This article was downloaded by:

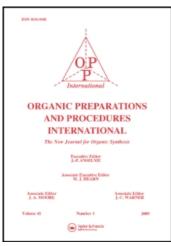
On: 27 January 2011

Access details: Access Details: Free Access

Publisher Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-

41 Mortimer Street, London W1T 3JH, UK



Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t902189982

N-METHYL OCTAHYDROCARBAZOLE

E. Schmitz^a; H. Fechner^a

^a Institut für Organische Chemie der Deutschen Akademie der Wissenschaften, Berlin-Adlershof, DDR

To cite this Article Schmitz, E. and Fechner, H.(1969) 'N-METHYL OCTAHYDROCARBAZOLE', Organic Preparations and Procedures International, 1:4,253-254

To link to this Article: DOI: 10.1080/00304946909458393 URL: http://dx.doi.org/10.1080/00304946909458393

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

N-METHYL OCTAHYDROCARBAZOLE

E. Schmitz and H. Fechner

Institut für Organische Chemie der Deutschen Akademie der Wissenschaften, Berlin-Adlershof, DDR

In an attempt to prepare the <u>bis</u>-enamine <u>l</u> from cyclohexanone and N,N'-dimethylhydrazine by the standard procedure of enamine synthesis, N-methyl octahydrocarbazole (<u>2</u>) was obtained in 80% yield. The preparation of pyrroles from ketazines (Piloty pyrrole synthesis²), requires drastic conditions, e.g., either in the presence of ZnCl₂ at 220-230° or with hydrogen chloride at 180°. Apparently, the presence of two methyl groups in the starting hydrazine permits the reaction to proceed under much milder conditions. The N-ethyl analogue was prepared in a similar fashion.

E. SCHMITZ AND H. FECHNER

EXPERIMENTAL

N-Methyl-1,2,3,4,5,6,7,8-octahydrocarbazole (2). A solution of 1.55 g (26 mmoles) of N,N'-dimethylhydrazine and 5.0 g (51 mmoles) of cyclohexanone in 10 ml of dry benzene is refluxed for 2 hrs. in the presence of 6 mg of p-toluenesulfonic acid. The benzene is removed in vacuo, and 5 ml of 2N sulfuric acid are added to the oily residue. The oil solidified and gave 3.95 g (80%) of crystals, m.p. 94° after recrystallization from alcohol-water, lit., 5 94.5°. The nmr spectrum showed signals at τ 6.75, 7.45-7.85, and 8.15-8.50 in the ratio of 3:8:8.

N-Ethyl-1,2,3,4,5,6,7,8-octahydrocarbazole. N,N'-Diethylhydrazine (3.2 g; 36 mmoles) and 7.15 g (73 mmoles) of cyclohexanone are dissolved in 15 ml of dry toluene and refluxed with 10 mg of p-toluenesulfonic acid for 3.5 hrs. Distillation of the reaction mixture gave a fraction boiling at $160-162^{\circ}/11_{\rm mn}$ which solidified to yield 4.2 g (58%) of crystals, m.p. $42-43^{\circ}$, lit., $6 +33^{\circ}$.

REFERENCES

- S. Hunig, E. Benzing and E. Lucke, Chem. Ber., <u>90</u>, 2833 (1957)
- 2. B. Robinson, Chem. Reviews, 63, 397 (1963).
- O. Piloty, Ber., 43, 489 (1910); A. N. Kost and I. I. Grandberg, Zhur. Obshchei Khim., 26, 565 (1956), Chem. Abs., 50, 11319i (1956).
- 4. G. M. Robinson and R. Robinson, J. Chem. Soc., 639 (1918).
- 5. S. G. P. Plant, ibid., 1595 (1930).
- 6. J. v. Braun and H. Ritter, Ber., <u>55</u>, 3792 (1922).

(Received May 28, 1969)