as 19%, whereas only traces of 2-acetyl-5-nitropyridine (**Vc**) were formed from dinitropyridinone **I** and biacetyl.

Scheme 1.



II, III, n = 1 (a), 3 (b), 4 (c), 5 (d), 6 (e), 7 (f); IV, V, R = *i*-Pr (a), HON=C(Me) (b), Ac (c), 3-MeOC₆H₄ (d), 2-naphthyl (e), 5-methylfuran-2-yl (f), 1*H*-pyrrol-2-yl (g), 1*H*-indol-3-yl (h).

* For communication V, see [1].



The described reaction may be used to synthesize 2-aryl-substituted 5-nitropyridines, in particular compounds **Vd** and **Ve** from 3-methoxyacetophenone and 1-(2-naphthyl)ethanone, respectively. In the reactions of **I** with heterocyclic ketones (2-acetyl-5-methylfuran, 2-acetyl-1*H*-pyrrole, and 3-acetyl-1*H*-indole), only 2-(5-methylfuran-2-yl)-5-nitropyridine (**Vf**) was obtained in a satisfactory yield, which is consistent with low reactivity of the acetyl group in 2-acetylpyrrole and 3-acetylindole.

An alternative method for the synthesis of difficultly accessible 2-aryl- and 2-hetaryl-3(5)-nitropyridines is based on hetarylation of the corresponding aromatic and heteroaromatic compounds with 2-chloro-5-nitropyridine (**VI**) in the presence of aluminum chloride as catalyst [9] (Scheme 2).

The reaction of indole with 2-chloro-5-nitropyridine in the presence of an equimolar amount of AlCl_3 gave 3-(5-nitropyridin-2-yl)-1*H*-indole (**Vh**) in 20% yield. The corresponding 2-aryl-5-nitropyridine **VII** was formed in 65% yield in analogous reaction of **VI** with resorcinol. Acylation of both hydroxy groups in **VII** with acetyl or benzoyl chloride resulted in the formation of diesters **VIIIa** and **VIIIb** (Scheme 2).

Nitropyridines **V** and **VIII** are key intermediate products in the synthesis of metacyclophanes with different lengths of the carbon chain, as well as of indoles [2, 10, 11]. Their structure was determined on the basis of analytical and spectral data.

EXPERIMENTAL

The IR spectra were recorded on a Specord 75IR spectrometer from solutions in CDCl_3 . The ^1H NMR spectra were measured on a Bruker AC-200 instrument

at 200.13 MHz using tetramethylsilane as internal reference. The mass spectra of compounds **IIIc**, **Vh**, **VIIIa**, and **VIIIb** were obtained on an Agilent 5973N mass spectrometer (electron impact, 70 eV; vaporizer temperature 230–250°C). The mass spectra of the other compounds were recorded on a Finnigan MAT-8200 high-resolution mass spectrometer (electron impact, 70 eV; vaporizer temperature 270–300°C). The molecular weights and elemental compositions of compounds **Va**, **Vd**, and **Vg** were determined from the high-resolution mass spectra. The elemental compositions of the other compounds were determined using a Perkin–Elmer analyzer. The progress of reactions and the purity of products were monitored by thin-layer chromatography on Silufol UV-254 plates (spots were visualized under UV light).

1-Methyl-3,5-dinitropyridin-2(1*H*)-one (**I**) was synthesized according to the procedures described in [12, 13]. Cyclooctanone (**IIb**), cyclononanone (**IIc**), cyclododecanone (**IIf**), 2-acetylnaphthalene (**IVe**), 3-methoxyacetophenone (**IVd**), and 2-acetyl-1*H*-pyrrole (**IVg**) were commercial products (Fluka) and were used without additional purification. Cyclodecanone (**IIc**) and cycloundecanone (**IIe**) were synthesized as described in [14]. Cyclopropyl methyl ketone (**Iva**) was prepared in two steps from γ -butyrolactone, following the procedure reported in [15]. 2-Acetyl-5-methylfuran (**IVf**) [16, 17], 3-acetyl-1*H*-indole (**IVh**) [18], 3-hydroxyiminobutan-2-one (**IVb**) [19], and 2-chloro-5-nitropyridine (**VI**) [20] were prepared by known methods.

Nitropyridines IIIb–IIIf (general procedure). A mixture of 0.395 g (2 mmol) of 3,5-dinitropyridin-2(1*H*)-one (**I**) and 4 mmol of cyclic ketone **IIb–IIf** in 40 ml of methanol saturated with ammonia was heated

for 3 h at 70°C in a flask equipped with a reflux condenser. The solvent was distilled off under reduced pressure, and the oily residue was purified by column chromatography on silica gel (100–160 μm) using benzene as eluent.

3-Nitro-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine (IIIb). Yield 65%, colorless crystals, mp 45–46°C (from hexane) [21].

3-Nitro-6,7,8,9,10,11-hexahydro-5*H*-cyclonona[*b*]pyridine (IIIc). Yield 64%, mp 14–15°C. IR spectrum, ν , cm^{-1} : 1541, 1324 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.24–1.48 m (6H, 7-H, 8-H, 9-H), 1.79–1.97 m (4H, 6-H, 10-H), 2.91 t (2H, 11-H, $J = 6.11$ Hz), 3.08 t (2H, 5-H, $J = 6.35$ Hz), 8.19 d (1H, 4-H, $J = 2.69$ Hz), 9.21 d (1H, 2-H, $J = 2.44$ Hz). Mass spectrum, m/z (I_{rel} , %): 220 (15.97) [M] $^+$, 205 (19.78), 192 (19.51), 191 (15.36), 179 (20.80), 178 (16.94), 177 (100), 165 (13.54), 164 (13.83), 163 (14.05), 152 (23.23), 131 (13.30), 77 (11.00). Found, %: C 65.53; H 7.34; N 12.68. $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$. Calculated, %: C 65.43; H 7.32; N 12.72.

3-Nitro-5,6,7,8,9,10,11,12-octahydrocyclodeca[*b*]pyridine (III d). Yield 54%, mp 77–78°C (from aqueous ethanol). IR spectrum, ν , cm^{-1} : 1541, 1338 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.01–1.23 m (4H, 8-H, 9-H), 1.49–1.57 m (4H, 7-H, 10-H), 1.82–2.10 m (4H, 6-H, 11-H), 2.98 t (2H, 12-H, $J = 6.59$ Hz), 3.11 t (2H, 5-H, $J = 6.59$ Hz), 8.25 d (1H, 4-H, $J = 2.68$ Hz), 9.24 d (1H, 2-H, $J = 2.44$ Hz). Mass spectrum, m/z (I_{rel} , %): 234 (17.20) [M] $^+$, 219 (16.60), 205 (16.51), 192 (14.54), 191 (100), 179 (11.04), 178 (15.52), 177 (42.47), 165 (16.93), 152 (54.05), 145 (13.04), 131 (13.38), 117 (11.23), 91 (10.44), 77 (17.65), 65 (11.58), 55 (11.21), 41 (23.47), 39 (14.99). Found, %: C 66.92; H 7.88; N 11.87. $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_2$. Calculated, %: C 66.64; H 7.74; N 11.96.

3-Nitro-6,7,8,9,10,11,12,13-octahydro-5*H*-cycloundeca[*b*]pyridine (IIIe). Yield 51%, mp 68–69°C (from aqueous ethanol). IR spectrum, ν , cm^{-1} : 1541, 1324 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 1.09–1.48 m (10H, 7-H, 8-H, 9-H, 10-H, 11-H), 1.75–1.92 m (2H, 6-H), 1.93–2.07 m (2H, 12-H), 2.91 t (2H, 13-H, $J = 6.35$ Hz), 3.02 t (2H, 5-H, $J = 6.35$ Hz), 8.25 d (1H, 4-H, $J = 2.44$ Hz), 9.21 d (1H, 2-H, $J = 2.44$ Hz). Mass spectrum, m/z (I_{rel} , %): 248 (26.09) [M] $^+$, 233 (13.97), 207 (17.35), 206 (12.38), 205 (82.46), 192 (18.88), 191 (100), 179 (20.21), 178 (13.62), 177 (25.10), 165 (30.20), 163 (13.68), 152 (70.65), 145 (11.41), 133 (10.45), 131 (12.26), 117 (11.40), 106 (10.39), 77 (16.11), 65 (11.19), 55 (13.96),

43 (16.36), 41 (27.94), 39 (13.15), 29 (14.82), 28 (20.28), 27 (11.26). Found, %: C 67.75; H 7.95; N 10.64. $\text{C}_{14}\text{H}_{20}\text{N}_2\text{O}_2$. Calculated, %: C 67.71; H 8.12; N 11.28.

3-Nitro-5,6,7,8,9,10,11,12,13,14-decahydrocyclododeca[*b*]pyridine (III f). Yield 45%, mp 83–84°C (from ethanol) [2].

Nitropyridines Va–Vh (general procedure). A mixture of 0.395 g (2 mmol) of dinitropyridinone **I** and 4 mmol of ketone **IVa–IVh** in 40 ml of methanol saturated with ammonia was heated for 3 h at 100°C in a sealed ampule. The ampule was cooled and opened, the solvent was distilled off under reduced pressure, and the oily residue was purified by column chromatography on silica gel (100–160 μm) using benzene as eluent.

2-Cyclopropyl-5-nitropyridine (Va). Yield 85%, mp 63–64°C (from hexane). IR spectrum, ν , cm^{-1} : 1554, 1324 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 2.09–2.23 m (1H, CH), 1.13–1.26 m (4H, CH_2), 7.33 d (1H, 3-H, $^3J = 8.55$ Hz), 8.31 d.d (1H, 4-H, $^3J = 8.65$, $^4J = 2.44$ Hz), 9.25 d (1H, 6-H, $^4J = 2.18$ Hz). Mass spectrum, m/z (I_{rel} , %): 164 (29.59) [M] $^+$, 163 (100), 118 (16.48), 117 (47.84), 91 (20.83), 65 (17.41), 39 (12.62). Found: m/z 164.0583 [M] $^+$. $\text{C}_8\text{H}_8\text{N}_2\text{O}_2$. Calculated: M 164.0586.

1-(5-Nitropyridin-2-yl)ethanone oxime (Vb). Yield 19%, mp 161–162°C (from benzene) [22].

1-(5-Nitropyridin-2-yl)ethanone (Vc). Yield 2%, mp 88–89°C (from cyclohexane) [22].

2-(3-Methoxyphenyl)-5-nitropyridine (Vd). Yield 93%, light yellow crystals, mp 105–106°C (from ethanol). IR spectrum, ν , cm^{-1} : 1541, 1324 (NO_2), 1190 (OCH_3). ^1H NMR spectrum (CDCl_3), δ , ppm: 3.91 s (3H, OCH_3), 7.07 d (1H, 4'-H, $^3J = 7.81$ Hz), 7.38–7.48 t (1H, 5'-H, $^3J = 7.81$, 8.04 Hz), 7.60–7.68 m (2H, 2'-H, 6'-H), 7.89 d (1H, 3-H, $^3J = 8.80$ Hz), 8.51 d.d (1H, 4-H, $^3J = 8.80$, $^4J = 2.68$ Hz), 9.45 d (1H, 6-H, $^4J = 2.20$ Hz). Mass spectrum, m/z (I_{rel} , %): 231 (14.97), 230 (100) [M] $^+$, 229 (78.71), 201 (13.78), 200 (29.27), 183 (15.55), 157 (14.75), 155 (10.15), 154 (22.35), 142 (10.84), 141 (10.40), 128 (10.72), 127 (20.42), 114 (16.53), 92 (15.97), 77 (22.71), 64 (11.53), 63 (10.61). Found: m/z 230.0692 [M] $^+$. $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$. Calculated: M 230.0691.

2-(2-Naphthyl)-5-nitropyridine (Ve). Yield 73%, mp 171–172°C (from propan-2-ol). IR spectrum, ν , cm^{-1} : 1554, 1338 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 7.50–7.61 m (2H, 6'-H, 7'-H), 7.85–8.04 m

(4H, 3'-H, 4'-H, 5'-H, 8'-H), 8.15 d.d (1H, 3-H, $^3J = 8.79$, $^4J = 1.71$ Hz), 8.51 d.d (1H, 4-H, $^3J = 9.23$, $^4J = 2.44$ Hz), 8.58 s (1H, 1'-H), 9.51 d (1H, 6-H, $^4J = 2.45$ Hz). Mass spectrum, m/z (I_{rel} , %): 251 (16.30), 250 (100) $[M]^+$, 204 (22.76), 177 (34.24), 176 (17.07), 127 (29.06), 88 (13.84). Found, %: C 71.77; H 3.64; N 10.37. $C_{15}H_{10}N_2O_2$. Calculated, %: C 71.99; H 4.03; N 11.19.

2-(5-Methylfuran-2-yl)-5-nitropyridine (Vf). Yield 68%, light yellow crystals, mp 130–131°C (from ethanol). IR spectrum, ν , cm^{-1} : 1527, 1324 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 2.45 s (3H, CH_3), 6.23 d (1H, 4'-H, $^3J = 2.69$ Hz), 7.20 d (1H, 3'-H, $^3J = 3.42$ Hz), 7.73 d (1H, 3-H, $^3J = 8.80$ Hz), 8.44 d.d (1H, 4-H, $^3J = 8.90$, $^4J = 2.44$ Hz), 9.36 d (1H, 6-H, $^4J = 2.44$ Hz). Mass spectrum, m/z (I_{rel} , %): 205 (11.36), 204 (100) $[M]^+$, 158 (10.86), 131 (52.72), 119 (10.74), 103 (11.17), 77 (14.91), 53 (10.05), 43 (24.90), 28 (10.88). Found, %: C 58.84; H 3.63; N 13.01. $C_{10}H_8N_2O_3$. Calculated, %: C 58.82; H 3.95; N 13.72.

5-Nitro-2-(1H-pyrrol-2-yl)pyridine (Vg). Reaction temperature 90°C. Yield 27%, orange crystals, mp 198°C (decomp., from ethanol). IR spectrum, ν , cm^{-1} : 3027 (NH); 1581, 1338 (NO_2). ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm: 6.27–6.29 m (1H, 4'-H), 7.08–7.10 m (2H, 3'-H, 5'-H), 7.89 d (1H, 3-H, $^3J = 9.03$ Hz), 8.48 d (1H, 4-H, $^3J = 8.79$ Hz), 9.25 s (1H, 6-H), 11.95 br.s (1H, NH). Mass spectrum, m/z (I_{rel} , %): 190 (10.38), 189 (100) $[M]^+$, 143 (24.27), 117 (11.66), 116 (66.64), 89 (17.36), 39 (11.93). Found: m/z 189.0537 $[M]^+$. $C_9H_7N_3O_2$. Calculated: M 189.0538.

3-(5-Nitropyridin-2-yl)-1H-indole (Vh). a. Following the general procedure, compound **Vh** was obtained in 23% yield as dark orange crystals with mp 173–174°C (from ethanol) [23, 24].

b. A mixture of 0.5 g (3.15 mmol) of 2-chloro-5-nitropyridine (**VI**) and 0.417 g (3.15 mmol) of AlCl_3 in 25 ml of anhydrous dichloroethane was stirred for 30 min at room temperature and cooled to 0°C, 0.369 g (3.15 mmol) of indole was added, and the mixture was stirred for 1 h at 0°C, heated for 24 h at 80°C, poured onto ice, and extracted with dichloroethane. The extract was washed with water, dried over anhydrous magnesium sulfate, and evaporated, and the residue was purified by column chromatography on silica gel (100–160 μm) using benzene–ethyl acetate (9:1) as eluent. Yield 20%, dark orange crystals, mp 173–174°C (from ethanol) [23, 24].

4-(5-Nitropyridin-2-yl)benzene-1,3-diyl diacetate (VIIIa). 4-(5-Nitropyridin-2-yl)benzene-1,3-diol (**VII**)

was synthesized from compound **VI** and resorcinol according to the procedure described in [9]. A mixture of 0.464 g (2 mmol) of compound **VII** and 3.5 ml of acetic anhydride was heated under stirring to 60°C, a catalytic amount of concentrated sulfuric acid was added, and the mixture was stirred for 2 h at that temperature. The mixture was then poured onto ice, and the precipitate was filtered off. Yield 70%, mp 142–143°C (from ethanol). IR spectrum, ν , cm^{-1} : 1748 ($\text{C}=\text{O}$); 1511, 1349 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 2.23 s (3H, 1- COOCH_3), 2.33 s (3H, 3- COOCH_3), 7.07 d (1H, 2-H, $^4J = 2.20$ Hz), 8.18 d.d (1H, 6-H, $^3J = 8.67$, $^4J = 2.20$ Hz), 7.75–7.85 m (2H, 5-H, 3'-H), 8.53 d.d (1H, 4'-H, $^3J = 8.78$, $^4J = 2.68$ Hz), 9.49 d (1H, 6'-H, $^4J = 2.42$ Hz). Mass spectrum, m/z (I_{rel} , %): 316 (0.22) $[M]^+$, 274 (24.65), 233 (12.44), 232 (100), 186 (12.53). Found, %: C 56.91; H 3.72; N 8.72. $C_{15}H_{12}N_2O_6$. Calculated, %: C 56.96; H 3.82; N 8.86.

4-(5-Nitropyridin-2-yl)benzene-1,3-diyl dibenzoate (VIIIb). Benzoyl chloride, 0.7 ml (4 mmol), was added dropwise under stirring over a period of 30 min to a solution of 0.464 g (2 mmol) of compound **VII** in 4.5 ml of anhydrous pyridine, cooled to 0°C. The mixture was stirred for 3 days and poured onto ice, and the precipitate was filtered off and washed with ice water. Yield 70%, mp 173–174°C (from toluene). IR spectrum, ν , cm^{-1} : 1747 ($\text{C}=\text{O}$); 1507, 1347 (NO_2). ^1H NMR spectrum (CDCl_3), δ , ppm: 7.31–7.72 m (8H, *m*-H, *p*-H, 2-H, 6-H), 7.84 d (1H, 3'-H, $^3J = 8.78$ Hz), 7.98 d (1H, 5-H, $^3J = 8.48$ Hz), 8.12–8.15 m (2H, *o*-H), 8.17–8.25 m (2H, *o*-H), 8.42 d.d (1H, 4'-H, $^3J = 8.78$, $^4J = 2.44$ Hz), 9.40 d (1H, 6'-H, $^4J = 2.44$ Hz). Mass spectrum, m/z (I_{rel} , %): 440 (0.74) $[M]^+$, 105 (100), 77 (21.13). Found, %: C 68.47; H 3.71; N 6.25. $C_{25}H_{16}N_2O_6$. Calculated, %: C 68.18; H 3.66; N 6.36.

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