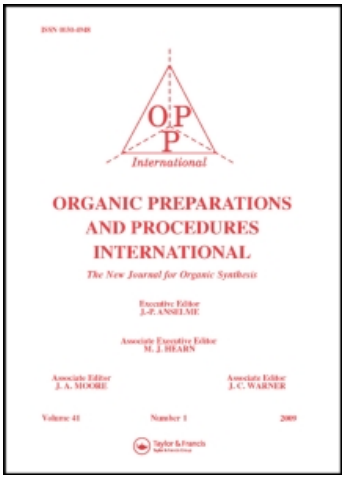


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# 2-INDENECARBOXAMIDE

D. Tabak[a]; P. A. M. de Oliveira[a]
[a] Nucleo Macromolecular, Universidade Federal do Rio de Janeiro, Rio de Janeiro, Brasil

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## REFERENCES

1. M. Tashiro, H. Watanabe and O. Tsuge, Org. Prep. Proced. Int., 6, 117 (1974).

2. M. S. Kharasch and B. S. Joshi, J. Org. Chem., 22, 1439 (1957).

3. O. Tsuge, M. Tashiro, and A. Torii, Kogyo Kagaku Zasshi, 70, 2289 (1967); C.A., 68, 10430 (1968).

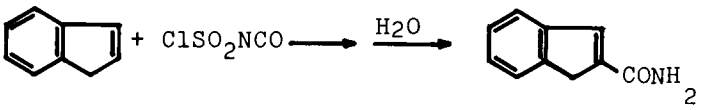


## 2-INDENECARBOXAMIDE

Submitted by D. Tabak* and P. A. M. de Oliveira† 7/6/76

Nucleo Macromolecular, Universidade Federal do Rio de Janeiro, C. P. 56020, Rio de Janeiro BRASIL

The title compound has been prepared by the direct hydrolysis of the product obtained from indene and chlorosulfonyl isocyanate (CSI).[1-3]




## EXPERIMENTAL

The infrared spectra were obtained as KBr pellets on a Perkin-Elmer spectrometer model 621 and the nmr spectra were determined in $CF_3CO_2H$ on a Varian XLFT-100 instrument while the mass spectra were run on a Varian instrument, model CH-5, at 70 eV.

To a solution of freshly distilled indene (10 ml; 854 mmoles) in 25 ml of dry ethyl ether, freshly distilled N-chlorosulfonyl isocyanate (7.5 ml; 854 mmoles) was added, under a dry nitrogen atmosphere. The exothermic reaction

maintained the ether at reflux. After stirring for 15 minutes a white solid precipitated. The reaction mixture was poured over a mixture of 200 ml of water and crushed ice and stirred at 60-70° until all the solid had dissolved. After cooling to room temperature, the reaction mixture was neutralized with calcium carbonate and kept overnight in the refrigerator. The white solid which formed was filtered and dried to give 9.5 g (70%) of crude 2-indenecarboxamide. Sublimation of this material at 150-60°/0.5 mm Hg, followed by recrystallization from 90% aqueous ethanol solution, yielded a product which melted at 197-198°.

Anal. Calcd for $C_{10}H_9NO$: C, 75.45; H, 5.70; N, 8.80.
Found: C, 75.20; H, 5.68; N, 8.85.

IR: 3380 and 3180 $cm^{-1}$ ($NH_2$), 1655-1645 $cm^{-1}$ (amide I and II) 750 $cm^{-1}$ (CH, o-dissubstituted aromatic); NMR: 3.3 (S, 2, $CH_2$), 7.0 (m, 4, ArH) and 7.7 (s, 1, =CH); MA: m/e 1599 ($M^{+\cdot}$), 116 (100%), 155, 89, 77, 65, 63 and 44.

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## REFERENCES

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1. R. Graf, Ann. Chem. 661, 111 (1963).
2. A. S. Gomes and A. M. Figueiredo, Org. Prep. Proc. Int., 5, 13 (1973).
3. E. Dunkelblum and A. Shaviv, Israel J. Chem., 10, 971 (1972).