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LETTERS TO THE EDITOR

SYNTHESIS OF N-(2-HYDROXYETHYL)PIPERIDINES AND N-(2-HYDROXYETHYL)PERHYDROQUINOLINES

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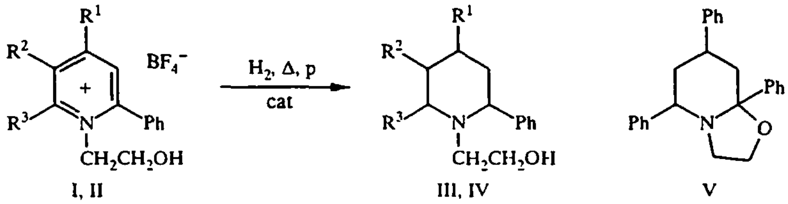
Cyclization products, namely, perhydrooxazolo[3,2-*a*]pyridines were obtained in the catalytic hydroethanolamination of acyclic and semicyclic 1,5-diketones instead of the expected N-(2-hydroxyethyl)piperidines and N-(2-hydroxyethyl)-perhydroquinolines [1].

We are the first to report that the hydrogenation of pyridinium tetrafluoroborate (I) and 5,6,7,8-tetrahydroquinolinium tetrafluoroborate (II) under heterogeneous catalysis conditions at 120°C and 10 MPa using an Ni/Ru catalyst gives N-(2-hydroxyethyl)-2,4,6-triphenylpiperidine (III) and N-(2-hydroxyethyl)-2,4-diphenylperhydroquinoline (IV) in 60-80% yield.



I, III $R^1 = R^3 = Ph$, $R^2 = H$; II, IV $R^1 = Ph$, $R^2 + R^3 = -(CH_2)_4-$

Products III and IV are colorless oils, which were purified and identified by thin-layer chromatography on Silufol UV-254 plates using 3:1:1 hexane–ether–acetone as the eluent. The hydrogenation of pyridinium salt I is complicated by a side-reaction leading to perhydrooxazolopyridine (V). The mixture of III and V was separated on an alumina column using 1:3 ether–hexane as the eluent.

The structures of III-V were established by IR spectroscopy on a Specord M-80 spectrometer in hexachlorobutadiene, Vaseline, or KBr pellets and NMR spectroscopy using a Varian FT-80 spectrometer at 80 MHz for the PMR spectra and 20 MHz for the ^{13}C NMR spectra in $CDCl_3$ using TMS as the internal standard. The ^{13}C NMR spectral data for IV indicate *cis* fusion of the carbocyclic and heterocyclic rings and axial orientation of the phenyl substituent at $C_{(2)}$.

This reaction is a convenient synthesis for N-(2-hydroxyethyl)piperidines and N-(2-hydroxyethyl)perhydroquinolines. The scope of this reaction is under investigation.

N-(2-Hydroxyethyl)-2,4,6-triphenylpiperidine (III) was obtained in 60% yield, R_f 0.245. IR spectrum: 3364 (OH), 3064, 3028 (ν-CH_{ring}), 2932, 2856 (CH_2), 762, 700 cm^{-1} (δ-CH_{ring}). PMR spectrum: 1.65 (1H, s, OH), 3.45 (2H + 2H, m, CH_2O, NCHPh), 2.42 (2H, t, $NC\underline{H}_2CH_2OH$), 1.94 (1H, m, 4-H), 1.08-1.19 (4H, m, 3-CH_2), 7.22-7.50 ppm (15H, m, Ph). Found: C, 84.31; H, 7.36; N, 4.12%. Calculated for $C_{25}H_{27}NO$: C, 84.03; H, 7.56; N, 3.92%.

N-(2-Hydroxyethyl)-2,4-diphenylperhydroquinoline (IV) was obtained in 80% yield, R_f 0.355. IR spectrum: 3416 (OH), 3064, 3068 (ν-CH_{ring}), 2928, 2860 (CH_2), 764, 700 cm^{-1} (δ-CH_{ring}). PMR spectrum: 1.65 (1H, s, OH), 3.48 (2H, t, CH_2O), 2.24 (2H, t, $NCH_2C\underline{H}_2OH$), 2.60 (3H, m, NCH, 4-CHPh), 1.25-2.00 (11H, m, 3-CH_2, 5-CH_2, 6-CH_2, 7-CH_2, 8-CH_2, 10-CH), 7.12-7.26 ppm (10H, m, Ph). ^{13}C NMR spectrum: 51.03 ($C_{(2)}$), 33.61 ($C_{(3)}$), 33.71 ($C_{(4)}$), 28.67 ($C_{(5)}$), 19.50 ($C_{(6)}$), 26.07 ($C_{(7)}$), 24.17 ($C_{(8)}$), 47.13 ($C_{(9)}$), 28.67 ($C_{(10)}$), 49.39 ($C_{(11)}$, $NCH_2\underline{C}H_2OH$), 61.10 ppm (COH). Found: C, 82.21; H, 8.76; N, 4.42%. Calculated for $C_{23}H_{29}NO$: C, 82.39; H, 8.66; N, 4.18%.

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5,7,8a-Triphenylperhydrooxazolo[3,2-*a*]pyridine(V) was obtained in 30% yield, R_f 0.845. IR spectrum: 3064, 3028 (ν-CH_{ring}), 2924, 2880 (CH_2), 1070 (COC), 762, 700 cm^{-1} (δ-CH_{ring}). Found: C, 85.22; H, 7.31; N, 4.13%. Calculated for $C_{25}H_{25}NO$: C, 84.51; H, 7.04; N, 3.94%.

REFERENCES

1. T. G. Nikolaeva, P. V. Reshetov, A. P. Kriven'ko, and V. G. Kharchenko, Khim. Geterotsikl. Soedin., No. 10, 1370 (1983).