was obtained in 42% yield (mp 94–98 °C; lit.[5] mp 96–98 °C). The chlorophosphine was reduced with $LiAlH_4$ in 64% yield to give bis(diisopropylamino)phosphine (bp 76–79 °C (0.4 mmHg); lit.[5] bp 83–85 °C (0.5 mmHg). The chlorophosphine also was converted to the analogous phosphenium tetrachloroaluminate according to the method of Cowley et al.[6]

Dimesitylhalophosphine. Magnesium (70 mmol) and 10 mL of tetrahydrofuran (THF) were placed into a 100-mL, three-necked flask equipped with a magnetic stirrer, a condenser, and an addition funnel. Mesityl bromide (58 mmol) in 50 mL of THF was added slowly, at such a rate as to keep the reaction mixture at a gentle reflux. The mixture was allowed to reflux for 1 h upon completion of the addition. In a separate 250-mL, three-necked flask was placed PCl_3 (29 mmol) in 65 mL of THF, and the Grignard reagent was added via an addition funnel, with the reaction mixture cooled to −78 °C under an N_2 atmosphere. The mixture was stirred at room temperature overnight. The solvent was removed by distillation, and the product was extracted with hexane. Distillation of the extracts gave a light yellow liquid that solidified overnight (87% based on a 1:1 mixture of chloride and bromide): bp 180–195 °C (0.3 mmHg).

Dimesitylphosphine. Dimesitylhalophosphine (25 mmol) and 50 mL of THF were placed in a 100-mL, three-necked flask. At −78 °C, 10 mmol of $LiAlH_4$ was added slowly with rapid stirring. The mixture was stirred overnight at room temperature, the solvent was removed by rotary evaporation, and the product was extracted with hexane. Evaporation of the hexane gave a light yellow solid, which was recrystallized from hexane to give a colorless solid (68%, mp 89–91 °C): ^1H NMR (CD_2Cl_2) δ 2.24 (s, 6 H), 2.25 (s, 12 H), 5.25 (d, 1 H, J_{P-H} = 232 Hz), 6.83 (s, 4 H); ^{31}P NMR (CD_2Cl_2) δ −91.3 (d, J_{P-H} = 232 Hz); IR (Nujol) 2492 (m) cm^{-1}; MS m/z 270 (M^+, 98.8).

Bis(2,4,6-triisopropylphenyl)phosphine was prepared in the same fashion as dimesitylphosphine but was not obtained in pure form.

Reaction of Phosphine with Trityl Perchlorate. The example of reaction with bis(diisopropylamino)phosphine is given. Stock solutions (0.1 M) were prepared from 0.1142 g (0.49 mmol) of bis(diisopropylamino)phosphine in 5 mL of CH_2Cl_2 and from 0.43 g (1.25 mmol) of trityl perchlorate in 12.5 mL of CH_2Cl_2. In an oven-dried, 10-mL NMR tube were placed 2 mL of CH_2Cl_2 and 1 mL of the 0.1 M stock solution of trityl perchlorate (0.1 mmol). At −78 °C, 1 mL of the phosphine solution (0.1 mmol) was added to the NMR tube under N_2. The resulting concentration was 0.025 M. The NMR tube was allowed to warm to room temperature with occasional shaking. The color of the mixture changed from yellowish brown to light yellow. After 3 h at room temperature, NMR spectra were recorded.

Reaction of Diphenylphosphine with Trityl Tetrafluoroborate. Trityl tetrafluoroborate (19.3 mmol) and CH_2Cl_2 (40 mL) were placed into a 100-mL, three-necked flask equipped with a magnetic stirrer and under N_2. Diphenylphosphine (19.3 mmol) in CH_2Cl_2 (20 mL) was added slowly to the flask at −78 °C with stirring. The mixture decolorized and a white solid appeared. The mixture was stirred overnight at room temperature. The solvent was evaporated, and the resulting white solid was washed five times with hexane and dried overnight under reduced pressure to give 8.92 g (90%) of diphenyl(triphenylmethyl)-phosphonium tetrafluoroborate: ^1H NMR (CD_2Cl_2) δ 7.19–7.28 (m, 10 H), 7.41–7.48 (m, 12 H), 7.68–7.73 (m, 3 H), 8.95 (d, 1 H, J_{P-H} = 508 Hz); ^{31}P NMR (CD_2Cl_2) δ 13.56 (d, J_{P-H} = 508 Hz); ^{11}B NMR (CD_2Cl_2) −1.21 (s) (trityl tetrafluoroborate was used as an external standard at δ −1.55); IR (Nujol) 2420 (w) cm^{-1}; MS m/z 429 ($Ph_2(Ph_3C)HP^+$, 75), 244 (Ph_3C^+, 100). Anal. Calcd for $C_{31}H_{28}BF_4P$: C, 72.11; H, 5.08; P, 6.00. Found: C, 71.95; H, 5.06; P, 5.70.

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Registry No. $(i\text{-Pr})_2NPN(Pr\text{-}i)_2^+ \cdot AlCl_4^-$, 68880-45-5; bis(diisopropylamino)chlorophosphine, 56183-63-2; bis(diisopropylamino)phosphine, 86660-77-7; mesityl bromide, 576-83-0; dimesitylbromophosphine, 86280-09-3; dimesitylchlorophosphine, 67950-05-4; dimesitylphosphine, 1732-66-7; bis(2,4,6-triisopropylphenyl)phosphine, 135665-28-0; diphenylphosphine, 829-85-6; diphenyl(triphenylmethyl)phosphonium tetrafluoroborate, 135665-30-4.

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N-Fluoropyridinium Pyridine Heptafluorodiborate: A Useful Fluorinating Agent

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The development of electrophilic fluorinating agents has been the subject of intense investigation by a number of research groups.[1] Most recently, *N*-fluoropyridinium triflates[2] and *N*-fluorosulfonamides[3] have been introduced as stable effective reagents with a wide range of strengths for the selective preparation of fluorine-containing molecules. These reagents, however, have the unfortunate disadvantages of tedious preparation and high cost in large-scale applications.

As part of our ongoing interest in fluorine chemistry, we report the ability of *N*-fluoropyridinium pyridine heptafluorodiborate (NFPy) (1) to react with enol compounds, under mild conditions, to selectively transfer fluorine to the reactive sites of organic compounds (Table I). NFPy is a cream colored crystalline solid, with an empirical formula of $C_5H_5NF(C_5H_5N)B_2F_7$, conveniently prepared by the reaction of fluorine with pyridine–boron trifluoride complex as described by Van Der Puy et al.[4] Treatment of enol acetates with 1 in refluxing acetonitrile gives α-fluoro ketones in good yields. Exposure of 1-acetoxy-4-*tert*-butylcyclohexene to NFPy affords a 62% yield of *cis*-/*trans*-2-fluoro-4-*tert*-butylcyclohexanone in a 3:1 ratio (entry 1). Similarly, the enol acetates derived from 17-keto steroids upon reaction with 1 yield high α/β ratios of the corresponding 16-fluoro steroids (entry 3).

Fluorine can be introduced selectively into the 6-position of steroids by employing their dienol acetates. For example, treatment of 3,5-androstadiene-3,17β-diol diacetate with 1 in acetontrile affords 6-fluorotestosterone acetate (entries 4 and 5). In all the reactions studied, fluorination occured at the terminus of the conjugated system and no complications were observed by the presence of carbonyl groups.

For steroids with two reactive sites of conjugated and nonconjugated enol acetates, NFPy showed a preference in fluorinating the terminus of the conjugated vinyl esters

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(2) Umemoto, T.; Tomita, K. *Tetrahedron Lett.* **1986**, *27*, 3271. Umemoto, T.; Kawada, K.; Tomita, K. *Tetrahedron Lett.* **1986**, *27*, 4465. Umemoto, T.; Tomizawa, G. *Bull. Chem. Soc. Jpn.* **1986**, *59*, 3625. Umemoto, T.; Tomita, K.; Kawada, K. *Org. Synth.* **1990**, *69*, 129. Umemoto, T.; Fukami, S.; Tomizawa, G.; Harasawa, K.; Kawada, K.; Tomita, K. *J. Am. Chem. Soc.* **1990**, *112*, 8563.
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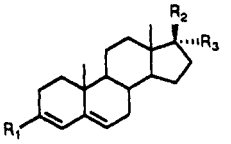
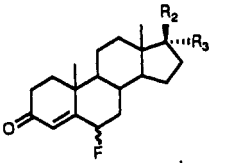
(4) Van Der Puy, M.; Nalewajek, D.; Shia, G. A.; Wagner, W. J. U.S. Patent 4,935,519, 1990. *N*-Fluoropyridinium pyridine heptafluorodiborate (NFPy) (1) is commercially available from Allied-Signal Inc., Buffalo Research Laboratory.

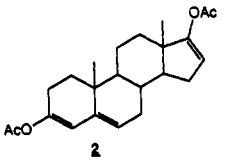
0022-3263/91/1956-5962$02.50/0

Table I. Fluorinations with NFPy

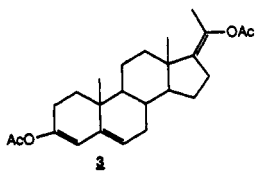
entry	substrate	product	cond (temp (°C), time)	yield (%)
1	1-acetoxy-4-*tert*-butylcyclohexene	2-fluoro-4-*tert*-butylcyclohexanone[a]	80, 18 h	62
2	3,4-dihydro-1-naphthyl acetate	2-fluoro-3,4-dihydronaphthalen-1(2*H*)-one[a]	80, 18 h	61
3	3-methoxy-17-acetoxy-1,3,5(10),16-estratetraene	16-fluoro-3-methoxy-1,3,5(10)-estratrien-17-one[b]	40, 3 d	68 (81:1, α:β)
				
4	$R_1 = R_2 = OAc$, $R_3 = H$	$R_2 = OAc$, $R_3 = H$[b]	80, 5 h	57 (4:1, α:β)
5	$R_1 = R_2 = OAc$, $R_3 = H$	$R_2 = OAc$, $R_3 = H$[b]	40, 2 d	96 (1:1; α:β)
6	$R_1 = OAc$, $R_2 = R_3 = O$	$R_2 = R_3 = O$[c]	80, 18 h	28 (6:1; α:β)
7	$R_1 = OAc$, $R_2 = R_3 = O$	$R_2 = R_3 = O$[c]	40, 4 d	60 (1:2; α:β)
8	$R_1 = OAc$, $R_2 = COCH_3$, $R_3 = H$	$R_2 = COCH_3$, $R_3 = H$[d]	80, 6 h	36 (only α)
9	$R_1 = OAc$, $R_2 = COCH_3$, $R_3 = H$	$R_2 = COCH_3$, $R_3 = H$[d]	40, 3 d	46 (1:2; α:β)
10	$R_1 = R_2 = OTMS$, $R_3 = H$	$R_2 = OH$, $R_3 = H$[e]	25, 18 h	88 (1:3; α:β)
11	(1-cyclohexenyloxy)trimethylsilane	2-fluorocyclohexanone[e]	0, 1 h	37
12	3β-acetoxy-17-acetamido-5,16-androstadiene	16-fluoro-3β-acetoxy-5-androsten-17-one[a]	25, 5 d	82 (15:1, α:β)
13	3β-acetoxy-17-acetamido-5,16-androstadiene	16-fluoro-3β-hydroxy-5-androsten-17-one[a]	25, 3 d	69 (6:1; α:β)
14	3β-acetoxy-17-acetamido-5,16-androstadiene	16-fluoro-3β-hydroxy-5-androsten-17-one[a]	80, 18 h	38 (1:1; α:β)
15	1-acetamido-4-*tert*-butylcyclohexene	2-fluoro-4-*tert*-butylcyclohexanone[a]	25, 2 d	93

[a] Rozen, S.; Menahem, Y. *J. Fluorine Chem.* **1980**, *16*, 19. [b] Chavis, C.; Mousseron-Canet, M. *Bull. Chem. Soc. Fr.* **1971**, 632. [c] Mann, J.; Pietrzak, B. *J. Chem. Soc., Perkin Trans. 1* **1983**, 2681. [d] Ephritikhine, M.; Levisalles, J. *Bull. Chem. Soc. Fr.* **1975**, 339. [e] Umemoto, T.; Kawasa, K.; Tomita, K. *Tetrahedron Lett.* **1986**, *27*, 4465.

over the enol acetates. Reaction of **2** with NFPy at 80 °C for 5 h gave a 39% yield of a 3:1 mixture of 6α- to 16α-fluorosteroid, whereas at 50 °C for 2 d a 72% yield of a 4:4:1 mixture of 6α-/6β-/16α-fluoro material was realized. Similarly, treatment of **3** with **1** in refluxing acetonitrile afforded the 6α-/17α-fluorosteroid in a 3:1 ratio in 87% yield, and reaction at 40 °C for 3.5 d gave a mixture of 6α-/6β-/17α- in a ratio of 2:2:1 in 99% yield.



2



3

Conjugated trimethylsilyl enol ethers react efficiently with NFPy in acetonitrile to afford C-6 fluoro steroids (entry 10). Fluorination of these systems favors the β isomer as contrasted to the high α ratios observed with dienol acetates. Unlike other N–F derived fluorinating agents, no competing C-4 fluorination was observed with **1**. Simple TMS enol ethers also reacted with NFPy to yield the expected α-fluoro ketones (entry 11).

Nakanishi and Jensen have demonstrated the reaction of perchloryl fluoride with enamides to furnish 16α-fluoro-17-keto steroids.[5] We have found that NFPy can be substituted for the explosive $FClO_3$ with excellent results. Treatment of the 3β-acetoxy-17-acetamino-5,16-androstadiene with **1** afforded, after mild acid hydrolysis, 16α-fluoro-3β-acetoxy-5-androsten-17-one in 82% yield (entry 12). If this reaction is worked up under basic conditions, 16-fluoro-3β-hydroxy-5-androsten-17-one is realized in 69% yield; however, considerable epimerization of the C-16 fluorine occurs (entries 13 and 14). Exposure of an acetonitrile solution of 1-acetamido-4-*tert*-butylcyclohexene to NFPy gives a 93% yield of *cis*-/*trans*-2-fluoro-4-*tert*-butylcyclohexanone in a 4:1 ratio (entry 15). This enamide derivative provided better selectivity in the fluorination reaction as compared to its corresponding enol acetate (entry 1). Unlike the reaction of vinyl esters with NFPy, which require refluxing acetonitrile, the electron-donating ability of the nitrogen in the enamide greatly enhances its reactivity, and thus the reactions proceed smoothly at room temperature.

As fluorinated organics gain increasing importance and utility in the medicinal, agricultural, and material sciences, *N*-fluoropyridinium pyridine heptafluorodiborate (NFPy) (**1**) ought to play an important role as an inexpensive, selective fluorinating agent. The utility of NFPy in other applications is currently under investigation.

(5) Nakanishi, S.; Jensen, E. V. *J. Org. Chem.* **1962**, *27*, 702. Nakanishi, S. *J. Med. Chem.* **1964**, *7*, 108.

Experimental Section

Typical Fluorination Procedure. 6-Fluorotestosterone Acetate. To a solution of 3,17β-diacetoxy-3,5-androstadiene (100 mg, 0.27 mmol) in acetonitrile (0.6 mL) was added *N*-fluoropyridinium pyridine heptafluorodiborate (**1**; 98 mg, 0.30 mmol) and the reaction stirred at 40 °C for 2 d. The mixture was poured into ether (10 mL), filtered through anhydrous $MgSO_4$, and evaporated to afford 90 mg (96% yield) of a 1:1.1 ratio of 6α-/6β-fluorotestosterone acetate. 6α-Fluorotestosterone acetate: ^{1}H NMR δ 0.85 (s, 3 H, Me-18), 1.2 (s, 3 H, Me-19), 5.01 (dqd, J = 47, 6, 2 Hz, 1 H, H-6); ^{19}F NMR δ −183 (dq, J = 47, 5 Hz). 6β-Fluorotestosterone acetate: ^{1}H NMR δ 0.87 (s, 3 H, Me-18), 1.15 (s, 3 H, Me-19), 5.00 (dt, J = 49, 3 Hz, 1 H, H-6); ^{19}F NMR δ −166 (tq, J = 49, 3.0 Hz).

Acknowledgment. The authors are grateful to Mr. R. J. Borowski for assistance in obtaining and interpreting nuclear magnetic resonance spectra.

Registry No. **1**, 131307-35-2; **2**, 102590-00-1; **3**, 135758-69-9; 1-acetoxy-4-*tert*-butylcyclohexene, 7360-39-6; 3,4-dihydro-1-naphthyl acetate, 19455-84-6; 3-methoxy-17-acetoxy-1,3,5(10),16-estratetraene, 6038-28-4; 3,5-androstadiene-3,17β-diol diacetate, 1778-93-4; 3-acetoxy-3,5-androstadien-17-one, 4968-05-2; 3-acetoxy-3,5-pregnadien-20-one, 4954-06-7; 3,17β-bis[(trimethylsilyl)oxy]-3,5-androstadiene, 25495-26-5; (1-cyclohexenyloxy)trimethylsilane, 6651-36-1; 3β-acetoxy-17-acetamido-5,16-androstadiene, 65732-71-0; 1-acetamido-4-*tert*-butylcyclohexene, 135663-21-7; *cis*-2-fluoro-4-*tert*-butylcyclohexanone, 7443-34-7; 2-fluoro-3,4-dihydronaphthalen-1(2*H*)-one, 71019-06-2; 16α-fluoro-3-methoxy-1,3,5(10)-estratrien-17-one, 2383-28-0; 6α-fluorotestosterone acetate, 855-55-0; 6α-fluoro-4-androstene-3,17-dione, 1093-88-5; 6α-fluoro-4-pregnene-3,20-dione, 2300-03-0; 6α-fluoro-17β-hydroxy-4-androsten-3-one, 1597-68-8;

16α-fluoro-3β-acetoxy-5-androsten-17-one, 2990-52-5; *trans*-2-fluoro-5-*tert*-butylcyclohexene, 23510-86-3; 17-acetoxy-6α-fluoro-4,16-androstadien-3-one, 108161-28-0; 3-acetoxy-16α-fluoro-3,5-androstadien-3-one, 108161-30-4; 17-acetoxy-6β-fluoro-4,16-androstadien-3-one, 108161-29-1; (*E*)-20-acetoxy-6α-fluoro-4,17(20)-pregnadien-3-one, 135663-22-8; 3-acetoxy-17α-fluoro-3,5-pregnadien-20-one, 135663-23-9; (*E*)-20-acetoxy-6β-fluoro-4,17(20)-pregnadien-3-one, 135663-24-0; 16α-fluoro-3β-hydroxy-5-androsten-17-one, 1649-27-0; 6β-fluorotestosterone acetate, 2627-94-3; 6β-fluoro-4-androstene-3,17-dione, 1650-83-5; 6β-fluoro-4-pregnene-3,20-dione, 2300-02-9; 6β-fluoro-17β-hydroxy-4-androsten-3-one, 1852-58-0; 16β-fluoro-3β-acetoxy-5-androsten-17-one, 82526-09-8; 16β-fluoro-3β-hydroxy-5-androsten-17-one, 135758-70-2; 2-fluorocyclohexanone, 694-82-6.

Selective Reduction of Carboxylic Acids into Alcohols Using $NaBH_4$ and I_2

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Reduction of carboxylic acids to alcohols is an important transformation in synthetic organic chemistry. Several methods are available for this purpose that utilize $LiAlH_4$ and boron reagents.[1-3] Unfortunately, $NaBH_4$ does not reduce carboxylic acids under ambient conditions.[3] We report here that carboxylic acids can be reduced directly to alcohols with some interesting selectivities by successive addition of $NaBH_4$ and I_2 to RCOOH and RCOOH to $NaBH_4$ in THF followed by I_2.

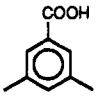
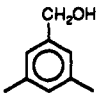
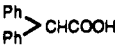
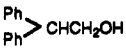
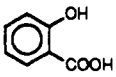
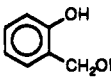
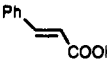
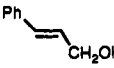
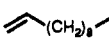
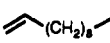
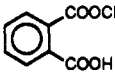
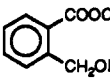
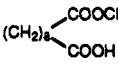
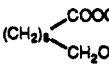
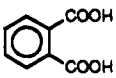
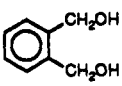
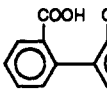
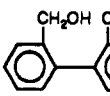
In a typical procedure, the carboxylic acid in THF is added slowly to the suspension of sodium borohydride in THF and the mixture stirred until gas evolution ceases. Iodine in THF is then added slowly at the temperature mentioned in Table I and the contents are stirred for 1 h at the same temperature. After the usual workup, the alcohol is obtained. The results are summarized in Table I.

Simple carboxylic acids (entries 1 to 7 in Table I) are reduced to the corresponding alcohols in very good yields. Reduction of cinnamic acid gives the corresponding α,β-unsaturated alcohol (entry 8). It is interesting to note that the reduction of this substrate with $LiAlH_4$ leads to 1-phenylpropanol.[4] Moreover, the olefinic group is not affected when it is away from the carboxylic group. For example, 10-undecenoic acid on reduction with $NaBH_4/I_2$ gives 10-undecenol (entry 9). However, the same substrate when treated with insufficient amount of BH_3–THF gives 1,11-undecanediol as the major product along with a minor amount of 11-hydroxyundecanoic acid.[5]

The present reagent system is also effective in reducing an acid group, leaving behind an ester group unaffected when both groups are present in the compound. This is so even when the ester group is near the acid group (entry 10).

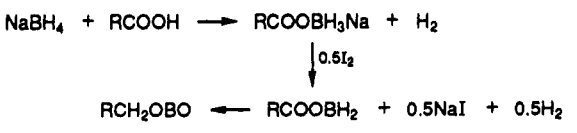
It has been reported that dicarboxylic acids react with borane reagents to give polymeric insoluble intermediates leading to incomplete reduction.[6] In some cases, however, the corresponding lactone is the major product.[7] On the other hand, the present reagent system completely reduces diacids such as phthalic acid and diphenic acid to the corresponding diols in very good yields (entries 12 and 13).

Table I. Selective Reductions of Carboxylic Acids to Alcohols[a]

no.	substrate	product	temp, °C	yield, %
1	C_6H_5COOH	$C_6H_5CH_2OH$	rt	93
2	$C_6H_5CH_2COOH$	$C_6H_5CH_2CH_2OH$	rt	98
3	*p*-ClC_6H_4COOH	*p*-$ClC_6H_4CH_2OH$	rt	98
4	$CH_3(CH_2)_8COOH$	$CH_3(CH_2)_8CH_2OH$	rt	95
5			rt	92
6			rt	96
7			rt	92
8			0	97
9			0	89
10			0	82
11			0	89
12			rt	86
13			rt	87

[a] All experiments were carried out by using $NaBH_4$ (12 mmol), carboxylic acid (10 mmol), and I_2 (5 mmol). Yields are of isolated and purified products.

The selectivities realized with the $NaBH_4/I_2$ system over the borane reagents such as BH_3–THF deserve an explanation. Hydroboration of olefins with the RCOOH/$NaBH_4$ system is relatively slow compared to hydroborations using BH_3–THF.[8,9] Also, the rates of reaction of cyclohexene and caproic acid with diborane are comparable.[10] Presumably, the present reagent system is more selective because the reactive $RCOOBH_2$ species is produced in the absence of more reactive borane species such as BH_3–THF.[11-15]



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