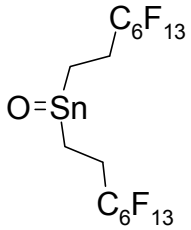
PRODUCT APPLICATION NOTE: Fluorous Tin Oxide as Catalyst in sulfonation of 1,2-diols

F026051

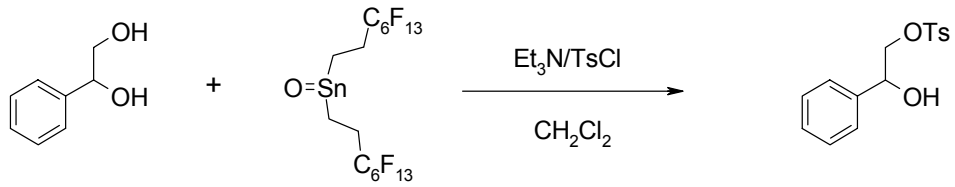


Bis(1H,1H,2H,2H-perfluorooctyl)tin oxide

Chemical Formula:	$C_{16}H_8F_{26}OSn$
Formula Weight:	828.89
Description:	Tin Compound
CAS Number:	324063-66-3
Appearance:	White solid
Soluble in:	Ether, acetone, methylene chloride
Stability:	Stable at room temps, but storage in brown bottle under N_2 is recommended

DESCRIPTION AND USES:

- Selective sulfonylation of 1,2-diols using tin oxide as a catalyst has been reported by Martinelli and co-workers.[1]
- The above fluorous tin oxide was used in selective sulfonylation of 1,2-diols with the advantage that the fluorous tin oxide can be recycled and reused easily using fluorous solid phase extraction (F-SPE).[2,3,4]



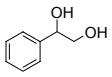
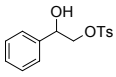
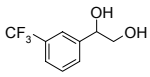
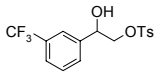
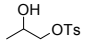
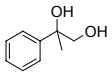
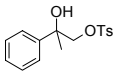
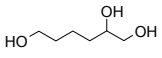
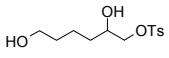
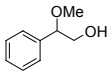
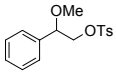
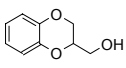
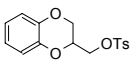
1 **2** 2% (cat. amount) **3**

TYPICAL PROCEDURE: [2]

1-Phenyl-1,2-ethane diol **1** (1 mmol) was dissolved in CH_2Cl_2 (5 mL). Triethylamine (1 mmol) and tin oxide **2** (0.02 mmol) were added. Tosyl chloride was added and the solution was stirred at room temperature for 50 min. Water (1mL) was added and the aqueous layer was washed with dichloromethane (2x10 mL). The combined organic layers were washed with H_2O (2x25 mL) and brine (2x25 mL). The organic layer was dried over $MgSO_4$. Removal of the solvent yielded a mixture of **3** and tin oxide. The crude mixture was transferred to a fluorous cartridge (Fluoro*Flash*®). The column was washed with a mixture of 9:1 MeOH-H_2O (3 mL), followed by THF (3 mL). Evaporation of the MeOH-H_2O fraction yielded **3**. To retrieve the catalyst, evaporate the THF fraction. Some representative examples are shown in **Table 1**.

Additional fluorous tin oxides, having different fluorous chain lengths, are available. For a complete listing, please see our catalog.

Table 1: Monotosylation of alcohols with 2% fluorous tin oxide

Entry	Substrate	Reaction Time[a]	Structure of monotosylate	Yield of monotosylate[b]
1[c]		1-2		75-80%
2		1		77%
3		1		72%
4		1		70%
5		1		74%
6		23		84%
7		23		94%

a. Reactions were followed by TLC
b. Isolated yields of purified product
c. Reaction time and yield ranges for multiple runs with recovered tin oxide

REFERENCES:

1. a) Martinelli, M. J.; Nayyar, N. K..; Moher, E.D.; Dhokte, U.P.; Pawlak, J.M.; Vaidyanathan, R. *Org. Lett.* **1999**, *1,* 447. b) Martinelli, M.. J.; Vaidyanathan, R.; Van Khau, V. *Tetrahedron Lett.* **2000**, *41*, 3773.
2. Bucher, B. and Curran, D. P. *Tetrahedron Lett.* **2000**, *41*, 9617.
3. Curran, D. P. *Synlett* **2001**,1488.
4. Please refer to FTI Application Note Fluorous Solid Phase Extraction.

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