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TWO DIRECTIONS FOR THE RECYCLIZATION OF N'-PHENYL-6-METHOXY-2,2,4-TRIMETHYL-1,2-DIHYDRO-1-QUINOLINECARBOXAMIDE

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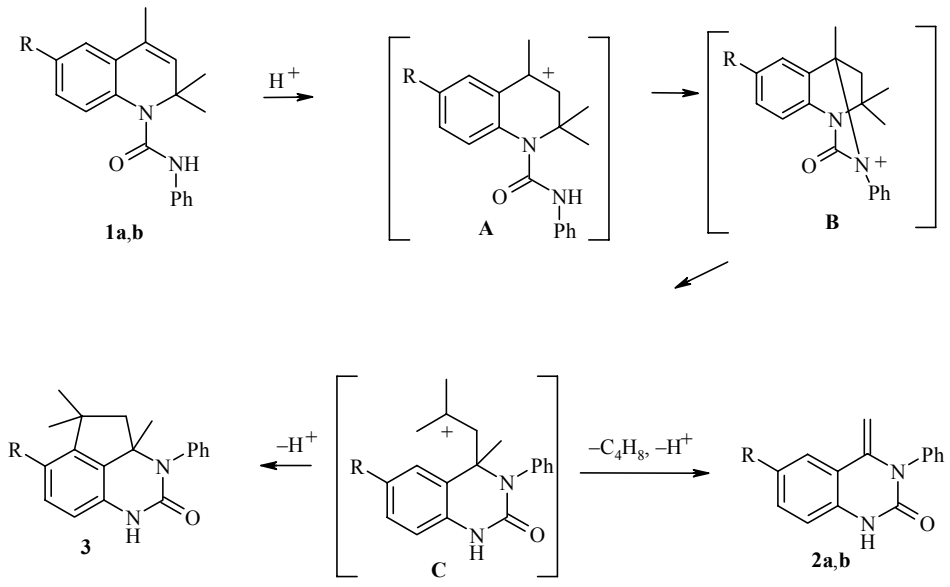
We have found that while the recyclization described by Brack [1] for N'-phenyl-2,2,4-trimethyl-1,2-dihydro-1-quinolinecarboxamide **1a**, which is unsubstituted in the aromatic ring, by the action of concentrated hydrochloric acid gives 4-methylenequinazolone **2a**, the analogous reaction of methoxy derivative **1b** is accompanied by the alternative formation of 1,2,3,3a,4,5-hexahydrocyclopenta[*de*]quinazolin-2-one **3**.



1, **2 a** R = H, **1b, 2b**, **3** R = OMe

Cation **C** formed through intermediates **A** and **B** [2] closes when an activating methoxy group is present in the aromatic ring to give 1,2,3,3a,4,5-hexahydrocyclopenta[*de*]quinazolin-2-one **3** by means of an intramolecular Friedel–Crafts reactions in addition to stabilization by means of loss of isobutylene molecule to give 4-methylenequinazolone **2b**.

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This reaction is carried out as follows. A sample of compound **1b** (3.22 g, 10 mmol) was dissolved in concentrated sulfuric acid (25 ml), maintained at 40-50°C for 1 h, cooled, and poured into ice water (200 ml). The precipitate formed was filtered off, washed with water, and treated with 25% aq. ammonia (10 ml). Recrystallization from toluene gave 0.87 g (27%) of **3**. The aqueous filtrate was treated with 25% aqueous ammonia to bring the pH to 9-10. The precipitate was filtered off, washed with water, and recrystallized from toluene to give 0.66 g (25%) **2b**.

6-Methoxy-4-methylene-3-phenyl-1,2,3,4-tetrahydro-2-quinazolinone (2b); mp 230°C (dec.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.45, 4.18 (2H, 2s, CH_2); 3.85 (3H, s, OMe); 6.70-7.41 (8H, m, Ar); 10.18 (1H, s, NH). Mass spectrum, *m/z*: 266 [M^+]. Found, %: C 72.25; H 5.41; N 10.32. $C_{16}H_{14}N_2O_2$. Calculated, %: C 72.10; H 5.26; N 10.51.

6-Methoxy-3a,5,5-trimethyl-3-phenyl-1,2,3,3a,4,5-hexahydrocyclopenta[*de*]quinazolin-2-one (3); mp 225°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm, *J* (Hz): 1.37, 1.40 (2×3H, 2s, CMe_2); 1.62 (3H, s, 3a-Me); 1.72 (1H, d, $^2J_{ae}$ = 12.2, $^3H_{a(e)}$, CH_2); 2.18 (1H, d, $^2J_{ae}$ = 12.2, $^3H_{a(e)}$, CH_2); 3.85 (2H, s, OMe); 6.45-7.37 (7H, m, Ar); 9.22 (1H, s, NH). Mass spectrum, *m/z*: 322 [M^+]. Found, %: C 74.69; H 6.58; N 8.60. $C_{20}H_{22}N_2O_2$. Calculated, %: C 74.54; H 6.82; N 8.68.

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