AN UNUSUAL REACTION OF 1,1-DICYANO-2,2-BIS(TRIFLUOROMETHYL)ETHYLENE

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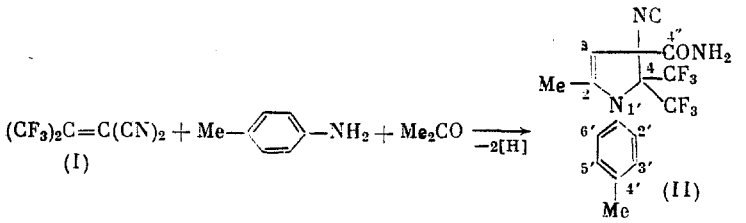
The reaction of 1,1-bis(trifluoromethyl)-2,2-dicyanoethylene (I) have not been studied sufficiently [1, 2]. We have found that the reaction of alkene (I) with an equimolar amount of p-toluidine and anhydrous acetone in Freon-113 at 20°C over 30 min leads to the formation of pyrroline (II) isolated in 49% yield. Alkene (I) is apparently a dehydrating agent [2] and the use of a two-fold excess of the compound permits an increase in the yield of (II) to 70%.



2-Methyl-1-(p-tolyl)-5,5-bis(trifluoromethyl)-4-cyano-2-pyrroline-4-carboxamide (II) was obtained as white crystals with mp 146-147°C, R_f 0.75 (10:1 $CHCl_3$—acetone on Silufol). ^{13}C NMR spectrum in DMSO-d_6 (δ, ppm, J, Hz): 155.0 ($C^{4''}$), 140.0 (C^2, $^2J_{C^2-H^3}$ = 6.0, $^2J_{C-Me}$ = 4.0, 139.0 ($C^{4'}$, $J_{C-H^{2,6}}$ = 6.5, J_{C-CH_3} = 6.5), 132.5 ($C^{1'}$, $J_{C^{1'}-H^{3',5'}}$ = 8.5), 129.7 ($C^{3',5'}$, ^1J = 163.0, J_{C-CH_3} = 6.0, $J_{C^{3'}-H^{5'}}$ = 6.0), 128.5 ($C^{2',6'}$, ^1J = 164.5, $J_{C^{2'}-H^{6'}}$ = 5.5), 123.0 (CF_3, $^1J_{C-F}$ = 285), 117.4 (CN), 88.0 (C^3, ^1J = 165.0, br. m), 51.0 (C^5, $^2J_{C-F}$ = 29.4), 45.0 (C^4, br), 19.0, 19.2 (Me). PMR spectrum in acetone-d_6 (δ, ppm, J, Hz): 7.33 m ($H^{2',6'}$), 7.17 m ($H^{3',5'}$), 5.30 (NH_2), 4.70 (CH, $^3J_{H-Me}$ = 1.2), 2.30 ($Me^{4'}$), 1.60 (Me^2). ^{19}F NMR spectrum in acetone-d_6 (δ, ppm): −5.4 s. Mass spectrum, m/z (rel. intensity, %): 361 $[M - NH_2]^+$ (4.1), 332 $[M - NH_2 - CO]^+$ (0.4), 291 $[M - NH_2 - CF_3]^+$ (100), 91 $[C_7H_7]^+$ (16.4), 65 $[C_5H_5]^+$ (13.5). IR spectrum (ν, cm^{-1}): 3480, 3404 (NH_2), 2192 (NC), 1692 (CO). Found: C, 51.30; H, 3.42; N, 11.23%. Calculated for $C_{16}H_{13}N_3F_6O$: C, 51.00; H, 3.45; N, 11.14%.

LITERATURE CITED

1. W. J. Middleton, J. Org. Chem., 1402 (1965).
2. W. J. Middleton and E. M. Bingham, J. Fluor. Chem., 397 (1982).

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