

Nitrogen Heterocycles from Trimethylbenzenes

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ABSTRACT: *The reaction between 1,2,4-trimethylbenzene 1, isobutyric aldehyde, and cyanoacetic ester afforded a mixture of three products: 3,3,5,6,8-pentamethyl-1,2,3,4-tetraisoquinolylidene-1-acetic acid ether (2) and its 3,3,5,6,7-pentamethyl-(3)- and 3,3,6,7,8-pentamethyl-1,2,3,4-tetraisoquinolylidene-1-acetic acid ether isomers (3) in ratio 1:5:1.*
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INTRODUCTION

The Ritter reaction in its original version involves electrophilic alkylation of nitriles with carbenium ions generated from olefins, or tertiary or secondary alcohols in the presence of acids, and results in N-substituted amides [1,2]. Several alternative methodologies were developed for the Ritter reaction [3,4], and the Ritter reaction was applied to the enan-

tiospecific synthesis of 3-aza-bicyclic compounds [5], *cis*-fused hexahydro-4a*H*-indeno[1,2-*b*]pyridine ring system [6], and six-five-six fused-ring structures [7]. Recently, we have reported that the reaction of activated arenes with nitriles and isobutyric aldehyde in concentrated sulfuric acid gives 3,3-dimethyl-3,4-dihydroisoquinoline derivatives [8,9].

Here we report the synthesis of nitrogen heterocycles derived from trimethylbenzenes in the reaction with isobutyric aldehyde and cyanoacetic ester.

RESULTS AND DISCUSSION

Preparation of Isoquinolylidene Derivatives

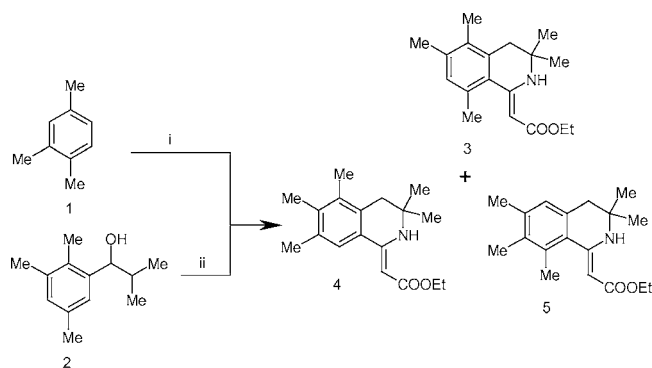
According to ¹H NMR, NOESY, MS, and TLC data, we found that the reaction between 1,2,4-trimethylbenzene (pseudocumol 1), isobutyric aldehyde, and cyanoacetic ester gives a mixture of three products, the structures of which were those of 3,3,5,6,8-pentamethyl-1,2,3,4-tetraisoquinolylidene-1-acetic acid ester (2) and its 3,3,5,6,7- and 3,3,6,7,8-isomers (3 and 4) in ratio 1:5:1 (according to integral intensity signals). The reaction involving preobtained carbinol 2 gave the same results (Scheme 1).

Compounds 4 and 5 were formed from 1,2,3-trimethylbenzene (6), isobutyric aldehyde, and cyanoacetic ester, as well as from carbinol 7 previously

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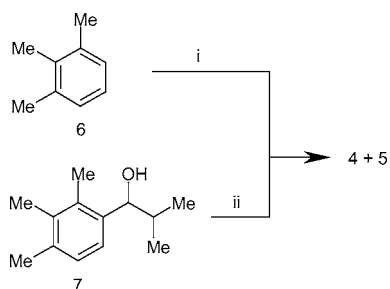
i: $(\text{CH}_3)_2\text{CHCHO}$, $\text{NCCH}_2\text{COOEt}$, H_2SO_4 , $5-10^\circ\text{C}$
 ii: $\text{NCCH}_2\text{COOEt}$, H_2SO_4 , $5-10^\circ\text{C}$, ratio **3:4:5** = 1:5:1

SCHEME 1

obtained from 1-bromo-2,3,4-trimethylbenzene and isobutyric aldehyde (**4:5** ratio = 5:1) in the case of the three-component synthesis (Scheme 2).

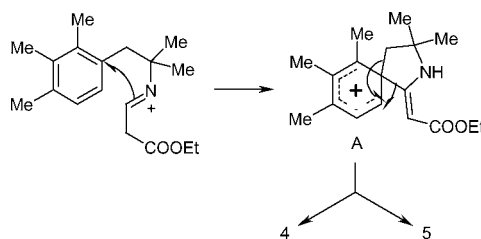
Formation of **4** and **5** from an arene **6** and carbinol **7** can be only due to the fact that the reaction proceeds via spiro-intermediate **A** that could be isomerized in two ways under the reaction conditions (Scheme 3).

Similar reactions were previously observed for benzoic acid phenethylamide and 2-(6'-methoxynaphthyl-2')-ethylamide cyclization based on the Bishler-Napieralski reaction [10] that is genetically related to the Ritter reaction as they both proceed via the same intermediate [11]. The reaction with pseudocumol **1** should probably proceed via spiro-intermediate **B** that must initially lead to isoquinolines **3** and **8**, with the latter being predominated. Further reaction course occurs via the Jacobs reaction [12] typical for tetra-alkyl-substituted benzenes in sulfuric acid. Since migration of alkyl substitute from EWG *ortho*-position to *para*-position was characteristic of the Jacobs reaction, isoquinoline **8** formed and protonated under the reaction condi-



i: $(\text{CH}_3)_2\text{CHCHO}$, $\text{NCCH}_2\text{COOEt}$, H_2SO_4 , $5-10^\circ\text{C}$
 ii: $\text{NCCH}_2\text{COOEt}$, H_2SO_4 , $5-10^\circ\text{C}$, ratio **4:5** = 5:1

SCHEME 2



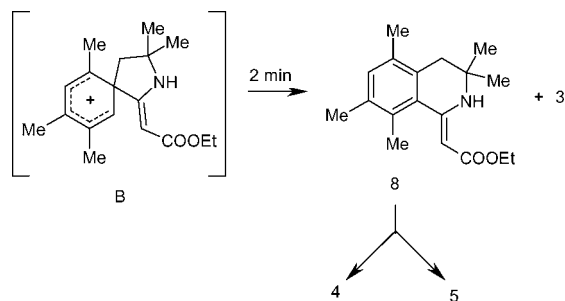
SCHEME 3

tions was probably exposed mainly to methyl group migration from position **8** to **6** allowing product **4**, and to a lesser extent to methyl group migration from position **5** to **6** allowing product **5**, as shown in Scheme 4.

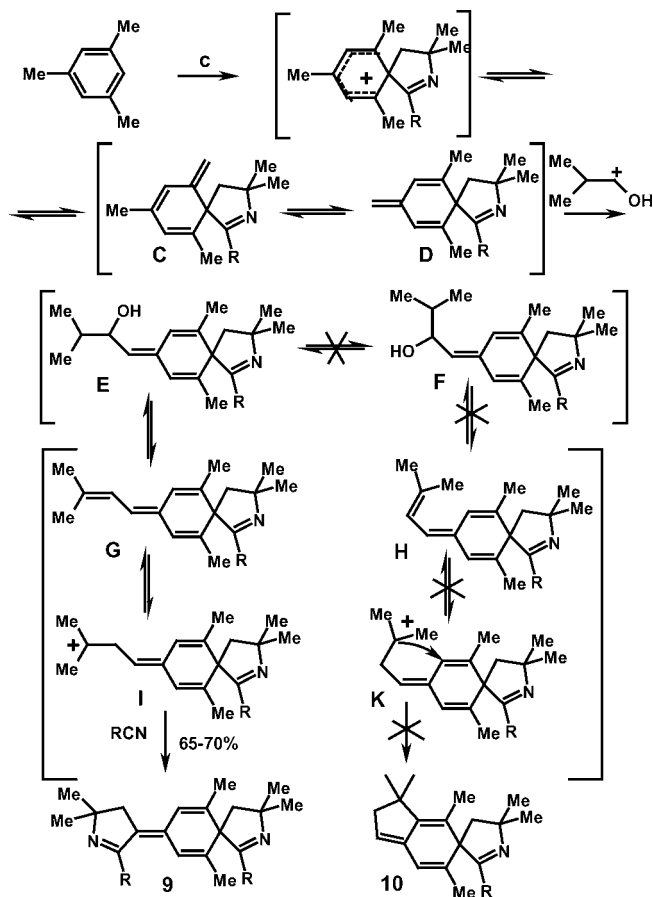
It is obvious that isoquinoline **3** having a methyl group at position 6 on the ring does not undergo any rearrangements. Based on the concept of a tandem three-component heterocyclization reaction via spiro-intermediates, it was of interest to use mesitylene in the reaction. Since **A**- and **B**-type intermediates may be presented as uncharged **C** and **D** structures, the reaction of these with a protonated isobutyric aldehyde seems most likely. That intermediate **C** was not found in the reaction products was probably due to the sterically hindered reaction owing to orthogonally positioned spirocycle [13].

Intermediate **D** may give **E** and **F** conformers that convert to *cis*-(**H**) and *trans*-(**G**)-conjugate tetraenes, respectively. When intermediate **H** is protonated, the formation of spiran **10** seems most likely because its reaction with nitrile is also hindered sterically. On the contrary, the reaction of *trans*-intermediate **G** with nitrile is preferential, and dispiroproduct **9** is the only reaction compound formed (Scheme 5).

Considering the fact that when mesitylene, isobutyric aldehyde, and nitriles (cyanoacetic ester; acetonitrile, methylrhodane) are used in equimolar ratios, and the cascade heterocyclization product **9**



SCHEME 4



Scheme 5. Reagents and conditions:
c) isobutyric aldehyde, RCN, sulfuric acid, 5–10°C
R = CHCOOEt Me SMe (9a–c)

SCHEME 5

is solely formed, one could suppose that the protonated isobutyric aldehyde attack on the intermediate **D** is not merely thermodynamically efficient, but also occurs with high enantioselectivity resulting in intermediates **E**, **G**, and **I** that afford ultimately the formation of the cascade heterocyclization product in total 65–70% yield for 10 stages [14].

¹H NMR spectrum of **9b** reveals duplications of all aliphatic proton signals. Thus, there are two triplets (δ = 1.12 and 1.17 ppm) of methyl groups and two quadruplets (3.85 and 4.01 ppm) of a methylene ring of two ethoxy groups which are difficult to assign. Two vinyl proton singlets at 3.83 and 4.78 ppm were assigned to spiropyrrolidine and pyrrolidene fragments, respectively. Vinyl proton signals of a cyclohexadiene ring are observed at 6.18 ppm (C7-H) and 6.73 ppm (C9-H). We failed to assign NH-proton signals, which were observed at 8.23 and 8.41 ppm, to an appropriate ring. Furthermore, two methyl group signals at C5' (1.24 and 1.25 ppm) and a singlet of two

methyl groups at C3 (1.43 ppm) were observed. The methyl groups were present at 1.86 ppm on the cyclohexadiene ring and observed as two singlets. Methylene proton signals at C4 were found at 2.00 ppm, while those at C4' was found at 2.65 ppm.

According to experimental data and discussion above, we demonstrated that the reaction of isomeric trimethylbenzenes with both an isobutyric aldehyde and nitriles, and the appropriate carbinols and nitriles, proceeds only via the formation of a five-member spiroheterocycle, the latter being either preserved unchanged or rearranged into 3,4-dihydroisoquinoline derivatives.

EXPERIMENTAL

Melting points were determined using a Reichert–Jung Thermovar apparatus and an Electrotherminal IA 9000 digital melting point apparatus and are uncorrected. IR spectra were measured in vaseline oil using a UR-20 spectrophotometer. ¹H NMR spectra were monitored using a Bruker AM-300 model (75.470 MHz) in DMSO-*d*₆ at 303°C K. NOESY spectral mixing time was 500 ms. The HBMBC experiment was optimized using KCCB 8 Hz. Mass spectra were measured using a Finnigan MAT apparatus under standard conditions (electron impact ionization, 70 eV). The reaction course and the purity of the formed product were monitored using a TLC method on Silufol UV-254 plates in chloroform–acetone (9:1); 0.5% chloranil solution in toluene was used as a developer.

Three-Component Synthesis of the **3** and **8** from 1,2,4-Trimethylbenzene

A mixture of pseudocumol (1.2 g, 10 mmol), isobutyric aldehyde (0.72 g, 10 mmol), and cyanoacetic ester (1.13 g, 10 mmol) was added, dropwise with stirring, to sulfuric acid (15 mL, 95%) at 5–10°C for 1 min. After 3–5 min. stirring the reaction mixture was poured on to 100 g of crushed ice and extracted with benzene (25 mL). The organic layer was separated, and the water layer was basified with ammonium carbonate up to pH 8–9. The resulting layer was extracted with chloroform (2 × 50 mL), dried over MgSO₄. After distilling off the solvent on a rotary evaporator, 2.3 g of the product mixture was obtained. Using column chromatography (triethanolamine-deactivated silica gel, 30/50 mesh; benzene–ester, 35:1), compound **3** (85% yield) was isolated with isoquinoline **4** (15%) as admixture, whereas compound **5** (80% yield) was obtained with isoquinoline **4** (20%) as admixture (according to ¹H NMR).

3,3,5,6,8-Pentamethyl-1,2,3,4-tetrahydroisoquinolylidene-1-acetic Acid Ether (**3**). ^1H NMR δ : 1.22 (s, 6H, *gem*-Me); 1.30 (t, 3H, OCH_2CH_3 , $J = 7.2$ Hz); 2.19 (s, 3H, 5'-CH₃); 2.32 (s, 3H, 6'-CH₃); 2.51 (s, 3H, 8'-CH₃); 2.78 (s, 2H, 4-CH₂); 4.20 (q, 2H, OCH_2CH_3 , $J = 7.2$ Hz); 4.81 (s, 1H, H_{vinyl}); 6.97 (s, 1H, 7-H_{aryl}); 9.11 (w.s., 1H, NH).

3,3,5,7,8-Pentamethyl-1,2,3,4-tetrahydroisoquinolylidene-1-acetic Acid Ether (**8**). mp 71–72°C (hexane). IR (ν/cm^{-1}): 3250; 2960; 2850; 1640; 1600; 1370. ^1H NMR (δ): 1.22 (s, 6H, *gem*-CH₃); 1.29 (t, 3H, OCH_2CH_3 , $J = 7.2$ Hz); 2.22 (s, 3H, 5'-CH₃); 2.26 (s, 3H, 7'-CH₃); 2.44 (s, 3H, 8'-CH₃); 2.67 (s, 2H, 4-CH₂); 4.15 (q, 2H, OCH_2CH_3 , $J = 7.2$ Hz); 4.76 (s, 1H, H_{vinyl}); 7.01 (s, 1H, H_{arom}); 9.12 (w.s., 1H, NH). It was found (%) C 75.19; H 8.72; N 4.93. $\text{C}_{18}\text{H}_{25}\text{NO}_2$. It was calculated (%) C 75.26; H 8.71; N 4.88.

Synthesis of **4** and **5** from 1,2,4-Trimethylbenzene

A mixture of 1,2,3-trimethylbenzene (1.2 g, 10 mmol), isobutyric aldehyde (0.72 g, 10 mmol), and cyanoacetic ester (1.13 g, 10 mmol) was added, dropwise with stirring, to sulfuric acid (15 mL, 95%) at 5–10°C for 3 min. After stirring for 15 min, the mixture was poured on to 100 g of crushed ice. The organic layer was extracted with 25 mL of benzene, and the water layer was basified with ammonium carbonate up to pH 8–9. The resulting layer was extracted with chloroform (2 \times 50 mL), dried over MgSO_4 . After removing the solvent, 2.3 g of the product mixture was obtained. Column chromatography (triethanolamine-deactivated silica gel, 30/50; benzene–ester, 35:1) afforded compound **4** in 52% yield (15 g, mp 83–84°C, hexane) and a mixture of **5** (80%) and **4** (20%) that were not separated into pure products (according to ^1H -NMR).

3,3,5,6,7-Pentamethyl-1,2,3,4-tetrahydroisoquinolylidene-1-acetic Acid Ether (**4**). IR (ν/cm^{-1}): 3260, 2960, 2850, 1630, 1600, 1380. ^1H NMR δ : 1.28 (s, 6H, *gem*-di-Me); 1.30 (t, 3H, OCH_2CH_3 , $J = 7.1$ Hz); 2.20 (s, 3H, 5'-Me); 2.23 (s, 3H, 6'-Me); 2.34 (s, 3H, 7'-Me); 2.76 (s, 2H, 4-CH₂); 4.16 (q, 2H, OCH_2CH_3 , $J = 7.1$ Hz); 5.12 (s, 1H, H_{vinyl}); 7.40 (s, 1H, 8-H); 9.00 (w.s., 1H, NH). ^{13}C -NMR δ : 14.8(CH_2CH_3); 15.4(5'-CH₃); 16.3 (6'-CH₃); 20.9 (7'-CH₃); 28.9 (*gem*-CH₃); 39.1 (4-CH₂); 49.0 (3-C); 58.4(CH_2CH_3); 77.0 (C_{vinyl}); 124.2 (8-C); 125.7 (8a-C); 131.2 (4a-C); 134.2 (5-C); 134.3 (6-C); 138.3(7-C); 156.5(1-C); 171.3(C=O). It was found (%) C 75.13; H 8.62; N 4.95. $\text{C}_{18}\text{H}_{25}\text{NO}_2$. It was calculated (%): C 75.26; H 8.71; N 4.88. Compound **5**: differential ^1H -NMR δ : 1.22 (s,

6H, *gem*-di-Me); 1.29 (t, 3H, OCH_2CH_3 , $J = 7.2$ Hz); 2.21 (s, 3H, 6'-Me); 2.30 (s, 3H, 7'-Me); 2.48 (s, 3H, 8'-Me); 4.15 (q, 2H, OCH_2CH_3 , $J = 7.2$ Hz); 4.77 (s, 1H, 4-CH₂); 6.81 (s, 1H, 5-H); 9.12 (w.s., 1H, NH).

Preparation of 2,3,4-Trimethylbromobenzene

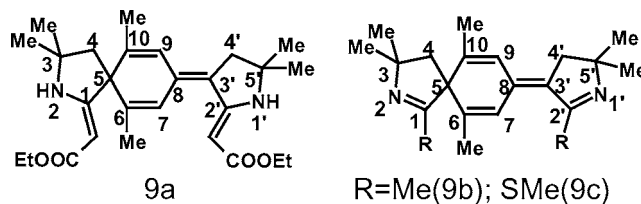
Water (100 mL) was added to 1,2,3-trimethylbenzene (60 g, 0.5 mol) in CCl_4 (400 mL) then bromine (80 g, 0.5 mol) was added, dropwise with stirring, to achieve slight staining of the solution. The reaction mixture was stirred for 30 min; the water layer was separated, and the organic layer was washed with water (2 \times 200 mL), next with a saturated sodium bicarbonate solution (200 mL) and finally with water to achieve pH 7. After removing the solvent on a rotary evaporator, alcohol (200 mL) and solid KOH (20 g) were added to the residue. The resultant layer was stirred for 20 min, heated for 30 min with the boiling solvent and poured into water (700 mL). This was further extracted with methyl-tert-butyl ester (2–300 mL) and dried over MgSO_4 . The solvent was removed on the water bath, and the residue was distilled in vacuum and collected in a fraction which was boiling away at 110–115°C at 15 mm. 2,3,4-trimethylbromobenzene was obtained in 65% yield (65 g). ^1H -NMR spectrum was similar to that described previously [6].

Preparation of 2-Methyl-1-(2',3',4'-trimethylphenyl)-propanol-1 (**7**)

Fresh-distilled isobutyric aldehyde (15 g, 0.21 mol) in 50 mL of dry THF was added, dropwise upon cooling with water and ice, to 0.2 mol of 2,3,4-trimethylphenylmagnesium bromide solution (prepared from 40 g (0.2 mol) of 2,3,4-trimethylbromobenzene and 5 g (0.21 mol) of magnesium in 15 g of dry THF). The reaction mixture was stirred with heating for 30 min, cooled, and dissolved with saturated NH_4Cl solution. The organic layer was separated, and the water layer was extracted with methyl-tert-butyl ether (2 \times 150 mL). Combined organic layers were washed with water, dried over MgSO_4 ; the solvent was removed on a water bath, and the residue was distilled at 5 mm and collected in a fraction which boiled away at 140–145°C. Product **7** was formed in 77% yield (~30 g) and used without being further purified. It was found (%) 81.33; H 10.61. $\text{C}_{13}\text{H}_{20}\text{O}$. It was calculated (%) C 81.20; H 10.48.

Reaction between Compound **7** and Cyanoacetic Ester

A mixture of **7** (1.92 g, 0.01 mol) and cyanoacetic ester (1.13 g, 0.01 mol) was added dropwise to



¹ H NMR δ, ppm.							
IR spectrum (v/cm ⁻¹)	3-(Me) ₂ , 6H, s	4-(CH) ₂ , 2H, s	6,10-(Me) ₂ , 6H, d	C ₇ H, C ₉ H two s.	4'-CH ₂ , 2H, (AB-system)	5'-(Me) ₂ , two s.	Others
9a 3355 (w.s.); 3335 (w.s.); 1735; 1723; 1650; 1600; 1580; 1500	1.43	2.00	1.86	6.18; 6.73	2.65	1.24 1.25	1.12 (3H, t, Me-ester.); 1.17 (3H, t, Me-ester.); 3.85 (2H, q, OCH ₂); 4.01 (2H, q, OCH ₂); 3.83 (1H, s, C ₁ -C _α H); 4.78 (1H, s, C ₂ -C _α H); 8.24 (1H, s, NH); 8.41 (1H, s, NH)
9b 1650; 1598; 1510	1.31	1.87	1.74	6.16; 6.81	2.55	1.15 1.16	1.58 (3H, s, 1-Me); 2.27 (3H, s, 2'-Me)
9c 1650; 1600; 1580	1.34	2.00	1.78	6.15; 7.09	2.68	1.19 1.20	2.31 (3H, s, 1-SMe); 2.42 (3H, s, 2'-SMe)

Mass-spectrometry data:

9a: (EI, 70 eV), m/z ($I_{\text{rel.}}(\%)$): 454 $[M]^+$ (100); 439 $[M-CH_3]^+$ (25); 409 $[M-OEt]^+$ (24); 393 $[M-OOEt]^+$ (23); 381 $[M-COOEt]^+$ (30); 341 $[M-NCCH_2COOEt]^+$ (98); 326 $[M-NCCH_2COOEt-Me]^+$ (97); 300 (90).

9b: (EI, 70 eV), m/z ($I_{\text{rel.}}(\%)$): 310 $[M]^+$ (5); 269 $[M-CH_3CN]^+$ (100); 254 $[M-CH_3CN-CH_3]^+$ (96); 231 $[M-CH_3CN-CH_3-CH_3CN]^+$ (25).

9c: (EI, 70 eV), m/z ($I_{\text{rel.}}(\%)$): 374 $[M]^+$ (5); 301 $[M-CH_3SCN]^+$ (100); 286 $[M-CH_3CN-CH_3]^+$ (47); 213 $[M-CH_3SCN-CH_3-CH_3SCN]^+$ (15).

FIGURE 1 Spectral characteristics of compounds **9 a–c**.

concentrated sulfuric acid (15 mL). The reaction mixture was treated following the procedure described for a three-component synthesis. The total yield and the **4:5** ratio was consistent with those obtained in the three-component synthesis.

Preparation of 8-[2'-R-5',5'-Dimethylpyrrolyl-3'-idene]-1-R-3,3,6,10-tetramethyl-2-azaspiro[4,5]-deca-1,6,9-trienes (**9a–c**)

A mixture of mesitylene (0.1 mol), isobutyric aldehyde (0.2 mol), and appropriate nitrile was added, dropwise upon cooling with water and ice, to concentrated sulfuric acid (50 mL). The mixture was stirred for 15 min and poured into water with ice (300 mL). The water layer was separated; the organic layer was treated with methyl-tert-butyl ether and 10% NaOH, washed with water and dried, then the solvent was removed on a rotary evaporator. The residue was crys-

tallized from the appropriate solvent. Compound **9a** was obtained in 67% yield (mp 143–144°C, hexane) (see Fig. 1). It was found (%) C 67.50; H 8.10; N 7.60; S 9.41. C₂₁H₃₀N₂S. It was calculated (%): C 67.38; H 8.02; N 7.49; S 9.36. Compound **9b** was obtained in 65% yield (mp 107–108°C, hexane) in a similar way. It was found (%) C 81.12; H 9.70; N 9.18. C₂₁H₃₀N₂. It was calculated (%): C 81.29; H 9.68; N 9.03. Compound **9c** was obtained in 71% yield (mp 190–191°C, alcohol). It was found (%) C 71.46; H 8.33; N 6.23. C₂₇H₃₈N₂O₄. It was calculated (%): C 71.37; H 8.37; N 6.23.

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