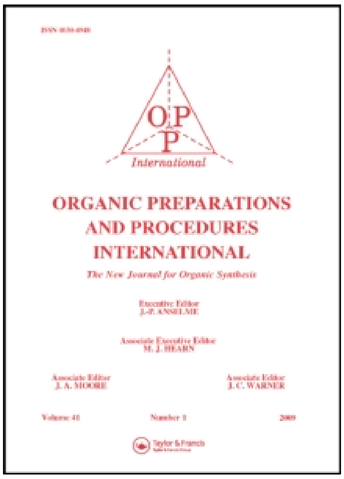


Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

PARTIAL TRANSALKYLATION OF 3,3',5,5'-TETRA-*t*-BUTYL-4,4'-DIHYDROXYBIPHENYL

M. Tashiro[a]; G. Fukata[a]
[a] Research Institute of Industrial Science, Kyushu University, Fukuoka, Japan

To cite this Article Tashiro, M. and Fukata, G.(1976) 'PARTIAL TRANSALKYLATION OF 3,3',5,5'-TETRA-*t*-BUTYL-4,4'-DIHYDROXYBIPHENYL', Organic Preparations and Procedures International, 8: 5, 241 — 243

To link to this Article: DOI: 10.1080/00304947609355634

URL: http://dx.doi.org/10.1080/00304947609355634

(500 ml) and ethanol (100 ml). Gentle heating of the mixture brought about an exothermic reaction and the resulting homogeneous solution was heated to 85° for 30 min, cooled, extracted with ether, the aqueous layer was acidified and the product was taken up in ether. The ethereal layer was dried, evaporated and the acid was crystallized from benzene to yield 103 g (87%), mp. 148-149°, lit.[1] mp. 146-147.5°.

REFERENCE

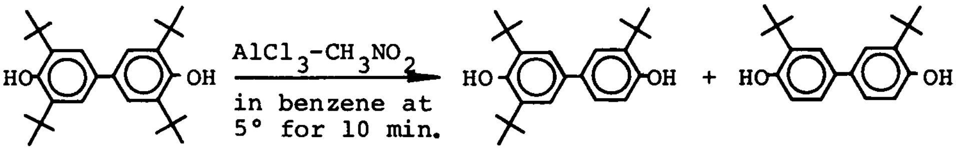
1. W. R. Purdum and K. D. Berlin, Org. Prep. Proced. Int., 7, 283 (1975).

PARTIAL TRANSALKYLATION OF 3,3',5,5'-TETRA-t-BUTYL-4,4'-DIHYDROXYBIPHENYL

Submitted by M. Tashiro* and G. Fukata
6/18/76

Research Institute of Industrial Science
Kyushu University
Hakozaki, Higashi-ku
Fukuoka 812
JAPAN

The partial transalkylation of the title compound (I) affords 3,3'-5-tri-t-butyl- (II) and 3,3'-di-t-butyl-4,4'-dihydroxybiphenyl (III) in 76% and 17% yields respectively.[1]



I II III

EXPERIMENTAL

All melting points are uncorrected. IR spectra were measured as KBr pellets on a Nippon Bunko IR-S spectrophotometer and NMR spectra were determined at 60 MHz with a Hitachi R-20 NMR spectrometer with TMS as an internal reference.

To a solution of 0.41 g (1 mmole) of 1[2] in 20 ml of benzene $AlCl_3$-CH_3NO_2 (0.3 g/0.6 ml) catalyst[3] was added at once at 5°. After the mixture was stirred for 5 min, it was quenched with 50 ml of 10% HCl. The organic layer was separated, dried over sodium sulfate and evaporated _in vacuo_ to leave the residue which was chromatographed on silica gel using benzene as an eluent affording 200 mg (73%) of II and 23 mg (7%) of III. II, mp. 183-184.5°, pale yellow needles (petroleum ether). Ir (KBr) cm^{-1}; 3640 (νOH). NMR ($CDCl_3$) δ ppm; 1.46 and 1.50 (27H, each s, $(C\underline{H}_3)_3$), 6.65 (1H, d, Jac = 8 Hz, aromatic proton), 7.23 (1H, dd, Jab = 2 Hz, Jac = 8 Hz, aromatic protons), 7.34 (2H, s, aromatic protons), and 7.45 (1H, d, Jab = 2 Hz, aromatic protons).

Anal. Calcd for $C_{24}H_{34}O_2$: C, 81.31; H, 9.67.
Found: C, 81.01; H, 9.69.

III, mp. 181-183.5°, colorless plates (petroleum ether). Ir (KBr) cm^{-1}; 3550, 3440 (νOH). NMR ($CDCl_3$) δ ppm; 1.45 (18H, s, _t_-$(C\underline{H}_3)_3$), 4.79 (2H, s, $O\underline{H}$), 6.67 (2H, d, Jab = 7.65 Hz, aromatic protons), 7.15 (2H, d, Jac = 2.25 Hz, aromatic protons) and 7.36 (2H, dd, Jab = 7.65 Hz, Jac = 2.25 Hz, aromatic protons.

Anal. Calcd for $C_{20}H_{26}O_2$: C, 80.54; H, 8.72.
Found: C, 80.31; H, 8.82.

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).

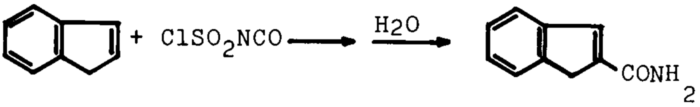
2-INDENECARBOXAMIDE

Submitted by 7/6/76

D. Tabak* and P. A. M. deOliveira†

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