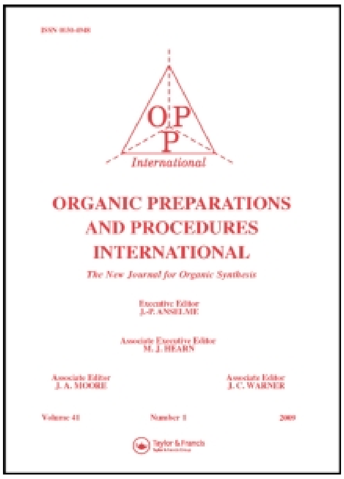

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A CONVENIENT METHOD FOR THE PREPARATION OF 2-AMINO-3',4,4',5-TETRACHLOROBIPHENYL

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sulfate and the solvent was removed in vacuo. The yellow syrup obtained was then extracted with six 50 ml portions of a 5:1 mixture of dichloromethane-carbon tetrachloride and 5 ml of water. The organic phase was dried and evaporated in vacuo to give white crystals. Recrystallization from hot ethyl acetate afforded 5.8 g (71%) mp. 155-157°.

Anal. Calcd. for $C_{14}H_{20}N_4O_2S$: C, 54.54; H, 6.49; N, 18.18.
Found: C, 54.75; H, 6.03; N, 18.72.

IR($CHCl_3$): 920, 1670, 1680 cm^{-1}

NMR ($CDCl_3$): 7.85 (s, 1H), 6.30 (m, 2H), 4.56 (s, 1H), 4.08 (m, 1H), 3.90 (s, 2H), 3.80 (m, 2H), 2.49 (s, 3H), 2.05 (m, 2H), 2.05 (s, 3H), 1.68 (s, 3H).

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A CONVENIENT METHOD FOR THE PREPARATION OF 2-AMINO-3',4,4',5-TETRACHLOROBIPHENYL

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2-Amino-3',4,4'5-tetrachlorobiphenyl (previously prepared in 24% yield by Hickmott and Hudson[1] from 3,3'-dichloro-6-nitrobenzidine) needed as a radioimmunoassay hapten, has been synthesized in 83% yield by the nitration of 3,3',4,4'-tetrachlorobiphenyl with nitric acid in acetic anhydride and subsequent reduction of the nitro compound by Sn (II)/HCl. The present method is not only more direct and selective but also gives a homogeneous compound. However, emulsion losses during work-up of the final product lowered the recovery to 48.5% overall relative to 3,3',4,4'-tetrachlorobiphenyl.

EXPERIMENTAL

Infrared spectra were measured in KBr discs on a Perkin Elmer 621 instrument. N.M.R. spectra were determined with a Varian XL-100 instrument in benzene-d_6 using TMS as internal standard. Gas chromatography (GC) analyses were carried out on a Varian model 2100 instrument using a 6'x 1/8" column packed with 4% SE-30 + 6% OV-210 on 80/100 Gas Chrome Q. Each run was programmed from 150°-220° at 10°/min. The carrier gas was nitrogen with a flow rate of 60 ml/min.

2-Nitro-3'-,4,4',5-Tetrachlorobiphenyl.- To a nitration mixture prepared by adding 29 ml of nitric acid slowly to 40 ml of acetic anhydride at 15-20°, cooled to 0°, was slowly added a suspension of 2.9 g (.01 mole) of 3,3',4,4'-tetrachlorobiphenyl; the temperature of the mixture was maintained at 0°. Then the mixture was heated slowly with vigorous stirring on a water bath; at 60° the solid material dissolved. The soln. was stirred at 60° for 5 mins. and then cooled and poured onto 200 g of ice. The precipitated solid, was filtered, washed several times with cold water, dried and recrystallized from ethanol yielding 3.10 g. (92.5%) mp. 86-88°, lit.[1] mp. 86-89°, of pure product. The structure and purity were confirmed by combined GC-mass spectrometry.

2-Amino-3',4,4',5-Tetrachlorobiphenyl.- To a solution of 2-nitro-3',4,4',5-tetrachlorobiphenyl (2.5 g.) in glacial acetic acid (120 ml) at 100° was added tin (II) chloride dihydrate (8.6 g.) in conc. hydrochloric acid (35 ml). The mixture was heated to reflux with stirring for 3 hrs and then allowed to stand overnight at room temperature. The solution was made basic with sodium hydroxide (1N) and extracted with two 100 ml portions of ether. The ethereal extracts were washed thoroughly with water and dried over sodium sulfate. Evaporation of the solvent *in vacuo* gave an oil which solidified on trituration with *n*-hexane. The resulting crude 2-amino-3',4,4',5-tetrachlorobiphenyl was recrystallized from aqueous ethanol affording 1.2 g. (52.5%), mp. 86°, lit.[1] mp. 79-82°, of pure compound. The purity was confirmed by GC to >98%.

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SULTAMS WITH POSSIBLE SCHISTOSOMICIDAL ACTIVITY. PART V†

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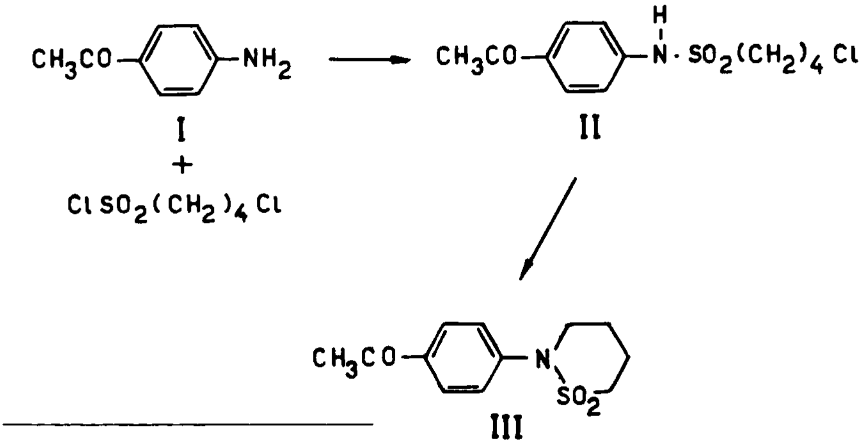
In continuation of our investigation of sultam derivatives with possible schistosomicidal activity, we have prepared compounds III-V from p-



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