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UNEXPECTED 1:2 ANNELATION OF 4,5,6,7-TETRAHYDROINDOLE WITH 1-BENZOYL-2-PHENYLACETYLENE

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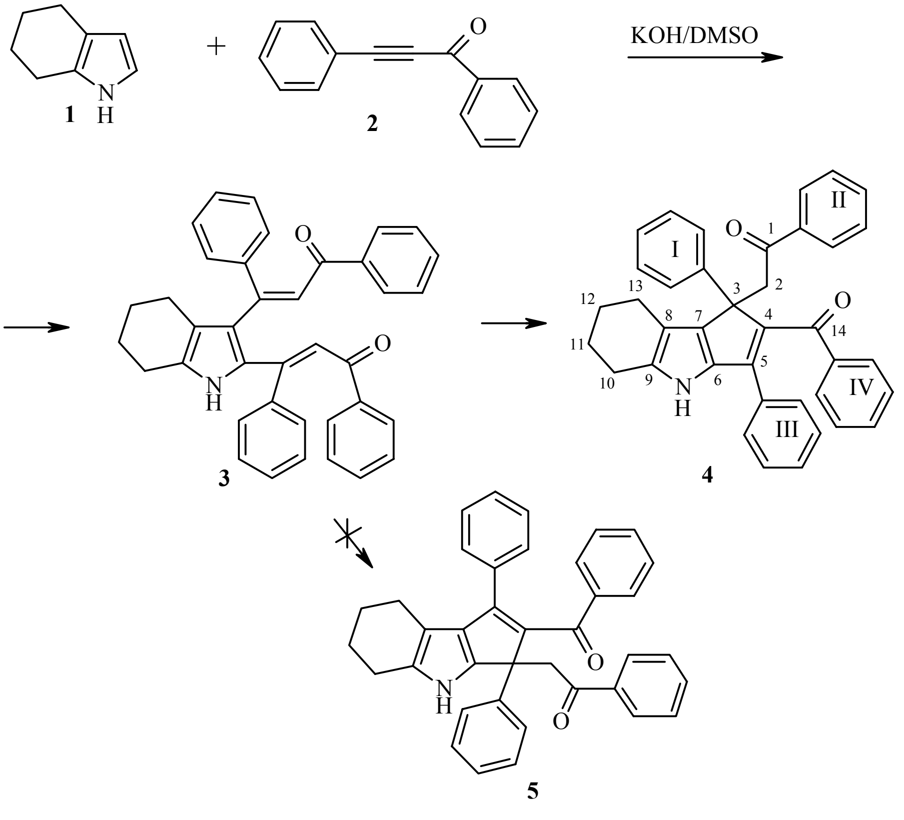
Addition of 4,5,6,7-tetrahydroindole (**1**) to benzoylacetylene in the superbasic KOH–DMSO system leads selectively to C-adducts. The analogous reaction with 1-benzoyl-2-phenylacetylene (**2**) yields an equilibrium mixture of N-and C-adducts [1].

We found that mono products are not the only products of the last reaction. By chromatographing the reaction mixture, we could isolate in 24% yield the product obviously formed upon intramolecular cyclization of the bis adduct 2,3-bis(2-benzoyl-1-phenylethenyl)-4,5,6,7-tetrahydroindole (**3**): 2-benzoyl-1-phenacyl-1,3-diphenyl-1,4,5,6,7,8-hexahydrocyclopenta[*b*]indole (**4**).



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The divinylpyrrole **3** itself was not detected in the reaction products. The choice between alternative structures for its cyclization (pyrroles **4** and **5**) was made based on two-dimensional ^{1}H and ^{13}C spectroscopy data (COSY, NOESY, HSQC, HMBC).

Reaction of 4,5,6,7-Tetrahydroindole with 1-Benzoyl-2-phenylacetylene. A mixture of pyrrole **1** (0.303 g, 2.5 mmol), KOH (0.140 g, 2.5 mmol) and acetylene **2** (0.520 g, 2.5 mmol) were stirred in DMSO (10 ml) for 3 h at ~20°C. The reaction mixture was diluted with a 20% NaCl solution. The precipitate formed was separated by filtration and fractionated by chromatography on a column with Al_2O_3. Upon elution with hexane, we isolated 0.460 g (56%) mixtures of N- and C-(1-benzoyl-2-phenylethenyl)pyrroles, the spectral characteristics of which match those given in [1]. Then by elution with ether, we isolated 0.160 g (24%) of 2-benzoyl-1-phenacyl-1,3-diphenyl-1,4,5,6,7,8-hexahydrocyclopenta[*b*]indole (**4**), yellow crystals; mp 154-155°C. ^{1}H NMR spectrum (250 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 7.76 (2H, m, *o*-H Ph-II); 7.67 (1H, br. s, NH); 7.45 (3H, m, *o*-H Ph-I, *p*-H Ph-II); 7.33 (4H, m, *m*-H Ph-I, Ph-II); 7.16 (3H, m, *p*-H Ph-I, *o*-H Ph-IV); 6.99 (6H, m, Ph-III, *p*-H Ph-IV); 6.86 (2H, m, *m*-H Ph-IV); 4.77, 4.21 (2H, dd, *J* = 13.23, $2CH_2$); 2.55 (2H, m, 10-CH_2); 2.41 (2H, m, 13-CH_2); 1.70 (2H, m, 11-CH_2); 1.59 (2H, m, 12-CH_2). ^{13}C NMR spectrum (62 MHz, $CDCl_3$), δ, ppm: 198.90 ($C_{(1)}$), 192.39 ($C_{(14)}$), 146.87 ($C_{(5)}$), 141.74 (*i*-C Ph-II), 141.26 ($C_{(4)}$), 140.97 ($C_{(7)}$), 140.86 (*i*-C Ph-IV), 139.00 (*i*-C Ph-I), 136.51 ($C_{(9)}$), 135.93 ($C_{(6)}$), 134.04 (*i*-C P-III), 129.86 (*p*-C Ph-IV), 128.93 (*o*-C Ph-IV), 128.23 (*p*-C Ph-III), 127.97 (*m*-C Ph-III), 128.83 (*o*-C Ph-III), 128.23 (*m*-C Ph-I), 132.03 (*p*-C Ph-II), 128.06 (*m*-C Ph-II), 127.81 (*o*-C Ph-II), 127.04 (*m*-C Ph-IV), 126.36 (*p*-C Ph-I), 126.06 (*o*-C Ph-I), 115.65 ($C_{(8)}$), 55.86 ($C_{(3)}$), 42.91 ($C_{(2)}$), 23.88 ($C_{(10)}$), 23.13 ($C_{(12)}$), 22.69 ($C_{(11)}$), 22.02 ($C_{(13)}$). Found, %: C 85.15; H 6.05; N 2.44. $C_{38}H_{31}NO_2$. Calculated, %: C 85.52; H 5.86; N 2.62.

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