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Regioselective Conversion of *O*-Protected Glycidols to Fluorohydrins Catalyzed by Tetrabutylammonium Dihydrogentrifluoride Under Solid-Liquid PTC Conditions

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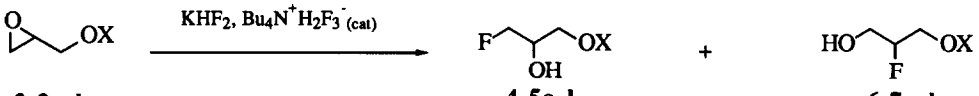
Abstract. A number of *O*-protected glycidols are regioselectively converted into the corresponding fluorohydrins $FCH_2CH(OH)CH_2OX$ by reaction with catalytic amounts of $Bu_4N^+H_2F_3^-$ and a molar excess of KHF_2. Most of the protective groups (X) examined are stable under the above conditions, moreover stereogenic carbons are not affected.

In a previous paper[1] we showed that tetrabutylammonium dihydrogentrifluoride (**1**) is a very efficient catalyst for the regio- and stereoselective hydrofluorination of epoxides, affording good or excellent yields of fluorohydrins in the presence of a molar excess of KHF_2 under solid-liquid phase transfer catalysis (SL-PTC) conditions. Moreover, the extraordinary importance of glycidol (**2**) and *O*-protected glycidols **3** in organic synthesis was recently stressed by Hanson.[2] The fluorohydrins deriving from **2** and **3** represent versatile starting materials for obtaining more complex organofluorine compounds *via* selective conversion of free and protected hydroxy groups.



2,3a-l **4,5a-l** **6,7a-l**

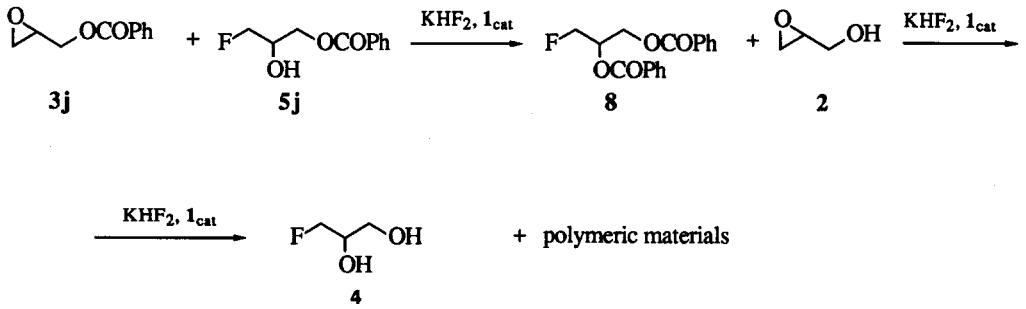
	2,4,6	a	b	c	d	e	f
X	H	Me	Bn	Tr	Allyl	Ph	4-ClC_6H_4
	g	**h**		**i**	**j**	**k**	**l**
X	MEM	$PhCH_2OCH_2$		THP	PhCO	Ms	Ts

Scheme 1

In the present paper we report a systematic study of hydrofluorination of **2** and a series of *O*-protected derivatives **3a-l** catalyzed by **1** under SL-PTC conditions, in order to define the stability of the protective groups, and the regiochemistry of the reaction (Scheme 1).

RESULTS AND DISCUSSION

The reaction (Scheme 1) was performed by stirring, at 120°C, a heterogeneous mixture of the substrate **2,3a-l** (1 mol), the PTC agent **1** (0.1 mol) and solid KHF_2 (2 mol) without solvent,[3] until complete conversion of the substrate was reached (TLC and/or GLC analyses). In 3-60h, under these conditions (Table 1), *O*-protected glycidols **3a-i** afforded the corresponding fluorohydrins **5a-i** and **6a-i** in 62-90% yield and with excellent stability of the most protecting groups examined. The hydrofluorination of glycidyl benzoate (**3j**) gave 3-fluoro-1,2-propanediol 1-*O*-benzoate (**5j**) in 45% yield together with 3-fluoro-1,2-propanediol 1,2-di-*O*-benzoate (**8**) (17%) and traces (≤2%) of 3-fluoro-1,2-propanediol (**4**), the remainder being polymeric material. Compound **8** is most likely formed *via* an acid catalyzed transesterification reaction between the starting epoxide **3j** and the initially formed fluorohydrin **5j** (Scheme 2).



3j **5j** **8** **2** **4**

Scheme 2

The presence of glycidol **2**, detected during the process, together with the other products cited above, accounts for this rationale. On the other hand, transesterification reactions in *O*-acylglycidols[2,4] and *O*-acylglicerols[5] were previously reported. Moreover, the hydrofluorination of glycidol (**2**) as such gave mainly polymeric products and only minor amounts of 3-fluoro-1,2-propanediol (**4**) (33%). Higher yields (47%) of **4** were obtained when the reaction was carried out at 80°C instead of 120°C.

According to the good nucleofugality of mesyl and tosyl groups, also towards quaternary ammonium polyhydrogenfluorides,[6] glycidyl mesylate (**3k**) and tosylate (**3l**) were found to be largely unstable under the above hydrofluorinating conditions. Indeed, from the reaction mixture of **3k** only 5% of 3-fluoro-1,2-propanediol 1-*O*-mesylate (**5k**) was obtained, whereas from **3l** 6% of 3-fluoro-1,2-propanediol 1-*O*-tosylate (**5l**) was isolated.

Table 1. Fluorohydrins **4-7** Prepared by Hydrofluorination of *O*-Protected Glycidols **2,3**.[a]

entry	starting epoxide	t,h[b]	yield,%[c]	fluorohydrins		
1	**2**	2.5	33[d]	**4**	$HOCH_2CH(OH)CH_2F$	
2	**2**	12.5	38[e]	**4**		
3	**2**	30	47[f]	**4**		
4	**3a**	3	74	**5a**	$MeOCH_2CH(OH)CH_2F$	
5	**3b**	6	74	**5b**	$BnOCH_2CH(OH)CH_2F$	96[g]
				7b	$BnOCH_2CHFCH_2OH$	4[g]
6	**3c**	60	84[h]	**5c**	$TrOCH_2CH(OH)CH_2F$	
7	**3d**	6	64	**5d**	$AllylOCH_2CH(OH)CH_2F$	
8	**3e**	8	90	**5e**	$PhOCH_2CH(OH)CH_2F$	
9	**3f**	8	62	**5f**	$4\text{-}ClC_6H_4OCH_2CH(OH)CH_2F$	
10	**3g**	14	63	**5g**	$MEMOCH_2CH(OH)CH_2F$	
11	**3h**	24	77	**5h**	$PhCH_2OCH_2OCH_2CH(OH)CH_2F$	94[g]
				7h	$PhCH_2OCH_2OCH_2CHFCH_2OH$	6[g]
12	**3i**	6	76	**5i**	$THPOCH_2CH(OH)CH_2F$	97[g]
				7i	$THPOCH_2CHFCH_2OH$	3[g]
13	**3j**	7.5	45	**5j**	$PhCOOCH_2CH(OH)CH_2F$	
			17	**8**	$PhCOOCH_2CH(OCOPh)CH_2F$	
14	**3k**	8	5	**5k**	$MsOCH_2CH(OH)CH_2F$	
15	**3l**	8	6	**5l**	$TsOCH_2CH(OH)CH_2F$	
16	**3m**	60	83	**5m**	$(+)\text{-}(2R)\text{-}TrOCH_2CH(OH)CH_2F$	

[a] Reaction conditions: epoxide **2,3a-l** (1 mol), $Bu_4N^+H_2F_3^-$ (**1**) (0.1 mol), KHF_2 (2 mol), 120°C. [b] Time for the complete conversion of the substrate **2,3a-l**. [c] Isolated yields. [d] At 120°C. [e] At 100°C. [f] At 80°C. [g] Distribution % of the two regioisomers **5** and **7**. [h] In the presence of PhCl as solvent.

Hydrofluorination of optically pure (+)-(2*R*)-[(triphenylmethoxy)methyl]oxirane (**3m**),[7] promoted by **1**, afforded optically pure (+)-(2*R*)-1-fluoro-3-(triphenylmethoxy)propan-2-ol (**5m**) in 83% yield, showing that the stereocenter was not affected. The optical purity of **5m** was determined by ^{19}F NMR (Fig. 1). The ^{19}F spectrum registered after treatment of (±)-1-fluoro-3-(triphenylmethoxy)propan-2-ol (**5c**) with (+)-(*S*)-2-methoxy-2-phenyl-2-trifluoromethyl acetic acid chloride ("Mosher's chloride") (Fig. 1a) showed two symmetrical signals: (A) at -224.139 and (B) at -224.603 ppm (dt, $^3J_{HF}$ = 19.2, $^2J_{HF}$ = 46.0) due to FCH_2. In the case of **5m** the spectrum of "Mosher's esters" exhibited only a multiplet at -224.603 ppm (Fig.1b).

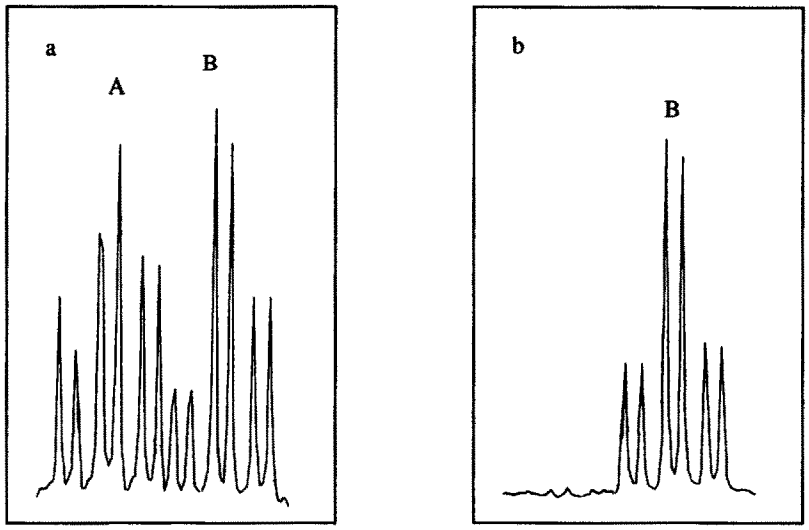


Fig. 1. ^{19}F NMR spectra of "Mosher's" esters of compounds **5c** (a) and **5m** (b).

Thus due to the recent commercial availability of both the enantiomers of glycidol (**2**) we can easily synthesize optically active polyfunctionalized building blocks bearing a fluorine atom and a stereocenter of known configuration.

As shown in Table 1, the hydrofluorination generally proceeds with high (entries 5, 11 and 12) or complete (entries 1-4, 6-10 and 13-16) regioselectivity. The fluorohydrins **5**, deriving from the attack of fluoride ion to the less substituted, and hence the less hindered, carbon atom of the oxirane ring, were the sole or the most prevalent regioisomers obtained. This regioselectivity is, we found, similar to that reported by Poulter *et al.*[8] and *Schlosser et al.*[9] in the reaction between epoxides and diisopropylamine trishydrofluoride (**9**)[8] and "Hunig's hydrofluoride", the hydrogen fluoride/ethyldiisopropylamine adduct (**10**),[9] respectively.

As already reported,[1] the reactions promoted by tetrabutylammonium dihydrogentrifluoride (**1**) are completely *trans*-stereoselective like those promoted by "Hunig's hydrofluoride" **10**.[9] Thus on the basis of this common regio- and stereochemical behaviour we can reasonably assume that in the hydrofluorinations with reagents **1** and **10** the mechanism proposed by Schlosser,[9] is operating: i.e. the addition of hydrogen fluoride proceeds in an anti-periplanar manner *via* a "conveyer belt process" where a polyhydrogenfluoride species, such as $H_2F_3^-$ or $(HF)_3$, is directly involved in the transition state.

Differently from the HF/amine adducts, such as **9** and **10**, tetrabutylammonium dihydrogentrifluoride (**1**)[10] can be advantageously used in catalytic amounts, since it can be regenerated *in situ* by solid potassium hydrogendifluoride *via* a well known SL-PTC process.[14] Moreover, **1** is an excellent non-corrosive source of hydrogen fluoride and, if the presence of moisture is avoided, it can be used in normal pyrex vessel.

EXPERIMENTAL

Starting *O*-protected glycidols **3a**, **3d**, **3e**, **3f** are commercially available. Products **3b**,[15] **3c**,[16] **3g**,[17] **3h**,[18] **3i**,[19] **3j**,[20] **3k**,[21] **3l**,[22] and **3m**[16] are known compounds and were prepared by literature methods. Tetrabutylammonuim dihydrogentrifluoride (**1**) was synthesized from the corresponding hydrogensulphate, as previously reported.[13] Potassium hydrogendifluoride was used as purchased. ^1H NMR and ^{19}F NMR spectra were recorded in $CDCl_3$ at 300 Mhz and 282 Mhz, respectively, using TMS for the ^1H- and $CFCl_3$ for the ^{19}F NMR spectra as external standards. The values of coupling costants are in Hz.

General Method for the Preparation of Fluorohydrins 4,5,7

A mixture of the epoxide **2,3** (10 mmol), KHF_2 (20 mmol) and tetrabutylammonium dihydrogentrifluoride (**1**) (1 mmol) is stirred at 120°C until the starting material is no longer detectable (GLC, NMR and/or TLC analyses). The reactions of **3c** and **3m** are conducted in the presence of PhCl (2 mL/10 mmol) as a solvent. After cooling, the crude is diluted with CH_2Cl_2 (30 mL), filtered on celite and the solvent is evaporated under reduced pressure. The residue is purified by distillation under vacuum or by flash or medium pressure liquid chromatography (MPLC) on silica gel (230-400 mesh).

Starting epoxide, reaction time, chromatographic eluant, yield and physical, spectroscopic and analytical data of fluorohydrins **4,5,7** are as follows.

3-Fluoro-1,2-propanediol (4). Glycidol (**2**); 2.5h; Et_2O. **4**, 33%; at 80°C **4** is obtained in 47% yield, after 30h; n_D^{26} 1.4221; bp 52-55°C/0.2 Torr (lit.[23] bp 55°C/0.2 Torr); ^1H NMR, δ, 3.58 (m, 2H), 3.81 (dm, 1H, $^3J_{HF}$ = 19.0), 4.06 (bs, 2H), 4.35 (dm, 2H, $^2J_{HF}$ = 47.6); ^{19}F NMR, δ, -226.1 (dt, $^3J_{HF}$ = 18.9, $^2J_{HF}$ = 47.7).

1-Fluoro-3-(methoxy)propan-2-ol (5a). [(Methoxy)methyl]oxirane (**3a**); 3h; the crude is distilled. **5a**, 74%; n_D^{20} 1.4022; bp 90-95°C/30 Torr; ^1H NMR, δ, 2.75 (s,1H), 3.34 (s, 3H), 3.42 (m, 2H), 3.95 (dm, 1H,

$^3J_{HF}$ = 21.0), 4.39 (dm, 2H, $^2J_{HF}$ = 48.0); ^{19}F NMR, δ, -226.6 (dm, $^2J_{HF}$ = 48.0). *Anal. calcd.* for $C_4H_9FO_2$: C, 44.43; H, 8.39. *Found*: C, 44.50; H, 8.33.

1-Fluoro-3-(phenylmethoxy)propan-2-ol (5b) and 2-fluoro-3-(phenylmethoxy)propan-1-ol (7b). [(Phenylmethoxy)methyl]oxirane (**3b**); 6h; Et_2O and petroleum ether (PE) (1 : 2). **5b**, 70%; n_D^{20} 1.5032 (lit.[9] n_D^{20} 1.5042); ^{1}H NMR, δ, 2.55 (bs, 1H), 3.59 (m, 2H), 4.06 (dm, 1H, $^3J_{HF}$ = 18.4), 4.48 (dm, 2H, $^2J_{HF}$ = 47.3), 4.58 (s, 2H), 7.31 (m, 5H); ^{19}F NMR, δ, -225.5 (dt, $^3J_{HF}$ = 19.2, $^2J_{HF}$ = 50.7). *Anal. calcd.* for $C_{10}H_{13}FO_2$: C, 65.19; H, 7.13. *Found*: C, 65.30; H, 7.02. **7b**, 4%; oil; ^{1}H NMR, δ, 2.50 (bs, 1H), 3.73 (dd, 2H, J_{HH} = 4.6, $^3J_{HF}$ = 20.5), 3.86 (dd, 2H, J_{HH} = 4.5, $^3J_{HF}$ = 23.0), 4.60 (s, 2H), 4.74 (dm, 1H, $^2J_{HF}$ = 48.1), 7.31 (m, 5H); ^{19}F NMR, δ, -189.7 (dm, $^2J_{HF}$ = 48.1).

1-Fluoro-3-(triphenylmethoxy)propan-2-ol (5c). [(Triphenylmethoxy)methyl]oxirane (**3c**); the reaction is conducted for 60h using PhCl as solvent (2 mL); Et_2O and PE (1 : 5). **5c**, 84%; mp 93-94°C; ^{1}H NMR, δ, 2.38 (s, 1H), 3.28 (d, 2H, J_{HH} = 5.4), 4.00 (dm, 1H, $^3J_{HF}$ = 20.5), 4.51 (dm, 2H, $^2J_{HF}$ = 48.0), 7.32 (m, 15H); ^{19}F NMR, δ, -225.4 (dt, $^3J_{HF}$ = 21.0, $^2J_{HF}$ = 54.0). *Anal. calcd.* for $C_{22}H_{21}FO_2$: C, 78.54; H, 6.30. *Found*: C, 78.27: H, 6.43.

1-Fluoro-3-(2-propenyloxy)propan-2-ol (5d). [(2-propenyloxy)methyl]oxirane (**3d**); 6h; Et_2O and PE (3 : 2). **5d**, 64%; n_D^{26} 1.4278; ^{1}H NMR, δ, 2.67 (bs, 1H), 3.52 (m, 2H), 4.00 (dm, 1H, $^3J_{HF}$ = 17.0), 4.02 (dm, 2H, J_{HH} = 6.9), 4.45 (dm, 2H, $^2J_{HF}$ = 47.4), 5.24 (m, 2H), 5.89 (m, 1H); ^{19}F NMR, δ, -225.5 (dt, $^3J_{HF}$ = 17.6, $^2J_{HF}$ = 47.4). *Anal. calcd.* for $C_6H_{11}FO_2$: C, 53.71; H, 8.28. *Found*: C, 53.77; H, 8.15.

1-Fluoro-3-(phenoxy)propan-2-ol (5e). [(Phenoxy)methyl]oxirane (**3e**); 8h; Et_2O and PE (1 : 1.5). **5e**, 90%; bp 112-114°C/5 Torr; n_D^{26} 1.5139; ^{1}H NMR, δ, 2.95 (bs, 1H), 4.05 (d, 2H, J_{HH} = 6.0), 4.24 (dm, 1H, $^3J_{HF}$ = 18.2), 4.58 (dm, 2H, $^2J_{HF}$ = 47.6), 6.96 (m, 3H), 7.29 (m, 2H); ^{19}F NMR, δ, -226.4 (dt, $^3J_{HF}$ = 19.2, $^2J_{HF}$ = 47.1). *Anal. calcd.* for $C_9H_{11}FO_2$: C, 63.51; H, 6.53. *Found*: C, 63.33; H, 6.60.

1-Fluoro-3-(4-chlorophenoxy)propan-2-ol (5f). [(4-Chlorophenoxy)methyl]oxirane (**3f**); 8h; Et_2O and PE (1 : 3). **5f**, 62%; mp 61-62°C; ^{1}H NMR, δ, 2.70 (d, 1H, J_{HH} = 5.3), 4.07 (d, 2H, J_{HH} = 5.2), 4.25 (dm, 1H, $^3J_{HF}$ = 18.7), 4.60 (dm, 2H, $^2J_{HF}$ = 47.1), 6.80 (m, 2H), 7.25 (m, 2H); ^{19}F NMR, δ, -226.4 (dm, $^2J_{HF}$ = 47.1). *Anal. calcd.* for $C_9H_{10}ClFO_2$: C, 52.82; H, 4.94. *Found*: C, 52.70; H, 5.00.

1-Fluoro-3-[[(methoxy)ethoxy]methoxy]propan-2-ol (5g). [[[(Methoxy)ethoxy]methoxy]methyl]oxirane (**3g**); 14h; Et_2O and PE (3 : 1). **5g**, 63%; n_D^{20} 1.4285; ^{1}H NMR, δ, 3.02 (d, 1H, J_{HH} = 5.0), 3.39 (s, 3H), 3.57 (m, 2H), 3.71 (m, 4H), 4.03 (dm, 1H, $^3J_{HF}$ = 17.8), 4.45 (ddd, 2H, J_{HH} = 1.5, 4.8, $^2J_{HF}$ = 47.1), 4.75 (s, 2H); ^{19}F NMR, δ, -225.1 (dm, $^2J_{HF}$ = 47.1). *Anal calcd.* for $C_7H_{15}FO_4$: C, 46.14; H, 8.31. *Found*: C, 46.28; H, 8.13.

1-Fluoro-3-[(phenylmethoxy)methoxy]propan-2-ol (5h) and 2-Fluoro-3-[(phenylmethoxy)methoxy]propan-1-ol (7h). [[(Phenylmethoxy)methoxy]methyl]oxirane (**3h**); 24h; Et_2O and PE (1 : 1). **5h**, 72%;

n_D^{20} 1.4983; ^{1}H NMR, δ, 2.80 (s, 1H), 3.71 (m, 2H), 4.12 (dm, 1H, $^3J_{HF}$ = 17.3), 4.45 (dd, 2H, J_{HH} = 4.9, $^2J_{HF}$ = 47.0), 4.62 (s, 2H), 4.80 (s, 2H), 7.35 (m, 5H); ^{19}F NMR, δ, -225.4 (dm, $^2J_{HF}$ = 47.0). *Anal. calcd.* for $C_{11}H_{15}FO_3$: C, 61.66; H, 7.07. *Found*: C, 61.74; H, 6.95. **7h**, 5%; ^{1}H NMR, δ, 2.78 (s, 1H), 3.82 (d, 2H, $^3J_{HF}$ = 20.8), 3.85 (d, 2H, $^3J_{HF}$ = 20.8), 4.61 (s, 2H), 4.72 (dm, 1H, $^2J_{HF}$ = 46.8), 4.79 (s, 2H), 7.35 (m, 5H); ^{19}F NMR, δ, -190.3 (dm, $^2J_{HF}$ = 48.8). *Anal. found*: C, 61.82; H, 6.83.

1-Fluoro-3-(2-tetrahydropyranyloxy)propan-2-ol (5i) and 2-fluoro-3-(2-tetrahydropyranyloxy)propan-1-ol (7i). [(2-Tetrahydropyranyloxy)methyl]oxirane (**3i**); 6h; Et_2O and PE (1 : 1.5). **5i**, 74%; n_D^{26} 1.4450; ^{1}H NMR (of the mixture of the two diasteroisomers), δ, 1.70 (m, 6H + 6H), 3.15 (d, 1H, J_{HH} = 4.1), 3.28 (d, 1H, J_{HH} = 2.9), 3.52 (m, 1H + 1H), 3.74 (m, 2H + 2H), 3.89 (m, 1H + 1H), 4.00 (m, 1H + 1H), 4.45 (dm, 2H + 2H, $^2J_{HF}$ = 47.1), 4.57 (m, 1H + 1H); ^{19}F NMR, δ, -224.2 (dt, 1F, $^3J_{HF}$ = 16.8, $^2J_{HF}$ = 49.2), -225.6 (dt, 1F, $^3J_{HF}$ = 19.5, $^2J_{HF}$ = 49.2). *Anal. calcd.* for $C_8H_{15}FO_3$: C, 53.92; H, 8.48. *Found*: C, 53.65; H, 8.37. **7i**, 2%; oil; ^{1}H NMR (of the mixture of the two diasteroisomers),δ, 1.70 (m, 6H + 6H), 2.10 (m, 1H), 2.20 (m, 1H), 3.53 (m, 1H + 1H), 3.70 (m, 2H + 2H), 3.90 (m, 3H + 3H), 4.63 (m, 1H + 1H), 4.72 (dm, 1H + 1H, $^2J_{HF}$ = 48.2); ^{19}F NMR, δ, -190.3 (m, 1F + 1F). *Anal. found*: C, 53.73; H, 8.38.

3-Fluoro-1,2-propanediol 1-O-benzoate (5j) and 3-Fluoro-1,2-propanediol 1,2-O-dibenzoate (8). Glycidyl benzoate (**3j**); 7.5h; Et_2O and PE (1 : 2). **5j**, 45%; n_D^{26} 1.5139; ^{1}H NMR, δ, 2.97 (bs, 1H), 4.21 (m, 1H), 4.41 (m, 2H), 4.53 (dm, 2H, $^2J_{HF}$ = 46.9), 7.44 (m, 3H), 7.98 (m, 2H); ^{19}F NMR, δ, -226.1 (dt, $^3J_{HF}$ = 17.5, $^2J_{HF}$ = 47.4). *Anal. calcd.* for $C_{10}H_{11}FO_3$: C, 60.59; H, 5.60. *Found*: C, 60.46; H, 5.43. **8**, 17%; mp 50°C (lit.[23] mp 50-51°C); ^{1}H NMR, δ, 4.65 (m, 2H), 4.76 (dd, 2H, J_{HH} = 4.6, $^2J_{HF}$ = 46.9), 5.61 (m, 1H), 7.47 (m, 6H), 8.03 (m, 4H); ^{19}F NMR, δ, -226.4 (dt, $^3J_{HF}$ = 17.5, $^2J_{HF}$ = 49.6).

3-Fluoro-1,2-propanediol 1-O-methanesulphonate (5k). Glycidyl methanesulphonate (**3k**); 8h; Et_2O and PE (2 : 1). **5k**, 5%; n_D^{26} 1.4402; ^{1}H NMR, δ, 3.04 (s, 3H), 3.15 (bs, 1H), 4.11 (dm, 1H, $^3J_{HF}$ = 21.0), 4.28 (m, 2H), 4.45 (dd, 2H, J_{HH} = 4.5, $^2J_{HF}$ = 48.0); ^{19}F NMR, δ, -225.6 (dt, $^3J_{HF}$ = 18.6, $^2J_{HF}$ = 50.5). *Anal. calcd.* for $C_4H_9FO_4S$: C, 27.83; H, 5.25. *Found*: C, 27.66; H, 5.15.

3-Fluoro-1,2-propanediol 1-O-(4-methylbenzene)sulphonate (5l). Glycidyl *p*-toluensulphonate (**3l**); 2.5h; AcOEt and CH_2Cl_2 (9 : 1). **5k**, 6%; oil; ^{1}H NMR, δ, 1.67 (bs, 1H), 2.45 (s, 3H), 3.77 (m, 1H), 4.10 (m, 2H), 4.43 (dd, 2H, J_{HH} = 4.2, $^2J_{HF}$ = 46.8), 7.37 (m, 2H), 7.55 (m, 2H); ^{19}F NMR, δ, -227.0 (dt, $^3J_{HF}$ = 18.9, $^2J_{HF}$ = 46.5). *Anal. calcd.* for $C_{10}H_{13}FO_4S$: C,48.37; H, 5.29. *Found*: C, 48.25; H, 5.10.

(+)-(2R)-1-Fluoro-3-(triphenylmethoxy)propan-2-ol (5m). (+)-(2*R*)-[(Triphenylmethoxy)methyl]oxirane (**3m**); the reaction conditions are the same as those reported for **3c**. **5m**, 83%; mp 70.6°C; $[\alpha]_D^{20}$ +2.47° (c = 0.03, CH_2Cl_2). The "Mosher's ester" of this compound (and of **5c**)was synthesized by treating 32 mg (0.095 mmol) of **5m** with 24 mg (0.097 mmol) of (+)-(*S*)-"Mosher's acid chloride", as reported in literature.[24]

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