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ORIGINAL ARTICLE

Synthesis of new (pyrazol-1-yl)(7-nitro-1h-indol-2-yl) ketone derivatives

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Ethyl 7-nitroindole-2-carboxylate;
7-Nitroindole-2-carbohydrazide;
Pyrazolylindole;
Acetylacetone

Abstract The condensation of 7-nitroindole-2-carbohydrazide derivatives with acetylacetone lead to (pyrazol-1-yl)(7-nitroindol-2-yl)ketones.

1. Introduction

The indole nucleus is probably the most widely distributed heterocyclic ring system found in nature (Kuethe et al., 2005). Due to the existence of a vast array of structurally diverse and biologically active indoles, it is not surprising that the indole nucleus is an important feature in many medicinal agents and the most important of all structural classes in drug discovery (Smith et al., 1998). The synthesis and reactivity of indole derivatives have been a topic of research interest for well over a century.

Compounds which contain the pyrazole functionality continue to attract great interest due to their varied and significant pharmacological effects. For example, the identification of new and selective cox-2 inhibitors (Penning et al., 1997), for the relief of pain and the treatment of the symptom of arthritis and related diseases has been an important advance in modern anti-inflammatory therapy. In a related area, heterocycle-appended pyrazoles have been reported (Dumas et al., 2000) to be potent and selective in inhibitors of the mitogen-activated protein kinase p38 and consequently provide a novel approach for the treatment of rheumatoid arthritis and related inflammatory diseases.

2. Results and discussion

Due to the potent biological activity exhibited by various indoles derivatives, there is a continuous demand for novel

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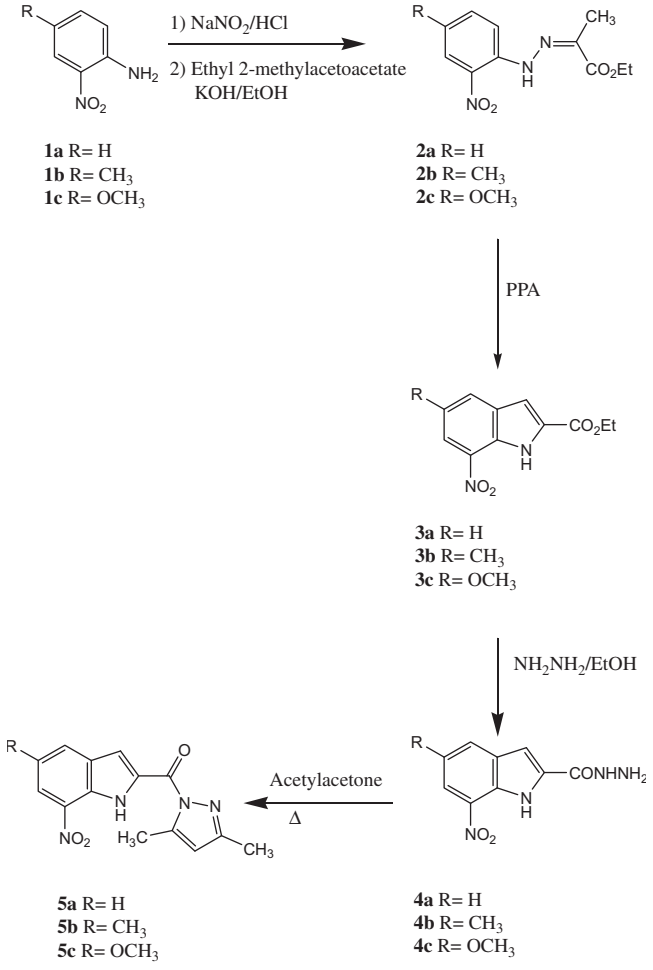
synthetic procedures in this area. In 1990s, it has attracted much attention, as it employs simple and readily available starting materials. In previous papers (El Kihel et al., 2007; El Ouar et al., 1995), we have reported some reactions of 7-aminoindoles, in this work; we have improved the synthesis of substituted ethyl 7-nitroindole-2-carboxylate **3(a–c)** that the synthesis of ethyl 7-nitroindole-2-carboxylate has been reported (Murakami et al., 1993, 1998). The starting compounds 7-nitroindole-2-carbohydrazides **4(a–c)** were prepared by the reaction of hydrazine hydrate with substituted ethyl 7-nitroindole-2-carboxylate. These hydrazides when reacted with acetylacetone yielded (3,5-dimethyl-1H-pyrazol-1-yl)(7-nitro-1H-indol-2-yl)ketone **5(a–c)**.

2.1. Preparation of substituted 2-nitrophenylhydrazones *2(a–c)*

Phenylhydrazones **2(a–c)** were generally prepared starting from substituted ortho-nitroanilines **1(a–c)** via diazotization, followed by Japp Klingemann reaction using ethyl a-methylacetoacetate in the presence of KOH/EtOH. Phenylhydrazones **2(a–c)** thus prepared consisted of Z- and E-geometrical isomers (Scheme 1). We showed that these geometrical isomers are rapidly interconvertible by the polyphosphoric acid as catalyst used for Fischer indolization and thus give the same result on Fischer indolization.



1a R= H
1b R= CH_3
1c R= OCH_3

2a R= H
2b R= CH_3
2c R= OCH_3

3a R= H
3b R= CH_3
3c R= OCH_3

4a R= H
4b R= CH_3
4c R= OCH_3

5a R= H
5b R= CH_3
5c R= OCH_3

Scheme 1

2.2. Fischer indolization

The Fischer indolization of the phenylhydrazones **2(a–c)** was carried out mainly with polyphosphoric acid (PPA) which caused the reaction to proceed the most rapidly instead of other catalysts (Scheme 1).

The identification of the indolic products **3(a–c)** was based on spectroscopic data. In the ^{1}HNMR spectra of these products, we noted the upfield-shifted proton NH to 10 ppm and the disappearance of the singlet of methyl group during Fischer indolization.

2.3. Synthesis of carbohydrazides *4(a–c)*

For the synthesis of the carbohydrazide **4(a–c)**, we have used the method reported by literature (Harrison et al., 2006; Narayana et al., 2005; Farghaly, 2004). The reaction between the ethyl 7-nitroindole-2-carboxylates and hydrazine hydrate lead to the titled products (Scheme 1). The identification of the structure was based on spectroscopic data.

The ^{1}HNMR spectra of the carbohydrazide **4a** displayed two singlets at 4.61 and 11.25 due to protons of $NHNH_2$ group instead of ethyl group protons in the ethyl 7-nitroindole-2-carboxylate.

2.4. Synthesis of pyrazolylindole derivatives *5(a–c)*

Acid-catalyzed substitution reactions on indole derivatives containing only 7-nitro substituent in the benzene ring, in general, are prohibited by the acid lability of the indole nucleus, and in those cases where these reactions are possible, the substituent orientation and the remaining functionality are not always the most desired. Ready access has provided the impetus to investigate synthetic schemes that might be expected to provide various indole-substituted by pyrazole moiety (Farghaly, 2004; Hiremath et al., 1988; Farhanullah et al., 2004; Jukic et al., 1999; Przheval'skii et al., 2004). This work describes general procedure by which (3,5-dimethyl-1H-pyrazol-1-yl) (7-nitro-1H-indol-2-yl)ketone derivatives **5(a–c)** may be conveniently prepared in neutral medium by the reaction of the carbohydrazides with acetylacetone.

The establishment of the structure of these compounds **5(a–c)** has been confirmed by spectroscopic data. The ^{1}HNMR spectra of the compound **5a** showed the presence of the sharp singlets at 2.36 and 2.62 due to protons of two methyl groups of pyrazole moiety and the methine proton appeared at 6.04 ppm. The ^{13}CNMR spectra of **5a** exhibited two signals at 13.2 and 13.6 assignable to carbons of two methyl groups. The molecular ion peak at m/z 284 was observed in the mass spectrum of **5a**. These spectra data and elemental analysis supported the structure of **5a**.

3. Conclusion

In this work, we report the condensation of 7-nitroindole-2-carbohydrate derivatives with acetylacetone leading to new (3,5-dimethylpyrazol-1-yl)(7-nitro-1H-indol-2-yl)ketones. The structures of obtained products were established with spectroscopic data of proton and carbon 13 NMR, mass.

4. Experimental

All compounds were characterized by their ^{1}H-NMR and ^{13}C-NMR spectra as well as by microanalysis or HRMS spectra. NMR spectra were recorded on Bruker ARX 200 (200 MHz for ^{1}H and 50.3 MHz for ^{13}C) spectrometer (δ-ppm/TMS, J-Hz); for ^{13}CNMR, the multiplicities were determined through DEPT. Microanalysis were performed by the "Laboratoire Central de Microanalyse du UATRS" (Rabat). Mass spectra were recorded on a Varian MAT 311 specrtometer. Melting points were measured using a Köfler appartus and were uncorrected.

*4.1. Preparation of o-nitrophenylhydrazones **2(a–c)***

Solid $NaNO_2$ (32 mmol) was added portion wise to a solution of substituted ortho-nitroaniline (29 mmol) and concentrated HCl (6.4 g) in H_2O (22 ml) at 0–4 °C. The resulting diazonium salt solution was added dropwise to a solution of ethyl α-methylacetoacetate (29 mmol) and 50% aqueous KOH in EtOH (28 ml) at 0–7 °C, and the whole was stirred for 1 h under ice-cooling. The reaction mixture was poured into H_2O and extracted with Et_2O. The organic solution was dried over $MgSO_4$ and evaporated. The residue was recrystallized from ethanol.

*4.1.1. Ethyl pyruvate 2-(2-nitrophenyl)hydrazone **2a***

Yield = 78%; mp = 116–118 °C (Ethanol). ^{1}HNMR (DMSO-d_6): 1.36(t, J = 7.9 Hz, 3H, CH_3); 2.21(s, 3H, CH_3); 4.38(q, J = 7.9 Hz, 2H, CH_2); 6.84(dd, J = 2.0 Hz, J = 8.1 Hz, 1H, H^6); 7.43(t, J = 8.1 Hz, 1H, H^4); 7.55(t, J = 8.1 Hz, 1H, H^5); 8.19(dd, J = 2.0 Hz, J = 8.1 Hz, 1H, H^3); 10.70(br s, 1H, NH). ^{13}CNMR (DMSO-d_6): 12.0(CH_3); 14.7(CH_3); 62.1(CH_2); 117,2(CH-5); 120.7(CH-4); 133.1(CH-3); 136.7(CH-6); 126.2, 139.7 (ArC); 141.0(C═N); 165.0(CO_2). HRMS, *m*/*z*: 251(M), calcd. for $C_{11}H_{13}N_3O_4$: 251.090, found: 251.091.

*4.1.2. Ethyl pyruvate 2-(4-methyl-2-nitrophenyl)hydrazone **2b***

Yield = 71%; mp = 144–146 °C (Ethanol). ^{1}HNMR (DMSO-d_6): 1.39(t, J = 7.3 Hz, 3H, CH_3); 2.24(s, 3H, CH_3); 2.34(s, 3H, CH_3); 4.38(q, J = 7.3 Hz, 2H, CH_2); 7.40(d, J = 9.1 Hz, 1H, H^6); 7.89(dd, J = 9.1 Hz, J = 1.8 Hz, 1H, H^5); 8.01(d, J = 1.8 Hz, 1H, H^3); 10.90(br s, 1H, NH). ^{13}CNMR (DMSO-d_6): 11.9(CH_3); 14.7(CH_3); 20.7(CH_3); 62.0(CH_2); 117,1(CH-6); 125.6(CH-3); 132.8(CH-5); 130.7, 132.9, 138.0(ArC); 139.0(C═N); 165.0(CO_2). HRMS, *m*/*z*: 265(M), calcd. for $C_{12}H_{15}N_3O_4$: 265.106, found: 265.106.

*4.1.3. Ethyl pyruvate 2-(4-methoxy-2-nitrophenyl)hydrazone **2c***

Yield = 73%; mp = 126–128 °C (Ethanol). ^{1}HNMR (DMSO-d_6): 1.32(t, J = 7.1 Hz, 3H, CH_3); 2.19(s, 3H, CH_3); 3.38(s, 3H, OCH_3); 4.30(q, J = 7.1 Hz, 2H, CH_2); 7.51(dd, J = 8.3, J = 2.9 Hz, 1H, H^5); 7.63(d, J = 2.9 Hz, 1H, H^3); 7.85(d, J = 8.3 Hz, 1H, H^6); 10.55(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 11.3(CH_3); 14.3(CH_3); 55.8(OCH_3); 61.3(CH_2); 107.1(CH-3); 118.1(CH-6); 126.4(CH-5); 132.6, 135.0(ArC); 138.7(C═N); 153.3(C–O); 164.6(CO_2). HRMS, *m*/*z*: 281(M), calcd. for $C_{12}H_{15}N_3O_5$: 281.101, found: 281.101.

*4.2. Synthesis of ethyl 7-nitroindole-2-carboxylate derivatives **3(a–c)***

A mixture of the hydrazone **2(a–c)** (7.5 mmol) and polyphosphoric acid (10 g) was heated at 120 °C for 30 min. The reaction mixture was poured into water and extracted with dichloromethane. The solvent was evaporated. The crude product was filtered and recrystallized from ethanol.

*4.2.1. Ethyl 7-nitroindole-2-carboxylate **3a***

Yield = 61%; mp = 94–96 °C (Ethanol). ^{1}HNMR (DMSO-d_6): 1.45(t, J = 8.4 Hz, 3H, CH_3); 4.47(q, J = 8.4 Hz, 2H, CH_2); 7.23 (s, 3H, CH^3); 7.70(t, J = 9.3 Hz, 1H, H^5); 8.09(dd, J = 9.3 Hz, J = 2.1 Hz, 1H, H^4); 8.31(dd, J = 9.3 Hz, J = 2.1 Hz, 1H, H^6); 10.29(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 14.8(CH_3); 62.0(CH_2); 110.1(CH-3); 120.5(CH-6); 122.5(CH-5); 130.6(CH-4); 130.9, 131,1, 131.8, 133.9 (ArC); 161.2(CO_2). HRMS, *m*/*z*: 234(M), calcd. for $C_{11}H_{10}N_2O_4$: 234.064, found: 234.064.

*4.2.2. Ethyl 5-methyl-7-nitroindole-2-carboxylate **3b***

Yield = 70%; mp = 108–110 °C (Ethanol). ^{1}HNMR (DMSO-d_6): 1.45(t, J = 7.1 Hz, 3H, CH_3); 2.50(s, 3H, CH_3); 4.47(q, J = 7.1 Hz, 2H, CH_2); 7.31(s, 1H, H^3); 7.64(d, J = 1.3 Hz, 1H, H^4); 8.03(d, J = 1.3 Hz, 1H, H^6); 10.25(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 14.8(CH_3); 21.4(CH_3); 61.9(CH_2); 109.1(CH-3); 123.7(CH-6): 130.5(CH-4); 128.6, 129.6, 131.0, 131.5, 133.3(ArC); 161.2(CO_2). HRMS, *m*/*z*: 248(M), calcd. for $C_{12}H_{12}N_2O_4$ 248.080, found: 248.080.

*4.2.3. Ethyl 5-methoxy-7-nitroindole-2-carboxylate **3c***

Yield = 55%; mp = 144–146 °C (Ethanol). ^{1}HNMR (DMSO-d_6): 1.44(t, J = 7.1 Hz, 3h, CH_3); 3.92(s, 3H, OCH_3); 4.61(q, J = 7.1 Hz, 2H, CH_2); 7.23(d, J = 2.3 Hz, 1H, H^4); 7.50(d, J = 2.3 Hz, 1H, H^3); 7.90(d, J = 2.3 Hz, 1H, H^6); 10,11(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 14.3(CH_3); 56.3(OCH_3); 61.5(CH_2); 108.5(CH-6); 110.8(CH-3); 113.7(CH-4); 125.3, 130.2, 131.2, 133.1, 153.5(ArC); 160.7(CO_2). HRMS, *m*/*z*: 264(M), calcd. for $C_{12}H_{12}N_2O_5$: 264.075, found: 264.075.

*4.3. Synthesis of 7-nitroindole-2-carbohydrazide derivatives **4(a–c)***

A mixture of (2 mmol) ethyl 7-nitroindole-2-carboxylate derivatives **3(a–c)**, 10 mmol hydrazine hydrate and 30 ml of ethanol was shaken at room temperature for 30 min. It was left for about 1 h, and then the hydrazide was separated out by filtration and crystallized from ethanol.

*4.3.1. 7-Nitroindole-2-carbohydrazide **4a***

Yield = 48%; mp > 300 °C. ^{1}HNMR (DMSO-d_6): 4.61(s, 2H, NH_2); 7.29(t, J = 7.1 Hz, 1H, H^5); 7.33(d, J = 1.7 Hz, 1H, H^3); 8.14(dd, J = 7 Hz, J = 1 Hz, 1H, H^4); 8.19(dd, J = 7 Hz, J = 1 Hz, 1H, H^6); 10,23(s, 1H, NH); 11.25(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 106.6(CH-6); 120.0(CH-3); 121.7(CH-5); 130.4(CH-4); 129.1, 131.4, 133.4, 134.0(ArC); 159.7(CO). HRMS, *m*/*z*: 220(M), calcd. for $C_9H_8N_4O_3$: 220.059, found: 220.060.

*4.3.2. 5-Methyl-7-nitroindole-2-carbohydrazide **4b***

Yield = 56%; mp > 300 °C. ^{1}HNMR (DMSO-d_6): 2.48(s, 3H, CH_3); 4.64(s, 2H, NH_2); 7.27(d, J = 1.7 Hz, 1H, H^3); 7.98(d, J = 1 Hz, 1H, H^4); 8.05(d, J = 1 Hz, 1H, H^6); 10,24(s, 1H, NH); 11.18(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 20.6(CH_3); 105.5(CH-6); 121.8(CH-3); 130.5(CH-4); 127.5,

129.6, 131.3, 132.7, 133.8(ArC); 159.4(CO). HRMS, *m/z*: 234(M), calcd. for $C_{10}H_{10}N_4O_3$: 234.075, found: 234.075.

4.3.3. 5-Methoxy-7-nitroindole-2-carbohydrazide ***4c***

Yield = 56%; mp > 300 °C. ^{1}HNMR (DMSO-d_6): 3.85(s, 3H, $0CH_3$); 4.59(s, 2H, NH_2); 7.22(d, J = 1.8 Hz, 1H, H^3); 7.72(d, J = 2.4 Hz, 1H, H^4); 7.74(s, J = 2.4 Hz, 1H, H^6); 10.19(s, 1H, NH); 11.06(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 56.2(OCH_3); 105.7(CH-6); 109.2(CH-4); 113.9(CH-3); 124.6, 131.9, 132.8, 135.0, 153.3(ArC); 159.7(CO). HRMS, *m/z*: 250(M), calcd. for $C_{10}H_{10}N_4O_4$: 250.070, found: 250.071.

4.4. Condensation of 7-nitroindole-2-carbohydrazide derivatives ***4(a–c)*** *with acetylacetone*

The 7-nitroindole-2-carbohydrazide derivatives **4(a–c)** (2.4 mmol) and acetylacetone (3.6 mmol) were heated under reflux for 5 h. After cooling, the obtained solid product was filtered off, and then recrystallized from ethanol.

4.4.1. (3,5-Dimethylpyrazol-1-yl)(7-nitro-1H-indol-2-yl)ketone ***5a***

Yield = 48%; mp > 300 °C. ^{1}HNMR (DMSO-d_6): 2.36(s, 3H, CH_3); 2.62(s, 3H, CH_3); 6.04(s, CHpyrazolic); 7.37(t, J = 8.5 Hz, 1H, H^5); 7.58(d, J = 1.9 Hz, 1H, H^3); 8.27(dd, J = 8.5 Hz, J = 2.1 Hz, 1H, H^4); 8.38(dd, J = 8.5 Hz, J = 2.1 Hz, 1H, H^6); 12,42(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 13.2(CH_3); 13.6(CH_3); 110.2(CHpyrazolic); 114.0(CH-6); 114.9(CH-3); 123.2(CH-5); 126.8(CH-4); 132.2, 136.0, 138.5, 138.9, 143.2, 152.3(ArC); 162.8(CO). HRMS, *m/z*: 284(M), calcd. for $C_{14}H_{12}N_4O_3$: 284.091, found: 284.090. Analysis: $C_{14}H_{12}N_4O_3$ (284.3); calcd. C 59.15, H 4.25, N 19.71; found C 59.17, H 4.41, N 18.81.

4.4.2. (3,5-Dimethylpyrazol-1-yl)(5-methyl-7-nitro-1H-indol-2-yl)ketone ***5b***

Yield = 56%; mp > 300 °C. ^{1}HNMR (DMSO-d_6): 2.39(s, 3H, CH_3); 2.50(s, 3H, CH_3); 2.65(s, 3H, CH_3); 6.07(s, 1H, CHpyrazolic); 7.26(d, J = 2.3 Hz, 1H, H^3); 7.63(d, J = 1.7 Hz, 1H, H^4); 8.11(d, J = 1.9 Hz, 1H, H^6); 12.28(s, NH). ^{13}CNMR (DMSO-d_6): 13.6(CH_3); 14.8(CH_3); 20.9(CH_3); 111.1(CHpyrazolic); 113.7(CH-3); 124.0(CH-6); 130.6(CH-4); 128.6, 129.5, 131.4, 132.1, 133.3, 146.2, 153.1(ArC); 157.8(CO). HRMS, *m/z*: 298(M), calcd for $C_{15}H_{14}N_4O_3$: 298.107, found: 298.106. Analysis: $C_{15}H_{14}N_4O_3$ (298.3); calcd. C 60.40, H 4.73, N 18.78; found C 60.60, H 4.94, N 18.04.

4.4.3. (3,5-Dimethylpyrazol-1-yl)(5-methoxy-7-nitro-1H-indol-2-yl)ketone ***5c***

Yield = 56%; mp > 300 °C. ^{1}HNMR (DMSO-d_6): 2.41(s, 3H, CH_3); 2.67(s, 3H, CH_3); 3.91(s, 3H, OCH_3); 6.08(s, 1H, CHpyrazolic); 7.54(d, J = 2.7 Hz, 1H, H^3); 7.69(d, J = 1.9 Hz, 1H, H^4); 7.95(d, J = 3.3 Hz, 1H, H^6); 12,27(s, 1H, NH). ^{13}CNMR (DMSO-d_6): 13.4(CH_3); 14.8(CH_3); 56.2(OCH_3); 111.1(CHpyrazolic); 112.0(CH-3); 113.3(CH-6); 113.4(CH-4); 109.7, 114.1, 125.8, 130.4, 132.9, 146.1, 153.4(ArC); 153.5(CO). HRMS, *m/z*: 314(M), calcd. for $C_{15}H_{14}N_4O_4$: 314.102, found: 314.103. Analysis: $C_{15}H_{14}N_4O_4$ (314.3); calcd. C 57.32, H 4.49, N 17.83; found C 57.41, H 4.69, N 17.09.

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