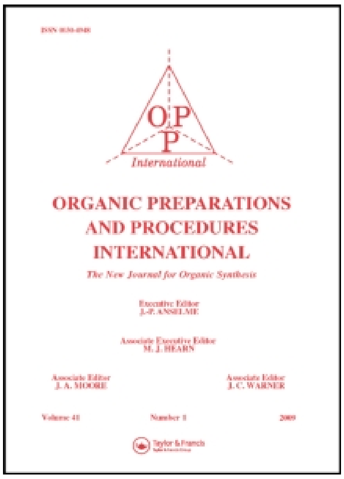


Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

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

Ailton de Sousa Gomes[a]; A. M. Figueiredo[a]
[a] Nucleo Macromolecular-NUMA Universidade Federal do Rio de Janeiro, Guanabara, BRASIL

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

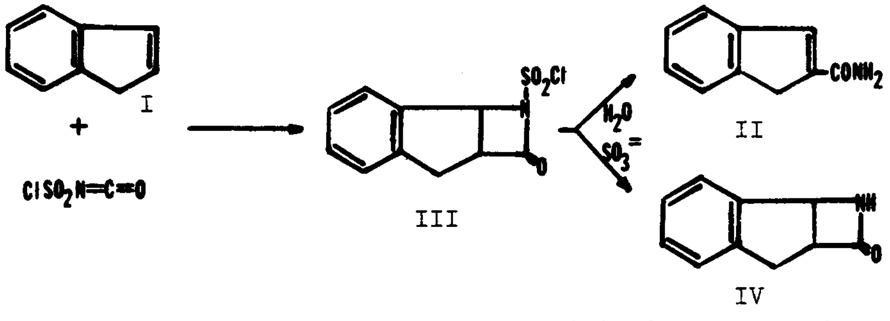
ORGANIC PREPARATIONS AND PROCEDURES INT. 5(1), 13-15 (1973)

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

Ailton de Sousa Gomes and A. M. Figueiredo

Nucleo Macromolecular-NUMA
Universidade Federal do Rio de Janeiro
Guanabara, BRASIL

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 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'Sullivan[3] 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.

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 $C_{10}H_9NO$: 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 (d_6)) showed signals centered at δ 2.8-3.0 (t, 2H, CH_2), 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.

REFERENCES

1. R. Graf, Ann., 661, 111 (1963).
2. R. Graf, Angew. Chem. internat. Edit., 7, 172 (1968).
3. T. Durst and M. J. O'Sullivan, J. Org. Chem., 35, 6 (1970).

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