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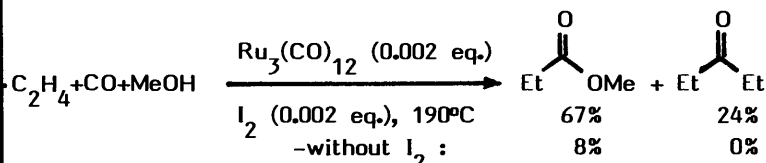
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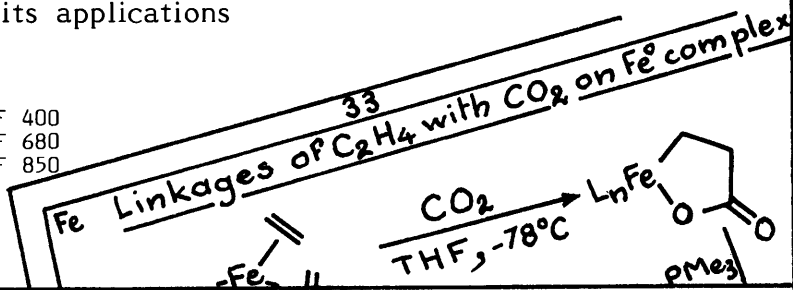
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Ru Hydroesterification and hydroacylation of olefins.



HIDAI, M.; KOYASU, Y.; CHIKANARI, K. and UCHIDA, Y.
J. Mol. Catal., 1987, 40 (2), 243-254.

Ru Cationic metallocyclophanes.



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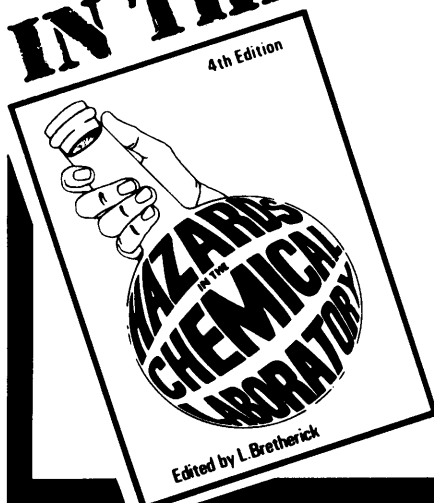
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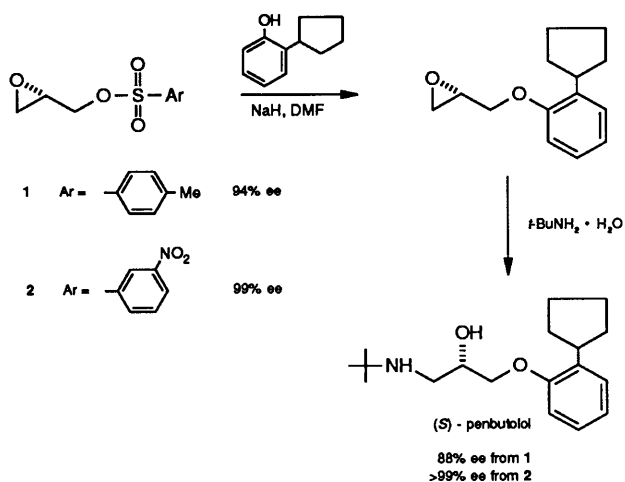
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The Sharpless Asymmetric Epoxidation

"Everything you ever wanted to know . . ."

If you are planning to run an asymmetric epoxidation (AE) you should read and probably commit to memory the recent publication from the Sharpless research group at MIT.¹ This meticulously crafted paper presents the first full account of the group's work on catalytic AE² (*i.e.*, epoxidation with 5-10 mole % Ti-tartrate in the presence of molecular sieves), nicely contrasting results from a variety of substrate allylic alcohols with the stoichiometric protocol.^{3,4} The paper also provides the real "do's and don't's" of this reaction with an in-depth discussion of important factors that influence the rate and enantioselectivity of the process, namely, catalyst stoichiometry, substrate concentration, catalyst aging, the use of molecular sieves, and the choice of oxidant and oxidation solvent. For example, Professor Sharpless now recommends the use of anhydrous isooctane (2,2,4-trimethylpentane) solutions of *tert*-butyl hydroperoxide (TBHP), which we offer in 3.0*M* and 5.5*M* concentrations,⁵ over the traditional dichloromethane or toluene solutions. Everything you ever wanted to know about catalytic asymmetric epoxidation is contained in the text, experimental section and footnotes of this paper!

A significant advantage to using catalytic AE is that the low catalyst concentration makes *in situ* derivatization of the epoxy alcohol products by sulfonylation or acylation preparatively feasible. Thus, low-molecular-weight epoxy alcohols can now be isolated in greatly improved yields as crystalline sulfonate and *p*-nitrobenzoate (PNB) esters, which can be enantiomerically enriched by recrystallization. As indicated below, these derivatized epoxy alcohols are important chiral building blocks (*e.g.*, β -blocker synthons). Noteworthy in this scheme is the use of the



new glycidyl sulfonate 2 which, unlike tosylate 1, can be recrystallized to >99% ee and shows greatly improved rate and selectivity in the aryl oxide displacement step providing the eutomer of penbutolol in >99% ee.^{6,7}

References and Notes:

- (1) Gao, Y.; Hanson, R.M.; Klunder, J.M.; Ko, S.Y.; Masamune, H.; Sharpless, K.B. *J. Am. Chem. Soc.* **1987**, *109*, 5765. (2) Hanson, R.M.; Sharpless, K.B. *J. Org. Chem.* **1986**, *51*, 1922. (3) Katsuki, T.; Sharpless, K.B. *J. Am. Chem. Soc.* **1980**, *102*, 5974. (4) For reviews on the stoichiometric AE, see: Finn, M.G.; Sharpless, K.B. In *Asymmetric Synthesis*; Morrison, J.D., Ed.; Academic Press: New York, 1985; Vol. 5, Chapter 8, p 247. Rossiter, B.E. *ibid.* Chapter 7, p 193. Pfenniger, A. *Synthesis* **1986**, 89. (5) Professor Sharpless prefers the 5.5*M* concentration for reasons contained in ref. 1, p 5770. (6) Klunder, J.M.; Onami, T.; Sharpless, K.B., manuscript in preparation. Sulfonate 4 shows similar improvements in the synthesis of (*S*)-propranolol and (*S*)-timolol. (7) The synthesis of (*S*)-propranolol with tosylate 2 is described in Klunder, J.M.; Ko, S.Y.; Sharpless, K.B. *J. Org. Chem.* **1986**, *51*, 3710.

Glycidyl sulfonates

30,051-9	(2 <i>R</i>)-(-)-Glycidyl tosylate,* 99%	1g \$16.45
30,052-7	(2 <i>S</i>)-(+)-Glycidyl tosylate,* 99% (1)	1g \$16.45
NEW 32,798-0	(2 <i>R</i>)-(-)-Glycidyl 3-nitrobenzenesulfonate, †	99%
		1g \$23.00
NEW 32,799-9	(2 <i>S</i>)-(+)-Glycidyl 3-nitrobenzenesulfonate, †	99% (2)
		1g \$23.00

* Licensed under U.S. Patent 4,471,130. † U.S. Patent applied for.

Glycidyl *p*-nitrobenzoates

30,043-8	(2 <i>R</i>)-(-)-Glycidyl 4-nitrobenzoate, 98%	1g \$16.45
30,044-6	(2 <i>S</i>)-(+)-Glycidyl 4-nitrobenzoate, 98%	1g \$16.45
30,575-8	(2 <i>R</i>)-(-)-2-Methylglycidyl 4-nitrobenzoate, 98%	1g \$19.00
30,576-6	(2 <i>S</i>)-(+)-2-Methylglycidyl 4-nitrobenzoate, 98%	1g \$19.00
30,045-4	(2 <i>R</i> ,3 <i>R</i>)-(+)-3-Methylglycidyl 4-nitrobenzoate, 98%	1g \$15.50
30,046-2	(2 <i>S</i> ,3 <i>S</i>)-(-)-3-Methylglycidyl 4-nitrobenzoate, 98%	1g \$15.50
30,053-5	(2 <i>R</i>)-(+)-3,3-Dimethylglycidyl 4-nitrobenzoate, 98%	1g \$14.80
30,054-3	(2 <i>S</i>)-(-)-3,3-Dimethylglycidyl 4-nitrobenzoate, 98%	1g \$14.80

These products are licensed under U.S. Patent 4,471,130.

Reagents for catalytic AE

NEW 31,037-9	<i>tert</i> -Butyl hydroperoxide, anhydrous, 3.0 <i>M</i> solution in 2,2,4-trimethylpentane	25g \$8.30
		100g \$19.90; 500g \$68.20
NEW 33,127-9	<i>tert</i> -Butyl hydroperoxide, anhydrous, 5.5 <i>M</i> solution in 2,2,4-trimethylpentane	25g \$17.50; 100g \$49.00
20,527-3	Titanium(IV) isopropoxide	100g \$13.05; 500g \$17.05; 2kg \$51.75
22,780-3	Diisopropyl D-tartrate, 99%	10g \$16.45; 50g \$54.20
22,918-0	Diisopropyl L-tartrate, 99%	25g \$13.75; 100g \$36.70
23,364-1	Molecular sieves, 3A, powder, undried	500g \$14.00; 1kg \$22.90; 2.5kg \$45.25
23,366-8	Molecular sieves, 4A, activated powder	500g \$15.00; 1kg \$24.50; 2.5kg \$48.55
23,367-6	Molecular sieves, 5A, powder, undried	500g \$15.10; 1kg \$24.60; 2.5kg \$48.65



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