

Sodium Metaperiodate: a Mild Oxidizing Agent for the Generation of Di-imide from Hydrazine

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Summary Di-imide, generated by the sodium metaperiodate oxidation of hydrazine is a particularly useful reducing system for compounds which contain readily oxidized functional groups.

RECENTLY, we had to convert the thiophen derivative (I) into its dihydro-analogue (II) by reduction. Catalytic procedures were ineffective presumably because of catalyst poisoning by (I). Di-imide reductions, carried out by oxidation of hydrazine with hydrogen peroxide¹ or ferricyanide,² while effective in reducing the double bond of (I) also caused oxidation of the sulphide function to its corresponding sulphone (III). Other non-oxidative methods of generating di-imide proved to be either difficult experimentally or not amenable to use on a small scale.³

In looking for other mild oxidative methods of generating di-imide from hydrazine, our attention was drawn to a report of the potassium periodate oxidation of substituted hydrazines.⁴ We found that sodium metaperiodate ef-

ficiently converts hydrazine into di-imide in a variety of aqueous organic solvent systems.

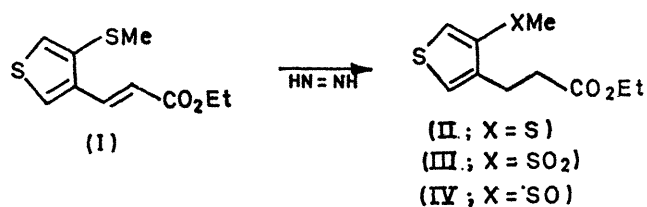
The use of sodium metaperiodate and hydrazine permits the quantitative di-imide reduction of thiophen (I) into (II) without concomitant formation of either sulphone (III) or sulfoxide (IV). A variety of other unsaturated compounds has been reduced by this method (Table). In a

TABLE

Substrate	Product	Yield (%)
Azobenzene	Hydrazobenzene	95
Acenaphthylene	Acenaphthene	91
Cinnamic acid	3-Phenylpropionic acid	87
Ethyl cinnamate	3-Phenylpropionic acid	80
Phenylpropionic acid	3-Phenylpropionic acid	85
Maleic anhydride	Succinic anhydride	95

typical procedure, 2.5 mmol of sodium metaperiodate in 5 ml of water was added dropwise (1 h) to a mixture of olefin

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(0.5 mmol), hydrazine hydrate (100%, 20 mmol), 2 drops of saturated copper sulphate, and 2 drops of glacial acetic acid dissolved in dimethyl sulphoxide (3 ml).[†]

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[†] Tetrahydrofuran, acetic acid, dioxan, ethanol, methanol, and glyme have been used as solvents for these reactions.

¹ E. J. Corey, W. L. Mock, and D. J. Pasto, *Tetrahedron Letters*, 1961, 347.

² S. Hunig and H. R. Muller, *Angew. Chem.*, 1962, **74**, 215.

³ For an excellent review on all aspects of di-imide reductions see, C. E. Miller, *J. Chem. Educ.*, 1965, **42**, 254.

⁴ D. J. Cram and J. S. Bradshaw, *J. Amer. Chem. Soc.*, 1963, **85**, 1108.