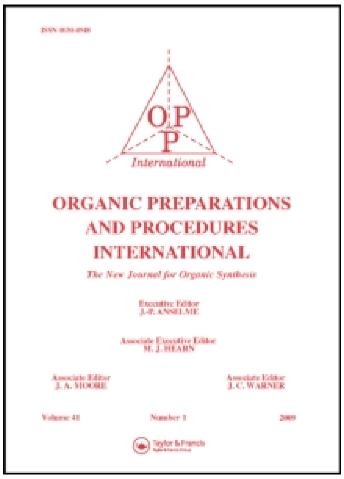

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A SYNTHESIS OF 5, 10-DIANISYLPERHYDROPHENAZINE

Jeremiah P. Freeman[a b]; Jacob Szmuszkovicz[a b]
[a] Department of Chemistry, University of Notre Dame, Notre Dame, IN [b] Research Laboratories, The Upjohn Company, Kalamazoo, MI

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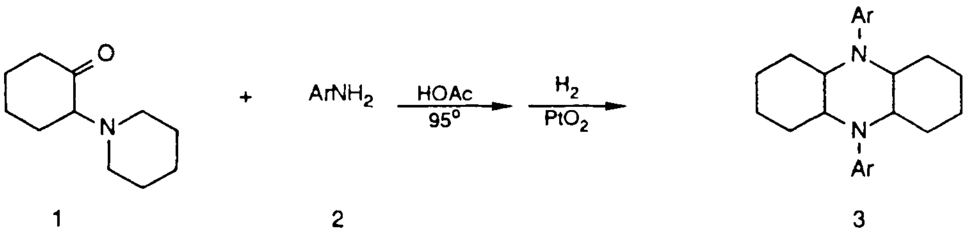
A SYNTHESIS OF 5,10-DIANISYLPERHYDROPHENAZINE

Submitted by (04/24/87)

Jeremiah P. Freeman* and Jacob Szmuszkovicz*

Department of Chemistry
University of Notre Dame
Notre Dame, IN 46556
and
Research Laboratories
The Upjohn Company
Kalamazoo, MI 49007

With a view to extending our studies of 1,2-cyclohexanediamine derivatives,[1] we attempted the reductive amination of 2-piperidinocyclohexanone (1) with p-anisidine. Instead of the expected diamine, we obtained a 27% yield of 5,10-dianisylperhydrophenazine 3 whose structure was assigned on the basis of the elemental analysis and its spectral properties. Although no authentic sample of a Schiff base of an α-aminoketone was found in the literature, phenylhydrazones have been reported;[2] on the other hand, some α-aminoketones have been reductively aminated successfully.[3] Perhydrophenazines, which are relatively rare compounds, have been prepared by hydrogenation of phenazine and some derivatives[4] and by the self-condensation of 1,2-cyclohexanediamine over a Ni-Al_2O_3 catalyst.[5]



Ar = p-$CH_3OC_6H_4$-

EXPERIMENTAL SECTION

Melting points were taken in capillary tubes and are corrected. Ultraviolet spectra were determined on a Cary Model 14 spectrophotometer, IR spectra on a Perkin-Elmer Model 421 spectrophotometer, mass spectra at 70 eV on an Atlas Model CH-4 spectrometer, and NMR spectra on a Varian Model A-60 spectrometer. NMR peaks are recorded in parts per million downfield from tetramethylsilane.

Tetradecahydro-5,10-bis(p-methoxyphenyl)phenazine (3).- A mixture of 21.75 g (0.12 mol) of 2-piperidinocyclohexanone,[6] 14.8 g (0.12 mol) of p-anisidine and 60 ml of acetic acid was heated on a steam bath under nitrogen for 22 hrs. The dark solution was cooled to room temperature, 1.2 g of platinum oxide was added and the mixture was shaken under an atmosphere of hydrogen (init. press. 50.5 psi). One equivalent of hydrogen was absorbed in 93 min. The resulting suspension was filtered and the product was separated from the catalyst by washing with methylene chloride. The bulk of the solvent was removed by evaporation and the residue crystallized slowly to yield 6.7 g (27%) of 3. Treatment of the material with Darco in methylene chloride followed by crystallization from CH_2Cl_2-CH_3OH gave a white solid, mp. 181-184°. Further crystallization from CH_2Cl_2-ether gave an analytical sample, mp. 183-185°.

Anal. Calcd. for $C_{26}H_{34}N_2O_2$: C. 76.81; H, 8.43; N, 6.89

Found: C, 76.89; H, 8.40; N, 6.99

The compound had the following spectral properties: mass spectrum, m/e 406 (M^+); UV mμ ($_{max}$): 225 (12,000), 258 (18,000); sh 285 (1980); 323 (2360); IR (Nujol) cm^{-1}: 2820, 2780, 1605, 1575, 1570, 1505, 1250, 1230, 1180, 1125, 1035, 805. ^1H NMR ($CDCl_3$): δ 0.8-2.0 (m, 16, CH_2), 3.45 (b, 4, NCH), 3.78 (s, 6, OCH_3), 6.9 (s, 8 aromatic).

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