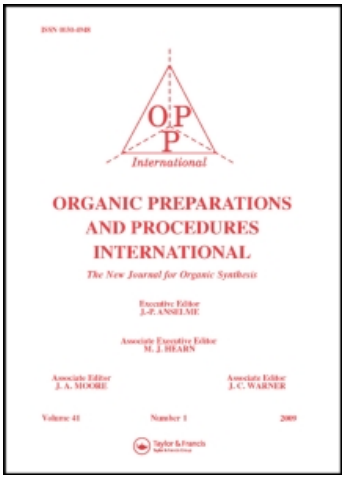

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Separation of cis- and trans-1,2-Cyclohexanediamines

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OPP REPORTS

(by John L. Ferrari, Associate Editor)

Separation of cis- and trans-1,2-Cyclohexanediamines

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A convenient method for the separation of cis- and trans-1,2-cyclohexanediamines (cis-I and trans-I) from a commercial mixture [1] of the isomers is a modification of the Smith procedure.[2]

Procedure

cis- and trans- Benzil Adducts (cis-II and trans-II). Equimolar amounts (1.0 mole) of a mixture of cis-I and trans-I and of benzil (1.0 mole)are refluxed in toluene solution (500 ml) until the theoretical amount of water has azeotroped. Recrystallization of the first crop from abs. EtOH (2.5 l.) yields 132 g. of trans-II, mp. 173-175° (lit.[2] 173-175°). The toluene filtrate is evaporated to ca. 250 ml., cooled to room temperature, filtered (residue discarded) and the filtrate is evaporated to dryness. Recrystallization from ethanol-chloroform (75:25) yields 44g. of cis-II (84% pure-nmr analysis). Allowing the filtrate to stand for a week yields and additional 4-5g. of cis-II (92% pure), mp. 131-134°

cis- and trans-Diamines (cis-I and trans-I). A mixture of 100g of trans-II, 125 ml. of conc. HCl and 350 ml. of water is heated on a steam bath for 0.5 hr. and cooled to ca. 70°C. Benzil is removed by filtration, the

filtrate is reduced to one-half its volume and made strongly basic with sodium hydroxide pellets. The solution is extracted with two 100 ml. portions of methylene chloride, the extracts are dried over potassium carbonate, filtered, and evaporated to dryness to give 32-35g. of trans-I (98.4% pure by gc analysis[1]). The product readily forms a carbonate in air and is more conveniently stored as the dihydrochloride salt, mp. 336-338° (lit.[3] 338-339°).

Similarly, cis-II (92% pure) is converted to cis-I (90.5% pure). Dihydrochloride salt, mp. 308-311° (lit.[3] 312-314°).

References

1. Aldrich Chemical Co., 66% trans-, 29% cis- and 5% unidentified matter (by gc). Column: 1/8 in x 15 ft. packed with 10% DC-710 on Chromosorb W pre-treated with KOH.

2. A.I. Smith, U.S. Patent 3,163,675 (1964); Chem. Abstr., 62, 7656f (1965).

3. G. Swift and D. Swern, J. Org. Chem., 32, 511 (1967).

Diphenyllead Dicarboxylates

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The preparation of several additional diphenyllead dicarboxylates according to a previously described procedure is reported. The following table lists the yields obtained and some physical properties.