CYCLOADDITION OF PHENYL AZIDE TO DIACETYLENE

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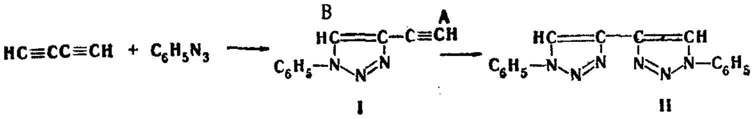
It is known [1] that bromomagnesium derivatives of diacetylenes react smoothly with aliphatic and aromatic azides to give ethynyltriazoles. For the first time we have subjected diacetylene itself to reaction with phenyl azide and have shown that cycloaddition takes place readily in benzene at 20-70°C. Depending on the amount of phenyl azide used, two compounds are obtained. Thus we obtained 1-phenyl-4(5)-ethynyl-1,2,3-triazole (I), with mp 75-76°C (sublimation), in 63% yield. IR spectrum: 3300 (≡CH); 3150, 3070 (=CH); 2125 (C≡C); 1630 (N=N); 1600 cm^{-1} (Ph). PMR spectrum (in CD_3OD): 4.35 (1H, s, A-H), 7.97 (1H, s, B-H), and 7.55 ppm (Ph, m). We also obtained 4,4 -bis(1-phenyl-1,2,3-triazolyl) (II), with mp 245-247°C (from benzene), in 55.5% yield. IR spectrum: 3140, 3110 (=CH); 1640 (N=N); 1600 cm^{-1} (Ph). PMR spectrum (in DMSO): 8.23 (=CH, d) and 7.23 ppm (Ph, m). The results of elementary analysis for C, H, and N were in agreement with the calculated values.



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