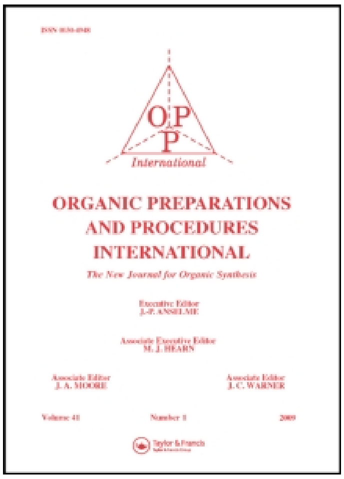

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POLY(2-VINYLPYRROLE)

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POLY(2-VINYLPYRROLE)

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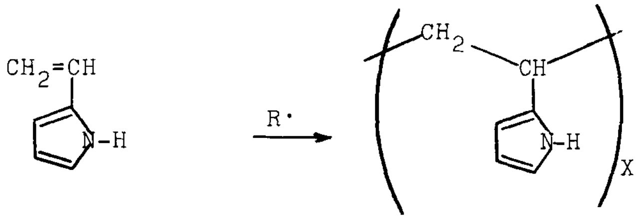

Monomeric 2-vinylpyrrole[2,3] was prepared in high purity by a modified Wittig reaction between methyltriphenylphosphonium bromide and 2-pyrrolecarboxaldehyde. This procedure is similar to that reported by Jones and Lindner.[3] The purified monomer could be homopolymerized by free radical or thermal initiation to give poly(2-vinylpyrrole) in high conversion.

Experimental

2-Vinylpyrrole. To 1000 ml. of anhydrous 1,2-dimethoxyethane under a nitrogen atmosphere was added 240 ml. (0.4 mole) of 1.67 M methyl lithium in ether. Methyl-triphenylphosphonium bromide (143 g., 0.4 mole) was added slowly over a period of 5-10 min. After stirring for 30 min., 25 g. (0.26 mole) of pyrrole-2-aldehyde[4] in 30 ml. of anhydrous benzene was added dropwise during a period of 10 min.

The solution was refluxed for 24 hr. and then poured into 1500 g. of ice. The two layers were separated and the aqueous layer was extracted with five 300 ml. portions of ether. The oil and ether extracts were combined and washed to neutrality with a 10% solution of sodium chloride.[5] The solution was then dried with anhydrous sodium sulfate, 10 mg. of hydroquinone was added, and the ether was removed by heating in vacuo. Upon distillation of the residue at 73-83°/24 mm. under a nitrogen atmosphere, 14.0 g. (57.2% yield) of crude 2-vinylpyrrole was obtained. Redistillation of the crude monomer with 10 mg. of hydroquinone gave 12.0 g. of purified 2-vinylpyrrole, m.p. 12°, b.p. 75-75.5°/20 mm. n_D^{25} 1.5672 (lit., b.p. 60-63°/10 mm.,[3] 64-67°/18 mm.,[2] n_D^{25} 1.5562[2]). The distilled product was colorless when stored under nitrogen, immediate yellow coloration forming on exposure to air.

Anal. Calcd. for C_6H_7N: C, 77.38; H, 7.58; N, 15.04. Found: C, 77.81; H, 7.43; N, 14.72.

Poly(2-vinylpyrrole). A solution of 1.09 g. of 2-vinylpyrrole and 14.0 mg. of azobisisobutyronitrile in 3 ml. of benzene was placed in a polymerization tube. The contents of the tube were frozen and flushed three times with nitrogen, and finally sealed under reduced pressure. Precipitation of the polymer occurred during the course of the reaction. The polymer was collected, dissolved in tetrahydrofuran, and precipitated into a ten-fold excess of pentane (61.4% conversion, $[\eta]^{30}$ = 0.3 (tetrahydrofuran).

Anal. Calcd. for C_6H_7N: C, 77.38; N, 15.04; H, 7.58. Found: C, 76.36; N, 14.43; H, 7.44.

References

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3. R. A. Jones and J. A. Lindner, Aust. J. Chem., 18, 875 (1965).
4. The aldehyde was prepared according to Organic Syntheses, Coll. Vol. IV, N. Rabjohn, ed., John Wiley and Sons, Inc., New York, N. Y., 1963, p. 831.
5. The sodium chloride was added to decrease the solubility of 2-vinylpyrrole in water.

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