Synthesis of 2,3-Diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-ol Derivatives by the Reaction of Aryl(3-isocyanopyridin-4-yl)methanones with Aryl *Grignard* Reagents

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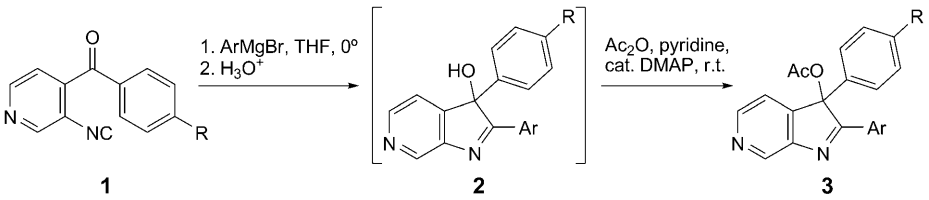
The reaction of aryl(3-isocyanopyridin-4-yl)methanones **1**, easily prepared from commercially available pyridin-3-amine, with aryl *Grignard* reagents gave, after aqueous workup, 2,3-diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-ols **2**. These rather unstable alcohols were *O*-acylated with Ac_2O in pyridine in the presence of a catalytic amount of 4-(dimethylamino)pyridine (DMAP) to afford the corresponding 2,3-diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl acetates **3** in relatively good yields.

Introduction. – As part of our program to explore the potential of the reactions of *ortho*-functionalized phenyl isocyanides with organometals for heterocycle synthesis [1], we have recently demonstrated that 2-isocyanophenyl ketones can be converted into 2,3-disubstituted 3*H*-indol-3-ols in generally good yields on treatment with *Grignard* reagents [1e]. As an extension of this synthesis, we envisioned that a synthesis of 2,3-disubstituted 3*H*-pyrrolo[2,3-*c*]pyridin-3-ols could be accomplished by reacting 3-isocyanopyridin-4-yl ketones with *Grignard* reagents. Here, we report the results of such a study. The investigation has indicated that the reactions of aryl(3-isocyanopyridin-4-yl)methanones **1** with aryl *Grignard* reagents provide a convenient synthetic approach to 2,3-diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-ol derivatives **3**. Due to instability of these alcohols during purification procedures, they were isolated as *O*-acetylated derivatives. There have been so far no reports on the synthesis of this heterocyclic skeleton, 3*H*-pyrrolo[2,3-*c*]pyridin-3-ol.

Results and Discussion. – The synthesis of 2,3-diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl acetates **3** from aryl(3-isocyanopyridin-4-yl)methanones **1** was performed as depicted in the *Scheme*. The starting isocyano ketones **1** could be easily prepared from commercially available pyridin-3-amine in a five-step sequence according to the procedure reported previously by us [2]. First, (3-isocyanopyridin-4-yl)(phenyl)methanone (**1a**) was chosen as a representative substrate and allowed to react with PhMgBr. The reaction was carried out in THF at 0°. The conditions were essentially the same as reported previously for preparing 2,3-disubstituted 3*H*-indol-3-ols from 2-isocyanophenyl ketones with *Grignard* reagents [1e]. The preferential initial addition of the phenyl anion to the isocyano C-atom occurred almost exclusively, and the subsequent cyclization by the intramolecular attack of the resulting imidoyl anion on

the CO C-atom proceeded immediately and cleanly to give, after aqueous workup, 2,3-diphenylpyrrolo[2,3-*b*]pyridin-3-ol (**2a**) in an almost quantitative yield, as judged by ^{1}H-NMR spectra of the concentrated extracts. However, we found that this product was quite unstable under purification procedures. Our attempts to isolate **2a** by chromatography on silica gel or alumina, or recrystallization were all unsuccessful. Therefore, the crude **2a** was quickly subjected to *O*-acylation. Thus, exposure of the crude product to Ac_2O in pyridine in the presence of a catalytic amount of 4-(dimethylamino)pyridine (DMAP) at room temperature afforded 2,3-diphenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl acetate (**3a**) in an excellent yield (93%).

Scheme



With this promising result in hand, the generality of this reaction was then investigated. Using two other aryl(3-isocyanopyridin-4-yl)methanones, **1b** and **1c**, and six arylmagnesium bromides, we prepared nine other 2,3-diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl acetates, **3b**–**3j**, in yields ranging from 73 to 95% as compiled in the *Table*. These data indicate that somewhat decreased yields of the corresponding desired products **3e** and **3f** using **1b** (*Entries 5* and *6*) were obtained, compared with those using the other starting materials **1a** and **1c**. The lower yields of these products might be explained by the Cl substituents on the benzene nucleus, which may cause the initial addition of *Grignard* reagent to the CO C-atom to some extent. It should be noted that the use of alkyl *Grignard* reagents, such as MeMgBr and EtMgBr, under the reaction conditions described above, resulted in the formation of complicated mixtures of products, probably including those arising from initial addition of *Grignard* reagents to

Table. *Preparation of 3*H*-Pyrrolo[2,3-*c*]pyridin-3-ol Derivatives* **3**

Entry	**1**	R	Ar in ArMgBr	**3**	Yield[a]) [%]
1	**1a**	H	Ph	**3a**	93
2	**1a**	H	3-Cl–C_6H_4	**3b**	86
3	**1a**	H	3-MeO–C_6H_4	**3c**	86
4	**1a**	H	3,4-$(MeO)_2$–C_6H_3	**3d**	95
5	**1b**	Cl	Ph	**3e**	73
6	**1b**	Cl	4-Cl–C_6H_4	**3f**	73
7	**1c**	MeO	Ph	**3g**	87
8	**1c**	MeO	4-Me–C_6H_4	**3h**	89
9	**1c**	MeO	4-Cl–C_6H_4	**3i**	95
10	**1c**	MeO	4-MeO–C_6H_4	**3j**	93

[a]) Yields of isolated products based on **1**.

the CO group of **1**. No desired products could be isolated after acetylation under the above mentioned conditions.

Finally, we planed to explore the reactions of alkyl(3-isocyanopyridin-4-yl)methanones with *Grignard* reagents. Unfortunately, however, such a ketone, *i.e.*, 1-(3-isocyanopyridin-4-yl)propan-1-one, could not be prepared in satisfactory purity and yield, and we could not carry out this exploration.

In conclusion, the presented results indicate that the reaction of aryl(3-isocyanopyridin-4-yl)methanones **1** with arylmagnesium bromides provides an efficient method for the preparation of 2,3-diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-ol derivatives **3** under mild reaction conditions. This method should be of use in synthesizing this class of heterocycles because of the simple manipulations and the ready availability of the starting materials.

Experimental Part

General. (3-Isocyanopyridin-4-yl)(4-methoxyphenyl)methanones **1a** and **1c** were prepared from 2,2-dimethyl-*N*-(pyridin-3-yl)propanamide [3] by the procedure reported previously by us [2]. All other chemicals used in this study were commercially available. All of the org. solvents used in this study were dried over appropriate drying agents and distilled prior to use. TLC: *Kieselgel 60 PF_{254}* (*Merck*). Column chromatography (CC): *Wako gel C-200E.* M.p.: *Laboratory Devices MEL-TEMP II*; uncorrected. IR Spectra: *Shimadzu FTIR-8300* spectrometer; $\tilde{\nu}$ in cm^{-1}. ^1H- and ^{13}C-NMR Spectra: *JEOL ECP500* FT-NMR spectrometer at 500 and 125 MHz, resp.; ^1H-NMR in $CDCl_3$; δ in ppm rel. to TMS as internal standard, *J* in Hz. LR-EI-MS: *JEOL JMS AX505 HA* spectrometer at 70 eV; in *m*/*z* (rel. %).

(4-Chlorophenyl)(3-isocyanopyridin-4-yl)methanone (**1b**) was prepared from 2,2-dimethyl-*N*-(pyridin-3-yl)propanamide [3], *via* the following precursors, under the conditions reported previously by us for the preparation of **1a** and **1c** [2].

N-*[4-(4-Chlorobenzoyl)pyridin-3-yl]-2,2-dimethylpropanamide*. Yield 59%. Yellow solid. M.p. 141–143° (hexane/Et_2O). IR (KBr): 3358, 1668, 1655. ^1H-NMR: 1.34 (*s*, 9 H); 7.33 (*d*, *J* = 5.0, 1 H); 7.52 (*AA′BB′*, *J* = 8.2, 2 H); 7.71 (*AA′BB′*, *J* = 8.2, 2 H); 8.47 (*d*, *J* = 5.0, 1 H); 9.90 (*s*, 1 H); 10.29 (br. *s*, 1 H). Anal. calc. for $C_{17}H_{17}ClN_2O_2$ (316.10): C 64.46, H 5.41, N 8.84; found: C 64.45, H 5.53, N 8.63.

(3-Aminopyridin-4-yl)(4-chlorophenyl)methanone. Yield: 79%. Yellow solid. M.p. 134–136° (hexane/THF). IR (KBr): 3447, 3306, 1633, 1607. ^1H-NMR: 5.81 (br. *s*, 2 H); 7.19 (*d*, *J* = 5.5, 1 H); 7.48 (*AA′BB′*, *J* = 8.7, 2 H); 7.64 (*AA′BB′*, *J* = 8.7, 2 H); 7.95 (*d*, *J* = 5.5, 1 H); 8.30 (*s*, 1 H). Anal. calc. for $C_{12}H_9ClN_2O$ (232.04): C 61.95, H 3.90, N 12.04; found: C 62.04, H 4.04, N 11.83.

N-*[4-(4-Chlorobenzoyl)pyridin-3-yl]formamide.* Yield 83%. Yellow oil. R_f (THF/hexane 1:1) 0.32. IR (neat): 3308, 1699, 1668. ^1H-NMR: 7.35 (*d*, *J* = 5.0, 1 H); 7.52 (*AA′BB′*, *J* = 8.7, 2 H); 7.73 (*AA′BB′*, *J* = 8.7, 2 H); 8.47–9.88 (*m*, 4 H). Anal. calc. for $C_{13}H_9ClN_2O_2$ (260.04): C 59.90, H 3.48, N 10.75; found: C 60.03, H 3.52, N 10.51.

Compound **1b**. Yield 79%. Yellow solid. M.p. 110–114° (hexane/Et_2O). IR (KBr): 2131, 1668. ^1H-NMR: 7.41 (*d*, *J* = 5.0, 1 H); 7.52 (*AA′BB′*, *J* = 8.7, 2 H); 7.75 (*AA′BB′*, *J* = 8.7, 2 H); 8.80 (*d*, *J* = 5.0, 1 H); 8.83 (*s*, 1 H). Anal. calc. for $C_{13}H_7ClN_2O$ (242.02): C 64.34, H 2.91, N 11.54; found: C 64.47, H 2.87, N 11.45.

*2,3-Diaryl-3*H*-pyrrolo[2,3-*c*]pyridin-3-ol Derivatives* **3** (*General Procedure*). To a stirred soln. of **1** (1.0 mmol) in THF (3 ml) at 0° was added one of the *Grignard* reagents (Et_2O soln.; 1.0 mmol). After 15 min, sat. aq. NH_4Cl (15 ml) was added, and the mixture was extracted with Et_2O (3 × 10 ml). The combined extracts were washed with brine, dried (anh. Na_2SO_4), and concentrated by evaporation. The residue was dissolved in pyridine (3 ml), and to this soln. Ac_2O (0.31 g, 3.0 mmol) and 4-(dimethylamino)pyridine (12 mg, 0.10 mmol) were added. The resulting mixture was then allowed to stir at r.t. overnight. Pyridine and excess Ac_2O were removed under reduced pressure to give a residue, which was purified by CC (SiO_2; AcOEt/hexane 1:1) to give the desired product.

*2,3-Diphenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3a**). White solid. M.p. 155–156° (hexane/CH_2Cl_2). IR (KBr): 1751. ^{1}H-NMR: 2.14 (*s*, 3 H); 7.08 (*dd*, *J* = 5.0, 0.9, 1 H); 7.26–7.33 (*m*, 3 H); 7.35–7.38 (*m*, 4 H); 7.41 (*tt*, *J* = 7.3, 1.4, 1 H); 7.96 (*dd*, *J* = 7.8, 1.4, 2 H); 8.43 (*d*, *J* = 5.0, 1 H), 8.98 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.92; 90.27; 116.39; 123.94; 128.69; 128.71; 128.78; 129.35; 130.42; 131.79; 135.26; 142.78; 148.09; 149.03; 149.25; 167.70; 177.69. MS: 328 (39, M^+), 285 (100). Anal. calc. for $C_{21}H_{16}N_2O_2$ (328.12): C 76.81, H 4.91, N 8.53; found: C 76.60, H 4.90, N 8.43.

*2-(3-Chlorophenyl)-3-phenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3b**). Pale-yellow solid. M.p. 185–187° (hexane/CH_2Cl_2). IR (KBr): 1748. ^{1}H-NMR: 2.15 (*s*, 3 H); 7.08 (*dd*, *J* = 5.5, 0.9, 1 H); 7.25–7.36 (*m*, 6 H); 7.39 (*dt*, *J* = 7.8, 0.9, 1 H); 7.73 (*dd*, *J* = 7.8, 0.9, 1 H); 8.06 (*t*, *J* = 0.9, 1 H); 8.45 (*d*, *J* = 5.5, 1 H); 8.99 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.91; 90.15; 116.44; 123.90; 126.65; 128.48; 128.99; 129.47; 129.91; 131.73; 132.16; 134.66; 134.92; 143.02; 148.49; 148.97; 149.00; 167.74; 176.42. MS: 362 (19, M^+), 319 (100). Anal. calc. for $C_{21}H_{15}ClN_2O_2$ (362.81): C 69.52, H 4.17, N 7.72; found: C 69.51, H 4.21, N 7.61.

*2-(3-Methoxyphenyl)-3-phenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3c**). Pale-yellow solid. M.p. 211–214° (hexane/CH_2Cl_2). IR (KBr): 1747, 1600. ^{1}H-NMR: 2.15 (*s*, 3 H); 3.82 (*s*, 3 H); 6.98 (*dd*, *J* = 7.8, 2.7, 1 H); 7.07 (*dd*, *J* = 4.6, 0.9, 1 H); 7.23 (*t*, *J* = 7.8, 1 H); 7.29–7.33 (*m*, 3 H); 7.35–7.37 (*m*, 2 H); 7.40 (*d*, *J* = 7.8, 1 H); 7.64 (*t*, *J* = 1.4, 1 H); 8.43 (*d*, *J* = 4.6, 1 H), 8.98 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.95; 55.29; 90.23; 112.63; 116.40; 118.70; 121.45; 123.91; 128.77; 129.32; 129.58; 131.67; 135.28; 142.78; 148.13; 149.09; 149.19; 159.69; 167.72; 177.64. MS: 358 (25, M^+), 315 (100). Anal. calc. for $C_{22}H_{18}N_2O_3$ (358.39): C 73.73, H 5.06, N 7.82; found: C 73.60, H 5.13, N 7.88.

*2-(3,4-Dimethoxyphenyl)-3-phenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3d**). Pale-yellow solid. M.p. 143–145° (hexane/CH_2Cl_2). IR (KBr): 1755. ^{1}H-NMR: 2.14 (*s*, 3 H); 3.88 (*s*, 3 H); 3.92 (*s*, 3 H); 6.75 (*d*, *J* = 8.2, 1 H); 7.06 (*dd*, *J* = 5.0, 0.9, 1 H); 7.28–7.33 (*m*, 4 H); 7.37 (*dd*, *J* = 8.2, 1.8, 2 H); 7.77 (*d*, *J* = 2.3, 1 H); 8.40 (*d*, *J* = 5.0, 1 H); 8.95 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.97; 55.86; 55.89; 90.15; 110.40; 110.68; 116.37; 123.07; 123.36; 123.87; 128.70; 129.28; 135.83; 142.33; 147.61; 148.87; 149.21; 149.49; 152.34; 167.61; 177.24. MS: 388 (26, M^+), 345 (100). Anal. calc. for $C_{23}H_{20}N_2O_4$ (388.42): C 71.12, H 5.19, N 7.21; found: C 71.08, H 5.33, N 7.15.

*3-(4-Chlorophenyl)-2-phenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3e**). White solid. M.p. 203–205° (hexane/CH_2Cl_2). IR (KBr): 1751. ^{1}H-NMR: 2.14 (*s*, 3 H); 7.06 (*dd*, *J* = 5.0, 0.9, 1 H); 7.28 (*d*, *J* = 9.2, 2 H), 7.30 (*d*, *J* = 9.2, 2 H); 7.37 (*dd*, *J* = 7.8, 7.3, 2 H), 7.45 (*tt*, *J* = 7.3, 1.4, 1 H); 7.94 (*dd*, *J* = 7.8, 1.4, 2 H); 8.45 (*d*, *J* = 5.0, 1 H); 8.98 (*s*, 1 H). ^{13}C-NMR ((D_6)DMSO): 20.58; 89.11; 116.90; 126.04; 128.11; 129.13; 129.51; 129.81; 132.23; 133.53; 134.21; 142.03; 148.43; 148.47; 148.58; 168.25; 176.74. MS: 362 (79, M^+), 319 (100). Anal. calc. for $C_{21}H_{15}ClN_2O_2$ (362.81): C 69.52, H 4.17, N 7.72; found: C 69.39, H 4.09, N 7.65.

*2,3-Bis(4-chlorophenyl)-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3f**). White solid. M.p. 202–203° (hexane/CH_2Cl_2). IR (KBr): 1753. ^{1}H-NMR: 2.15 (*s*, 3 H); 7.06 (*dd*, *J* = 4.6, 0.9, 1 H); 7.28 (*d*, *J* = 9.2, 2 H); 7.29 (*d*, *J* = 9.2, 2 H); 7.35 (*d*, *J* = 8.7, 2 H); 7.87 (*d*, *J* = 8.7, 2 H); 8.46 (*d*, *J* = 4.6, 1 H); 8.97 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.87; 89.70; 116.37; 125.41; 128.61; 129.21; 129.73; 129.84; 133.49; 134.99; 138.35; 142.85; 148.30; 148.61; 149.06; 167.48; 176.27. MS: 396 (74, M^+), 353 (100). Anal. calc. for $C_{21}H_{14}Cl_2N_2O_2$ (397.25): C 63.49, H 3.55, N 7.05; found: C 63.47, H 3.62, N 6.82.

*3-(4-Methoxyphenyl)-2-phenyl-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3g**). Pale-yellow amorphous powder. R_f (AcOEt/hexane 1:1) 0.31. IR (neat): 1749, 1609. ^{1}H-NMR: 2.12 (*s*, 3 H); 3.75 (*s*, 3 H); 6.82 (*d*, *J* = 8.7, 2 H); 7.01 (*d*, *J* = 4.6, 1 H); 7.2 (*d*, *J* = 8.7, 2 H); 7.37 (*dd*, *J* = 7.8, 7.3, 2 H); 7.44 (*t*, *J* = 7.3, 1 H); 7.97 (*dd*, *J* = 7.8, 0.9, 2 H); 8.43 (*d*, *J* = 4.6, 1 H); 8.96 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.88; 55.18; 90.08; 114.66; 116.23; 125.37; 127.02; 128.65; 128.71; 130.49; 131.74; 142.69; 148.00; 149.06; 149.20; 159.79; 167.75; 177.69. MS: 358 (28, M^+), 315 (100). Anal. calc. for $C_{22}H_{18}N_2O_3$ (358.39): C 73.73, H 5.06, N 7.82; found: C 73.55, H 5.09, N 7.55.

*3-(4-Methoxyphenyl)-2-(4-methylphenyl)-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3h**). White solid. M.p. 187–190° (hexane/CH_2Cl_2). IR (KBr): 1755, 1609. ^{1}H-NMR: 2.12 (*s*, 3 H); 2.35 (*s*, 3 H); 3.74 (*s*, 3 H); 6.82 (*d*, *J* = 9.2, 2 H); 7.08 (*dd*, *J* = 4.6, 0.9, 1 H); 7.17 (*d*, *J* = 7.8, 2 H); 7.28 (*d*, *J* = 9.2, 2 H); 7.87 (*d*, *J* = 7.8, 2 H); 8.41 (*d*, *J* = 4.6, 1 H); 8.94 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.94; 21.60; 55.20; 90.04; 114.63; 116.25; 125.38; 127.22; 127.76; 128.75; 129.47; 142.36; 142.50; 147.66 (two overlapped C-atoms); 149.22; 159.75; 167.74; 177.72. MS: 372 (49, M^+), 329 (100). Anal. calc. for $C_{23}H_{20}N_2O_3$ (372.42): C 74.18, H 5.41, N 7.52; found: C 73.95, H 5.47, N 7.42.

*2-(4-Chlorophenyl)-3-(4-methoxyphenyl)-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3i**). White solid. M.p. 161–163° (hexane/CH_2Cl_2). IR (KBr): 1757, 1609. ^{1}H-NMR: 2.13 (*s*, 3 H); 3.75 (*s*, 3 H); 6.82 (*d*, *J* = 9.2, 2 H); 7.07 (*d*, *J* = 5.0, 1 H); 7.25 (*d*, *J* = 8.7, 2 H); 7.35 (*d*, *J* = 9.2, 2 H); 7.91 (*d*, *J* = 8.7, 2 H); 8.44 (*d*, *J* = 5.0, 1 H); 8.95 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.87; 55.20; 89.93; 114.79; 116.25; 125.32; 126.62; 128.96; 129.03; 129.90; 138.00; 142.81; 148.24; 128.92; 149.01; 159.89; 167.72; 176.57. MS: 392 (71, M^+), 349 (100). Anal. calc. for $C_{22}H_{17}ClN_2O_3$ (392.84): C 67.26, H 4.36, N 7.13; found: C 67.25, H 4.48, N 6.99.

*2,3-Bis(4-methoxyphenyl)-3*H*-pyrrolo[2,3-*c*]pyridin-3-yl Acetate* (**3j**). White solid. M.p. 191–193° (hexane/CH_2Cl_2). IR (KBr). 1753, 1607. ^{1}H-NMR: 2.12 (*s*, 3 H); 3.74 (*s*, 3 H); 3.81 (*s*, 3 H); 6.82 (*d*, *J* = 8.7, 2 H); 6.88 (*d*, *J* = 9.2, 2 H); 7.06 (*d*, *J* = 4.6, 1 H); 7.28 (*d*, *J* = 8.7, 2 H); 7.94 (*d*, *J* = 9.2, 2 H); 8.39 (*d*, *J* = 4.6, 1 H); 8.92 (*s*, 1 H). ^{13}C-NMR ($CDCl_3$): 20.94; 55.18; 55.30; 89.99; 114.15; 114.58; 116.15; 123.17; 125.35; 127.45; 130.66; 142.27; 147.51; 148.88; 149.31; 159.70; 163.43; 167.70; 177.12. MS: 388 (69, M^+), 345 (100). Anal. calc. for $C_{23}H_{20}N_2O_4$ (388.42): C 71.12, H 5.19, N 7.21; found: C 71.06, H 5.22, N 7.14.

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