

SODIUM PERCARBONATE: A CONVENIENT REAGENT FOR EFFICIENTLY OXIDIZING ORGANOBORANES

George W. Kabalka*, Prakash P. Wadgaonkar, and Timothy M. Shoup

Departments of Chemistry and Radiology, University of Tennessee, Knoxville, TN 37996.

Abstract: Sodium percarbonate, a readily available, inexpensive and easy to handle reagent, efficiently oxidizes organoboranes. The yields of alcohols are essentially identical to those obtained using standard oxidation procedure.

The hydroboration of alkenes followed by *in situ* oxidation of the organoboranes formed is a very convenient procedure for the *anti*-Markovnikov hydration of double bonds.¹ The traditional method for oxidizing organoboranes involves heating the boron reagent with 30% hydrogen peroxide and 3 N sodium hydroxide at 50°C.² The other reagents which have been employed for the oxidation of organoboranes to the corresponding alcohols are amine N-oxides,^{3,4} perbenzoic acid,⁵ sodium hypochlorite⁶ and sodium perborate.⁷

Sodium percarbonate⁸ ($\text{Na}_2\text{CO}_3 \cdot 3/2\text{H}_2\text{O}_2$) is a very inexpensive large scale industrial chemical which is extensively used in the detergent industry as a bleaching agent.⁹ Surprisingly, there has been very little study of the possible applications of this stable and easily handled oxidant to organic synthesis. To our knowledge, it has only been used for the oxidation of sulfides¹⁰ and amines¹¹ and in the epoxidation reactions of alkenes.¹⁰

We wish to report that sodium percarbonate efficiently oxidizes a variety of organoboranes to the corresponding alcohols. A series of representative alkenes were hydroborated with borane-tetrahydrofuran complex and the organoboranes produced were oxidized to the corresponding alcohols using sodium percarbonate as the oxidizing agent. A comparative study was performed on the oxidation of organoboranes by the standard peroxide oxidation procedure. The yields of product alcohols in sodium percarbonate procedure were essentially identical to those obtained using the standard oxidation procedure. The results are presented in Table I.

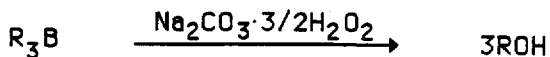


Table I. Comparison of the Efficiencies of Sodium Percarbonate and Hydrogen Peroxide Oxidation Procedures a,b,c

Alkene	Product	Relative Amounts, % ^d		Total Yield, % ^e	
		Sodium Percarbonate	Hydrogen Peroxide	Sodium Percarbonate	Hydrogen Peroxide
1-Hexene	1-Hexanol	94	94	100	100
	2-Hexanol	6	6		
2-Methyl-1-pentene	2-Methyl-1-pentanol			99	99
<i>trans</i> -3-Hexene	3-Hexanol			99	99
Cyclohexene	Cyclohexanol			97	98
Norbornene	<i>exo</i> -Norborneol			96	98
Styrene	2-Phenylethanol	85	85	100	100
	1-Phenylethanol	15	15		
1-Methylcyclopentene	<i>trans</i> -2-Methylcyclopentanol	.99 ^f	.99 ^f	100	100

^aThe organoboranes were prepared by the addition of BH₃-THF (3 mmol) to the alkene (9 mmol) dissolved in 3 mL of THF at 0°C. After an appropriate interval, the reaction mixture was oxidized.

^bThe oxidation was carried out by adding water (3 mL) and a stoichiometric quantity of Na₂CO₃·3/2H₂O₂ (3 equiv. of H₂O₂/R₃B). The mixture was stirred at 50 °C for 1 h, saturated with potassium carbonate and the product extracted into ether. ^cThe peroxide oxidations were carried out by adding hydrogen peroxide (3 equiv.) as a 30% aqueous solution and 1 equiv. of sodium hydroxide (3 N) to the organoborane and heating to 50°C for 1 h. ^dBy GLC analysis. ^eBy GLC analysis using an internal standard. ^fLess than 1% *cis* isomer.

In conclusion, sodium percarbonate is a very useful reagent for efficiently oxidizing organoboranes. Its stability, commercial availability and ease of handling should make sodium percarbonate a popular reagent for oxidizing organoboranes. The application of sodium percarbonate oxidation to a variety of organoborane synthesis is currently under investigation.

Acknowledgement This work was supported by the Department of Energy (DE-FG05-85ER60434).

REFERENCES

- Pelter, A.; Smith, K.; Brown, H. C. *Borane Reagents*, Academic Press Inc. (Best Synthetic Methods): New York, 1988.
- Zweifel, G.; Brown, H. C. *Organic Reactions* 1963, 13, 1.
- Kabalka, G. W.; Hedgecock, H. C. *J. Org. Chem.* 1975, 40, 1776.
- Koster, R.; Morita, Y. *Angew. Chem. Intern. Ed.* 1966, 5, 580.
- Johnson, J. R.; Van Campon, M. A. *J. Am. Chem. Soc.* 1938, 60, 121.
- Brown, H. C. U.S. Patent, 3,439,046, April 15, 1969.
- Kabalka, G. W.; Shoup, T. M.; Goudgaon, N. M. *Tet. Lett.* 1989, 30, 1483.
- This material is available from Fluka.
- Das, T. K.; Mandavawalla, A. K.; Datta, S. K.; *Colourage* 1983, 301, 25.
- Ando, T.; Cook, D. G.; Kimura, T. *Chem. Lett.* 1986, 665.
- Zajac, W. W.; Walters, T. R.; Woods, J. M. *Synthesis* 1988, 808.

(Received in USA 27 June 1989)