

A NEW METHOD FOR DEOXYGENATION OF VICINAL DIOLS #

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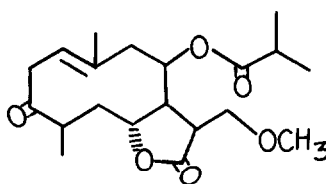
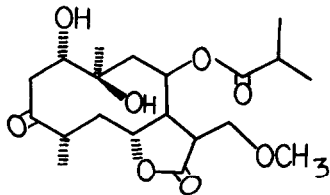
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Summary: Cis and trans vicinal diols have been converted into olefins in one step reaction with chlorotrimethylsilane and sodium iodide.

Procedures exist in literature¹⁻⁹ for the conversion of vicinal diols to olefins but most of them require prior formation of a suitable derivative or employ costly reagents and the stringent reaction conditions lead to poor yields. Barton's bis-xanthate method¹⁰ provides alkenes at low temperature under mild neutral conditions using inexpensive reagents. Garegg *et al.*¹¹ have converted vicinal diols to corresponding olefins in one step but the method is applicable only to trans-1,2 diols. Besides, these methods were designed primarily to convert bis secondary vicinal diols to the corresponding olefins. We now describe one step method for transforming both cis and trans secondary-tertiary vicinal diols to olefins in excellent yields under mild neutral conditions (Table - I).

Table-I. Conversion of vicinal diols to olefins

Substrate ^a	Olefins ^a	Amount (m mol) ^b of NaI	Time/ rt ^c in min	Yield ^d (%)
Cholestan-5 α , 6 α -diol	Cholest-5-ene	1.0	30	96
Cholestan-5 α , 6 β -diol	Cholest-5-ene	1.6	20	82
3 β -Hydroxycholestan-5 α , 6 α -diol	Cholesterol	1.4	25	80
3 β -Hydroxycholestan-5 α , 6 β -diol	Cholesterol	1.0	30	95
3 β -Methoxycholestan-5 α , 6 α -diol	3 β -Methoxycholest-5-ene	1.2	10	98
3 β -Methoxycholestan-5 α , 6 β -diol	3 β -Methoxycholest-5-ene	1.5	15	95



2.0 5 90

- a) All compounds mentioned in Table-I gave satisfactory analysis, IR, NMR and mass spectral data.
 b) In every case 0.5 m mol of substrate and 1.0 m mol of chlorotrimethylsilane was used.
 c) rt is room temperature.
 d) Yields of the isolated products of > 90% purity as determined by t.l.c., IR and NMR spectroscopy.

In a typical experiment a solution of the diol (0.5 m mol) in dry acetonitrile (2 ml) is treated with a solution of sodium iodide (1.0 m mol) dissolved in 2 ml acetonitrile at room temperature and the mixture stirred for 5 minutes. Chlorotrimethylsilane (1.0 m mol) is then added to the above mixture and the stirring is continued at room temperature till the reaction is completed (monitored on t.l.c.).

Acknowledgement : We are indebted to Dr.J.N. Baruah, Director, Regional Research Laboratory, Jorhat for providing necessary facilities for this work.

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#/ Dedicated to Prof. W. Herz of Department of Chemistry, The Florida State University, Tallahassee, U.S.A. on his 60th birthday.

(Received in UK 27 January 1982)