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3-ARYLMETHYLIDENE-3H-PYRROL-2-ONES UNDER FRIEDEL-CRAFTS REACTION CONDITIONS

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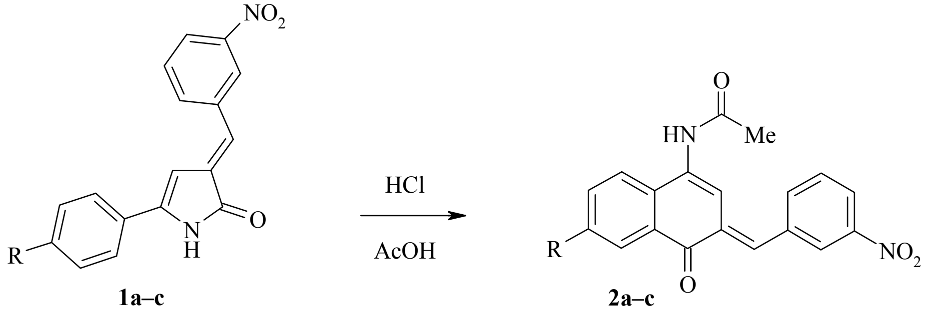
Arylmethylidene derivatives of 5R-3H-pyrrol-2-ones have attracted attention mainly as intermediates in the synthesis of various complex systems. The Friedel-Crafts reaction is a promising method for the synthesis of both aromatic and nonaromatic compounds, facilitating the formation of new C–C bonds and the introduction of various additional functional groups. The Friedel-Crafts reaction of arylmethylidene derivatives of oxygen heterocycles has already been studied by Ward [1] and Guirguis [2] but, in all cases, these authors indicated the formation of naphthoic acid derivatives.

Heating 3-arylmethylidene-3H-pyrrol-2-ones in a mixture of hydrochloric and acetic acids leads to products characterized as N-(6-R-3-[(3-nitrophenyl)methylidene]-4-oxo-3,4-dihydronaphthalen-1-yl)acetamides **2a-c**.



1, 2 a R = H, **b** R = Me, **c** R = OMe

The presence and arrangement of the signals in the ^{1}H and ^{13}C NMR spectra are in complete accord with the proposed structure. Protonation of the heteroatom in pyrrolones **1a-c** occurs under acid catalysis conditions, which leads to cleavage of the N–C(2) bond, formation of a carbocation, and concurrent acylation of the amino group.

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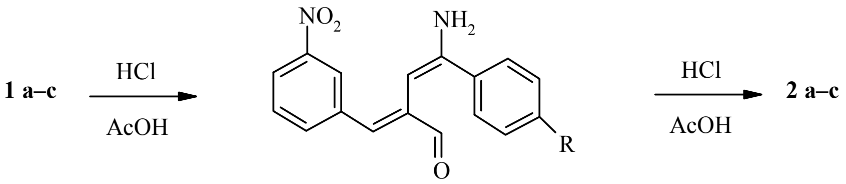
Attack of the carbon atom in the *ortho* position of the activated benzene ring leads to the formation of acetamides **2a-c**.



The ^{1}H and ^{13}C NMR spectra were taken on a Bruker MSL-400 spectrometer at 400 and 100 MHz, respectively, at 20-25°C in $CDCl_3$ with TMS as the internal standard. Thin-layer chromatography was carried out on Silufol UV-254 plates with 2:2:1 hexane–ethyl acetate–chloroform as the eluent. Iodine vapor was used for development.

N-(6-R-3-[(3-Nitrophenyl)methylidene]-4-oxo-3,4-dihydronaphthalen-1-yl)acetamides 2a-c. A mixture of 5-R-3-arylmethylidene-3H-pyrrol-2-one **1a-c** (0.01 mol) and hydrochloric acid (0.01 mol) was stirred at reflux for 2-3 h in 15 ml glacial acetic acid. The precipitated crystals were filtered off and recrystallized from acetic acid.

N-(3-[(3-Nitrophenyl)methylidene]-4-oxo-3,4-dihydronaphthalen-1-yl)acetamide (2a) was obtained in 79% yield; mp 198-200°C. ^{1}H NMR spectrum, δ, ppm: 2.32 (3H, s, CH_3); 6.91 (1H, s, =CH); 7.35 (1H, s, H-2); 7.96-8.27 (8H, m, Ar); 9.48 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 25.7, 111.9, 112.7, 114.5, 115.7, 116.1, 118.2, 121.3, 123.1, 126.5, 127.5, 129.5, 130.4, 132.9, 134.6, 136.8, 138.9, 167.6, 183.4. Found, %: C 68.01; H 4.39; N 8.47. $C_{19}H_{14}N_2O_4$. Calculated, %: C 68.26; H 4.22; N 8.38.

N-(6-Methyl-3-[(3-Nitrophenyl)methylidene]-4-oxo-3,4-dihydronaphthalen-1-yl)acetamide (2b) was obtained in 84% yield; mp 208-210°C. ^{1}H NMR spectrum, δ, ppm: 2.45 (3H, s, CH_3); 2.52 (3H, s, CH_3); 6.88 (1H, s, =CH); 7.58 (1H, s, H-2); 7.94-8.19 (7H, m, Ar); 9.59 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 22.4, 26.3, 111.3, 114.3, 115.1, 116.8, 117.9, 119.2, 120.6, 123.9, 124.1, 129.4, 133.5, 135.4, 137.5, 139.2, 144.6, 149.6, 168.3, 181.7. Found, %: C 69.23; H 4.78; N 7.75. $C_{20}H_{16}N_2O_4$. Calculated, %: C 68.96; H 4.63; N 8.04.

N-(6-Methoxy-3-[(3-nitrophenyl)methylidene]-4-oxo-3,4-dihydronaphthalen-1-yl)acetamide (2c) was obtained in 85% yield; mp 224-226°C. ^{1}H NMR spectrum, δ, ppm: 2.56 (3H, s, CH_3); 3.86 (3H, s, OCH_3); 6.97 (1H, s, =CH); 7.21 (1H, s, H-2); 8.14-8.48 (7H, m, Ar); 9.75 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 25.5, 49.6, 116.1, 117.4, 119.0, 121.4, 124.6, 127.3, 129.2, 134.5, 136.2, 137.2, 139.2, 143.1, 150.2, 152.3, 154.6, 169.3, 184.5. Found, %: C 66.15; H 3.98; N 7.93. $C_{20}H_{16}N_2O_5$. Calculated, %: C 65.93; H 4.43; N 7.69.

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