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Publisher *Taylor & Francis*

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Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

SYNTHESIS OF 6,7-BENZO-2-AZABICYCLO[3.2.0]HEPTAN-3-ONE

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To cite this Article Gomes, Ailton de Sousa and Figueiredo, A. M.(1973) 'SYNTHESIS OF 6,7-BENZO-2-AZABICYCLO[3.2.0]HEPTAN-3-ONE', *Organic Preparations and Procedures International*, 5: 1, 13 – 15

To link to this Article: DOI: 10.1080/00304947309356456

URL: <http://dx.doi.org/10.1080/00304947309356456>

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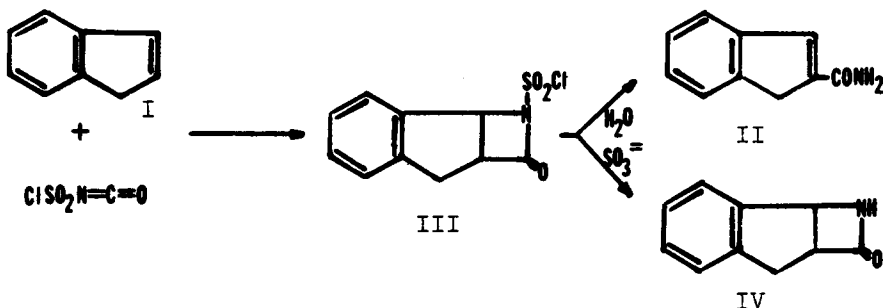
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SYNTHESIS OF 6,7-BENZO-2-AZABICYCLO[3.2.0]HEPTAN-3-ONE

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A substantial amount of the β -lactam of indene, 6,7-benzo-2-azabicyclo[3.2.0]heptan-3-one, was required for polymerization studies. A search of the literature indicated that this lactam had never been previously prepared. An excellent method for the preparation of β -lactams from olefins is the reaction of N-chlorosulfonyl isocyanate (NCSI) with olefins.¹ Graf has reported that indene (I) reacts with this reagent to give product II, isomeric with the β -lactam, upon hydrolysis of the N-chlorosulfonyl intermediate III.



The infrared spectrum of III exhibited a strong absorption at 1810 cm^{-1} ascribable to the carbonyl group of III. However, the infrared spectrum of the hydrolysis product did not show the expected band of a carbonyl of β -lactams in the region

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around 1750 cm^{-1} . We concluded that β -lactam was being destroyed during the hydrolysis of intermediate III. However, the method of Durst and O'Sullivan³ using a 20% aqueous solution of sodium sulfite at pH 7-8 gave the β -lactam of indene (IV) in 98% yield.

EXPERIMENTAL

N-Chlorosulfonyl-6,7-benzo-2-azabicyclo[3.2.0]heptan-3-one (III). - A 2 l. three-necked flask fitted with a gas inlet adapter, a magnetic stirring Teflon bar and a condenser, was immersed in a water bath at 20° and provided with a magnetic stirrer and charged with 1 l. of dried ethyl ether, 65.8 g. (0.56 mole) of pure indene and 80.1 g. (0.56 mole) of freshly distilled NCSI. The solution was stirred for 10 hrs. at 20° . After approximately 3 hrs. of reaction, a white solid began to precipitate. The end of the reaction was checked by TLC (silica gel). An IR (KCl) spectrum of the solid portion of this intermediate taken within a few minutes from the reaction mixture showed a strong carbonyl absorption band in the 1810 cm^{-1} region. This compound can be stored in the refrigerator without decomposition during a week.

6,7-Benzo-2-azabicyclo[3.2.0]heptan-3-one (IV). - Into a 4 l. beaker immersed in a water bath at 20° containing 300 ml. of CHCl_3 and 350 ml. of a 20% aqueous solution of Na_2SO_3 and provided with magnetic stirrer was slowly added the above mixture; the pH of the reaction mixture was kept between 7-8 by the addition of a 3N NaOH. The control of pH was made potentiometrically or with phenol-red as the indicator.

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During this reduction, some of the product started to precipitate. Evaporation of the organic layer gave a white solid, which was collected by filtration and washed with cold water until the washings were neutral. The white crude product was dried in vacuo at 60° to give 88.4 g. (98% yield) mp. 175-176°. An analytical sample was obtained by sublimation at 120°/10⁻⁴ mmHg, mp. 179-180°.

Anal. Calcd. for C₁₀H₉NO: C, 75.5; H, 5.7; N, 8.8

Found: C, 75.5; H, 6.0; N, 8.1

IR(KCl): 3170, 1730 and 1710 cm⁻¹; ms (70 ev), m/e 159 (M⁺), 116 (100%). Its nmr spectrum (Varian A60-A; TMS as internal reference in DMSO (d₆)) showed signals centered at δ 2.8-3.0 (t, 2H, CH₂), 3.8 (m, H, methine), 4.82 (d, H, benzylic), 7.25 (m, 4H, aromatic) and 8.3 (N-H); the coupling constants between the methine and methylene protons were determined by decoupling techniques and were found to be 8.5 cps (trans) and 4.5 cps (cis); the coupling constant between the benzylic and the methine protons was also 4.5 cps.

Acknowledgment. - Financial support from CAPES, BNDE and CNPq are gratefully acknowledged.

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(Received November 27, 1972; in revised form February 22, 1973)