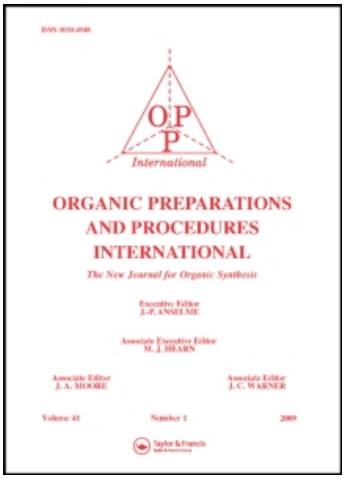

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A SIMPLIFIED PREPARATION OF *CIS*-1,3-CYCLOPENTANEDICARBOXYLIC ACID

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A SIMPLIFIED PREPARATION OF CIS-1,3-CYCLOPENTANEDICARBOXYLIC ACID

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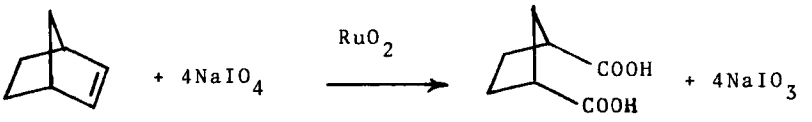
The oxidation of norbornene to cis-1,3-cyclopentanedicarboxylic acid in 75%-95% yield using sodium permanganate has been reported by Birch, Oldham and Johnson.[1] The procedure requires the use of carbon dioxide as a buffer, sulfur dioxide to dissolve the manganese dioxide, and is generally messy. Furthermore, sodium permanganate is relatively expensive. Ozone has also been used successfully,[2] but frequently gives mixed isomers, and many laboratories are not set up to handle ozone properly. Other common oxidizing agents such as potassium permanganate and acetic acid with 30% hydrogen peroxide are reported to produce little or none of the desired dicarboxylic acid[1].

We now report a simple preparation of cis-1,3-cyclopentanedicarboxylic acid using a stoichiometric quantity of sodium metaperiodate catalysed by ruthenium dioxide or ruthenium chloride to oxidize norbornene. The procedure is easy to



carry out, gives the product in 80-90% yield, and is less expensive than the earlier procedures. Furthermore, the reaction is free from significant by-products, giving an 80-90% yield of the desired product (based on the oxidizing agent) even when only one half or one fourth of the stoichiometric quantity of oxidizing agent is employed. It is probable that the yield could be increased further by adding excess sodium metaperiodate to the reaction mixture, but the toxicity hazard caused by the presence of ruthenium tetroxide produced by the excess sodium metaperiodate does not make this modification worthwhile.

EXPERIMENTAL

To 100 ml. of chloroform in a three liter flask placed in a hood, was added 9.4 g (0.1 M) of norbornene and 0.2 g of hydrated ruthenium chloride (Fisher Scientific Company) or 0.2 g of ruthenium dioxide recovered from a previous oxidation. The mixture was stirred with a glass-enclosed magnetic stirrer while 85.6 g (0.4M) of sodium metaperiodate dissolved in 800 ml of water were slowly added. The flask was stoppered and stirring was continued with occasional shaking. The reaction was allowed to proceed until the black precipitate of ruthenium dioxide that collected on the upper portions of the flask did not dissolve on vigorous shaking. Two or three days were usually required depending upon the effi-

ciency of the stirring.

The layers were separated and filtered to remove the black ruthenium dioxide precipitate, which was saved for reuse. The aqueous layer was extracted with ether in a continuous extractor for 48 hrs. The product was obtained by evaporating the ether. Recrystallization from benzene-ether gave 13.1 g (83%), MP. 120-121°, (lit.[1,3] MP. 119.9-120.6°, [3]120-121°).

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REFERENCES

1. S.F. Birch, W.J. Oldham, and E.A. Johnson, J. Chem. Soc., 818-820 (1947).

2a. M.I. Fremery and E.K. Fields, U.S. Patent. 3,284,492. Assigned to Standard Oil Co. (Indiana).

b. P.S. Bailey, U.S. Patent 3,238,250. Assigned to Esso Research and Engineering Co.

c. S.C. Temin and M.E. Baum, U.S. Patent 3,126,410. Assigned to Koppers Co., Inc.

3. J.D. Roberts, C.C. Lee, and W.H. Saunders, J. Am. Chem. Soc., 76, 4501-4510 (1954).

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