Table I. Conversion of RCO_2H to RBr (HgO-Br_2-light)[a]

RCO_2H	RBr: % yield (with light)[b]	RBr: % yield (without light)
p-$ClC_6H_4CO_2H$	80	~0[e]
p-$NO_2C_6H_4CO_2H$	95	42, 0,[e] 70[f]
3,5-$(NO_2)_2C_6H_3CO_2H$	97	0[e]
3-NO_2-4-Cl-5-$MeOC_6H_2CO_2H$	96[c]	~40
m-$BrC_6H_4CO_2H$	83	
p-$BrC_6H_4CO_2H$	86	
o-$ClC_6H_4CO_2H$	81	
m-$ClC_6H_4CO_2H$	75	
o-$MeOC_6H_4CO_2H$	[d]	
o-phthalic acid	42	85[f]
1-naphthoic acid	[d]	
n-$C_{13}H_{27}CO_2H$	99	91[f]
n-$C_{15}H_{31}CO_2H$	90	90[f]
n-$C_{17}H_{35}CO_2H$	85	95[f]

[a] Reactions run in carbon tetrachloride at reflux, molar ratios of RCO_2H-HgO-Br_2 are 1.00:1.50:1.50, respectively. [b] All yields are for pure (distilled or crystallized) products and were identified by comparison with published IR and NMR data (Aldrich or Sadtler). [c] J. M. Kane and A. I. Meyers, *Tetrahedron Lett.*, 771 (1977). [d] Gave only polybrominated products. [e] See ref 5. [f] Based on carbon dioxide produced. Actual isolated yields were much lower in those instances where isolation was attempted (see ref 4).

acids form a diacyloxy mercury salt **1**, which then leads to the acyl hypobromite **2** followed by thermal decomposition to the alkyl bromide. During the course of other

$$2RCO_2H \xrightarrow{HgO} \underset{\mathbf{1}}{(RCO_2)_2Hg} \xrightarrow{2Br_2} \underset{\mathbf{2}}{2RCO_2Br} \rightarrow 2RBr$$

studies, we had occasion to investigate the CFH reaction with aromatic acids. The observed trend[4-6] is that aromatic acids form rather insoluble mercuric salts and give lower yields than aliphatic acids. We now describe a further modification of the CFH reaction which simply involves irradiating the reaction mixture with a 100-W bulb. In this fashion excellent and reproducible yields of aryl and alkyl bromides were obtained (Table I). For those aromatic acids containing electron-rich substituents (e.g., anisic acid) the reaction gave only polybrominated products.

Experimental Section

General Procedure for Light-Assisted Bromodecarboxylation. Into a dry, nitrogen-purged, three-neck, 100-mL flask containing a spin bar and fitted with a reflex condenser were placed the carboxylic acid (10.0 mmol), carbon tetrachloride (reagent grade, 50 mL), and red mercuric oxide (B & A, 15.0 mmol). The flask and an adjacent 100-W bulb were enclosed in aluminum foil. The mixture was heated to reflux in an oil bath while being irradiated, and bromine (Baker, "purified", 15.0 mmol) was cautiously added via syringe. The mixture was heated at reflux and irradiated for 3 h, and cooled to room temperature. Saturated aqueous $NaHCO_3$ (30 mL) was added and the mixture was vigorously stirred for 15 min. The two-phase mixture was vacuum filtered through a Celite pad and the pad was washed several times with chloroform. The organic phase of the filtrate was washed with saturated $NaHCO_3$ (30 mL) and brine (30 mL) and dried ($MgSO_4$). Removal of solvent and distillation gave the bromide.

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Registry No. p-$ClC_6H_4CO_2H$, 74-11-3; p-$NO_2C_6H_4CO_2H$, 62-23-7; 3,5-$(NO_2)_2C_6H_3CO_2H$, 99-34-3; 3-NO_2-4-Cl-5-$MeOC_6H_2CO_2H$, 71001-78-0; m-$BrC_6H_4CO_2H$, 585-76-2; p-$BrC_6H_4CO_2H$, 586-76-5; o-$ClC_6H_4CO_2H$, 118-91-2; m-$ClC_6H_4CO_2H$, 535-80-8; o-$MeOC_6H_4CO_2H$, 579-75-9; o-phthalic acid, 88-99-3; 1-naphthoic acid, 86-55-5; n-$C_{13}H_{27}CO_2H$, 544-63-8; n-$C_{15}H_{31}CO_2H$, 57-10-3; n-$C_{17}H_{35}CO_2H$, 57-11-4; p-ClC_6H_4Br, 106-39-8; p-$NO_2C_6H_4Br$, 586-78-7; 3,5-$(NO_2)_2C_6H_3Br$, 18242-39-2; 3-NO_2-4-Cl-5-$MeOC_6H_2Br$, 63603-16-7; m-BrC_6H_4Br, 108-36-1; p-BrC_6H_4Br, 106-37-6; o-ClC_6H_4Br, 694-80-4; m-ClC_6H_4Br, 108-37-2; o-BrC_6H_4Br, 583-53-9; n-$C_{13}H_{27}Br$, 765-09-3; n-$C_{15}H_{31}Br$, 629-72-1; n-$C_{17}H_{35}Br$, 3508-00-7.

Maximum Optical Rotation of 2-Fluorooctane? Survey of Fluorinating Reagents

Jacques Leroy,[1a] Eric Hebert,[1b] and Claude Wakselman[1a]*

C.N.R.S., 94320 Thiais, France

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Optically active 2-alkyl halides are of particular interest as they can serve as models in the study of numerous reactions of organic chemistry. 2-Chloro-, 2-bromo-, or 2-iodooctane is commonly used[2] and their optical purities are determined without ambiguity.[3] By contrast, however, the maximum optical rotation of 2-fluorooctane remains unknown, as no chemical correlation has been established, owing to the poor leaving-group character of fluorine. Thus, only an estimate of optical purity has been made for 2-fluorooctane (**1**) based on a correlation between molecular rotation and bond refraction.[4]

Recent results obtained with methyltri-n-butylfluorophosphorane[5] (**2**) leading to 2-fluorooctane of higher optical rotation than that of San Filippo and Romano[4] (stated as about 100% optical purity) have prompted us to check other fluorinating reagents.[6] Phenyltetrafluorophosphorane[7] (**3**), N-(2-chloro-1,1,2-trifluoroethyl)diethylamine[8] (**4**) (FAR), and (diethylamino)sulfur trifluoride[9] (**5**) (DAST) were used according to slightly modified procedures.

$$C_6H_{13}CH(OTs)CH_3 + \underset{\mathbf{2}}{(n\text{-}C_4H_9)_3PFCH_3} \rightarrow \underset{\mathbf{1}}{C_6H_{13}CHFCH_3} + \text{octenes} + (n\text{-}C_4H_9)_3P(OTs)CH_3 \quad (1)$$

$$C_6H_{13}CH[OSi(CH_3)_3]CH_3 + \underset{\mathbf{3}}{C_6H_5PF_4} \rightarrow 1 + C_5H_{11}CHFCH_2CH_3 + \text{octenes} + FSi(CH_3)_3 + C_6H_5POF_2 \quad (2)$$

$$C_6H_{13}CHOHCH_3 + \underset{\mathbf{4}}{(C_2H_5)_2NCF_2CHFCl} \rightarrow 1 + \text{octenes} + (C_2H_5)_2NCOCHFCl + HF \quad (3)$$

$$C_6H_{13}CHOHCH_3 + \underset{\mathbf{5}}{(C_2H_5)_2NSF_3} \rightarrow 1 + \text{octenes} + (C_2H_5)_2NSOF + HF \quad (4)$$

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Table I

substrate	reagent	solvent	% fluoro-octane-(s)[a]	% oc-tenes[a]	% yield[b] 2-fluoro-octane	$[\alpha]^{20}_{589}$,[c] deg ($CHCl_3$)	% optical purity
(−)-(*R*)-$C_6H_{13}CH(OTs)CH_3$	$(n\text{-}C_4H_9)_3PFCH_3$	THF	40	60	15	14.8[d]	100
(+)-(*S*)-$C_6H_{13}CH[OSi(CH_3)_3]CH_3$	$C_6H_5PF_4$	none	58[e]	42	(11)	−10	67.6
(+)-(*S*)-$C_6H_{13}CHOHCH_3$	FAR	diethyl ether	78	22	40	−13	88
(+)-(*S*)-$C_6H_{13}CHOHCH_3$	DAST	CH_2Cl_2	48	52	23	−14.5	97.6
(−)-(*R*)-$C_6H_{13}CH(OTs)CH_3$	KF[f]	triethylene glycol	50	50	31	13.5	90.7

[a] These mean values were obtained in two ways: GLC and (or) from bromination of octenes. [b] Isolated yield. In small-scale preparation, isolated yields are underestimated. [c] Best value of several assays, corrected for optically pure starting 2-octanol. [d] $[\alpha]^{20}_{589}$ (neat) = 13.6°. Optical purity of San Filippo's sample: 90.7 %. [e] 78% 2-fluorooctane, 22% 3-fluorooctane (by ^{19}F NMR). [f] Method developed by J. San Filippo and L. J. Romano.[4]

A general feature is the formation of octene isomers in variable amounts according to the reagent. This predominates when **2** is used but is minor with FAR (Table I). With reagent **2**, as with FAR and DAST, rearranged halocarbons cannot be detected by ^{19}F NMR. In contrast, with reagent **3**, optically inactive 3-fluorooctane is produced along with 2-fluorooctane. In this case, isomeric fluorocarbons could not be separated and the optical purity of **1** was corrected to 100% isomeric purity. In each case, the optical purity of **1** was corrected to 100% enantiomeric purity for the starting 2-octanol. The mechanism of these reactions is not known. Nevertheless, the enantioselectivity of each of them can be obtained directly, assuming that 2-fluorooctane prepared with methyltri-*n*-butylfluorophosphorane is optically pure.

For all the fluorinating reagents, except phenyltetrafluorophosphorane, enantioselectivity, i.e. *inversion* of configuration, appears to be good (FAR and KF) or even excellent (DAST and methyltri-*n*-butylfluorophosphorane). The results obtained with these reagents are consistent with a clean S_N2 step for carbon–fluoride bond formation. For phenyltetrafluorophosphorane, as already suggested,[7] fluorination proceeds, at least in part, via a carbocation, owing to the relatively low enantioselectivity.

Hence, in order to obtain 2-fluorooctane with acceptable yield and good optical purity, it is recommended to use the FAR reagent or potassium fluoride. The DAST reagent and methyltri-*n*-butylfluorophosphorane give the highest optical purity but yields are somewhat lower.

Experimental Section

Caution! It has been reported recently[10] that DAST can decompose violently upon contact with water and by heating at temperatures higher than about 50 °C. Suitable safety precautions must be observed in working with that reagent.

^1H NMR spectra were recorded at 60 MHz on a Perkin-Elmer R24 spectrometer with Me_4Si as internal standard. ^{19}F NMR spectra were recorded at 56.4 MHz on a JEOL C-60 HL spectrometer with $CFCl_3$ as internal standard. ^{13}C NMR spectra were run at 20 MHz with a Varian CFT-20 spectrometer. The optical rotations were determined at 20 °C, in a thermostated 1-dm cell using a Perkin-Elmer Model 141 M spectropolarimeter. The concentrations of 2-fluorooctane in chloroform varied from 3 to 20 g/100 mL.

We have checked the absence of a special concentration effect on the $[\alpha]^{20}_{589}$ values. Analytical and preparative GLC were performed on a Varian Aerograph Model 920 chromatograph (SE 30 columns). The purity of 2-fluorooctane samples was checked by analytical GLC (sample purity >99%) and NMR.

Optically active 2-octyl tosylate was prepared from optically active 2-octanol (Aldrich) by Streitwieser's procedure.[11] Phenyltetrafluorophosphorane was prepared by a described procedure.[12] (Diethylamino)sulfur trifluoride was a commercial product.[13]

Table II

	δC^a (*J*, Hz)	δC^b (*J*, Hz)	δC^c (*J*, Hz)	δC^d (*J*, Hz)
X = F, Y = H	21.1 (23)	90.8 (165)	37.2 (21)	25.2 (5)
X = H, Y = F	9.4 (5)	28.3 (22)	95.5 (167)	34.9 (21)

Fluorination of (−)-(*R*)-2-Octyl Tosylate with Methyltri-*n*-butylfluorophosphorane. To a stirred solution of 5 g (0.018 mol) of (−)-(*R*)-2-octyl tosylate in 25 mL of anhydrous tetrahydrofuran at −70 °C was added dropwise 6 g (0.027 mol) of methyltri-*n*-butylfluorophosphorane.[14] The reaction mixture was slowly warmed to room temperature and stirred for 1 h. The mixture was poured into water (100 mL) and extracted with diethyl ether. The organic layer was dried ($MgSO_4$) and concentrated in vacuo by rotary evaporation. The volatile products of the residue were distilled bulb-to-bulb at 0.05 Torr (room temperature) into a receiver cooled by liquid nitrogen to give 1.1 g of a mixture of (+)-(*S*)-2-fluorooctane and isomeric octenes.

Preparative GLC of the mixture afforded 0.36 g (0.0027 mol) of (+)-(*S*)-2-fluorooctane. Comparable yields were obtained if the volatile part of the crude was treated by a slight excess of bromine in carbon disulfide (3 mL) at 0 °C to separate octenes.

After removing carbon disulfide by rotary evaporation, the mixture was diluted in diethyl ether, washed with aqueous sodium bicarbonate and thiosulfate and water, and dried over $MgSO_4$. Careful distillation on a small Vigreux column afforded pure (+)-(*S*)-2-fluorooctane: bp 54 °C (38 Torr); ^1H NMR ($CDCl_3$) δ 1.26 (dd, 3 H, J = 23, 6 Hz), 4.50 (dm, 1 H, J = 48 Hz); ^{19}F NMR ($CDCl_3$) 165 (m).

(+)-(*S*)-2-(Trimethylsilyloxy)octane. To a solution of 4.5 g (0.035 mol) of (+)-(*S*)-2-octanol in 10 mL of anhydrous diethyl ether was added dropwise 7.2 g (0.051 mol) of *N*-(trimethylsilyl)imidazole. The reaction mixture was stirred for 1 h at room temperature, then an additional hour at 60 °C. After cooling, imidazole was filtered off and the solution was poured into water (100 mL) and extracted with diethyl ether. Distillation afforded 6.05 g (0.03 mol) of (+)-(*S*)-2-(trimethylsilyloxy)octane: bp 66–67 °C (10 Torr); ^1H NMR ($CDCl_3$) δ 0.1 (s, 9 H), 1.08 (d, 3 H, J = 6 Hz), 3.7 (m, 1 H). Anal. Calcd for $C_{11}H_{26}OSi$: C, 65.27; H, 12.95. Found: C, 65.10; H, 12.83.

Fluorination of (+)-(*S*)-2-(Trimethylsilyloxy)octane with Phenyltetrafluorophosphorane. To 4.13 g (0.022 mol) of phenyltetrafluorophosphorane cooled to −40 °C was added dropwise 4.53 g (0.022 mol) of (+)-(*S*)-2-(trimethylsilyloxy)octane.

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After adding, the reaction mixture was warmed to room temperature and then heated to 80 °C for 20 min. After cooling, the crude reaction mixture was neutralized with an aqueous saturated sodium bicarbonate solution and extracted with diethyl ether. The organic layer was dried ($MgSO_4$) and filtered. The solvent was evaporated under reduced pressure and the residue was distilled bulb-to-bulb at 0.05 torr (room temperature) into a receiver cooled by liquid nitrogen. Octenes were converted into the corresponding dibromoalkanes as described above. (−)-(*R*)-2-Fluorooctane was obtained by bulb-to-bulb distillation (0.05 torr, room temperature) as an inseparable mixture (0.4 g; 0.003 mol) with isomeric 3-fluorooctane: ^{19}F NMR ($CDCl_3$) 174 (m); ^{13}C NMR ($CDCl_3$, Me_4Si) {^1H}, general formula $C_4H_9C^dH_2C^cHYC^bHXC^aH_3$ (see Table II).

Fluorination of (+)-(*S*)-2-Octanol with FAR. A solution of 16.2 g (0.085 mol) of *N*-(2-chloro-1,1,2-trifluoroethyl)diethylamine[8] in 10 mL of anhydrous diethyl ether was added to a solution of 10 g (0.077 mol) of (+)-(*S*)-2-octanol in 30 mL of ether cooled to 0 °C. The reaction mixture was allowed to stand at 0 °C for 24 h, and then washed with an aqueous saturated sodium bicarbonate solution and water until neutral. The organic layer was dried ($MgSO_4$), concentrated, and distilled bulb-to-bulb at 15 torr (bath temperature 50 °C) into a receiver cooled to −75 °C to separate the diethylamide of chlorofluoroacetic acid. The volatile products were treated with bromine in the usual way. Distillation bulb-to-bulb at 0.01 torr (room temperature) afforded 4.5 g (0.034 mol) of (−)-(*R*)-2-fluorooctane.

In another experiment, the crude reaction mixture, after hydrolysis, was distilled on a spinning-band column (40 torr) to give (−)-(*R*)-2-fluorooctane free of octenes, in comparable yield and optical purity.

Fluorination of (+)-(*S*)-2-Octanol with DAST. A solution of 4.25 g (0.033 mol) of (+)-(*S*)-2-octanol in 6 mL of dichloromethane was added dropwise to a stirred solution of 5.18 g of DAST in 15 mL of methylene chloride cooled to −60 °C. The reaction mixture was slowly warmed to room temperature and stirred overnight. After washing with aqueous sodium bicarbonate and water until neutral, the organic layer was dried, concentrated, and distilled bulb-to-bulb at 0.01 torr (room temperature) to give a mixture of 2-fluorooctane and octenes. This mixture was treated by bromine in the usual way. Distillation on a small Vigreux column afforded 1.0 g (0.0076 mol) of (−)-(*R*)-2-fluorooctane.

Registry No. (−)-(*R*)-2-Octyl tosylate, 27770-99-6; (+)-(*S*)-2-fluorooctane, 56772-74-8; (+)-(*S*)-2-(trimethylsilyloxy)octane, 65500-76-7; (+)-(*S*)-2-octanol, 6169-06-8; (−)-(*R*)-2-fluorooctane, 54632-06-3; 3-fluorooctane, 20469-83-4.

Bromomaltol: Structure and Conversion to Novel Pyridone and Pyridine Derivatives

James H. Looker,* Robert J. Prokop, William E. Serbousek, and Michael D. Cliffton

Department of Chemistry, The University of Nebraska, Lincoln, Nebraska 68588

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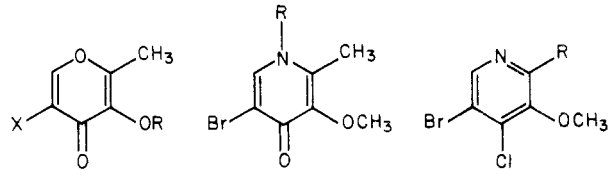
Maltol (**1**), known since 1862,[1] has been obtained from several plant, food, and beverage sources,[2] and by synthesis from methyl-α-furylcarbinol.[3] The mesylate of **1** was prepared several years ago.[4] In the present paper, we report bromination of maltol by *N*-bromosuccinimide (NBS) and by bromine, chemical and spectral data which establish structure of the bromination product, and conversion of the latter to new pyridone and pyridine derivatives.

Bromination of the methyl group of **1** with NBS is an obvious first step in functionalization. However, when **1** reacted with NBS in carbon tetrachloride containing benzoyl peroxide, there resulted a bromomaltol (**2**) which reacted with silver acetate–acetic anhydride to give an ester (**3**), which retained bromine. ^{13}C NMR data confirmed



1, X = R = H
2, X = Br; R = H
3, X = Br; R = Ac
4, X = Br; R = CH_3

5, R = H
9, R = CH_3

6, R = CH_3
7, R = CO_2H
8, R = H

a nuclear bromine; hence the acetoxyl group resulted from enolic hydroxyl acetylation, not from bromine displacement.

Direct bromination in three haloalkane solvents also was studied. Yields of **2** in both carbon tetrachloride and methylene chloride were approximately 30%. When **1** was reacted with excess bromine in 1,1,2,2-tetrachloroethane and the reaction mixture was swept continually with nitrogen, the yield of **2** was 58%. Sweeping with nitrogen removed not only oxygen, but also hydrogen bromide, which conceivably could interact with **1** and **2** to form insoluble oxonium salts.

The position of the chemically inert bromine was not apparent. Conversion of kojic acid to pyridine derivatives was structurally useful;[5] hence a multistep sequence leading to a known bromopyridine[6] was attempted. Bromomaltol was methylated to give the ether **4**. Reaction of **4** to give the pyridone (**5**) required only concentrated aqueous ammonia at room temperature, in contrast to procedures[7] using elevated temperature and pressure. Assignment of a pyridone structure to **5** is not unequivocally certain, for tautomerization to a pyridin-4-ol derivative is possible.[8] Reaction of phosphoryl chloride gave 2-methyl-3-methoxy-4-chloro-5-bromopyridine (**6**). The difference in melting points of **5** and **6** is striking: mp of **5**, 233 °C; mp of **6**, 35 °C. Oxidation of **6** with neutral permanganate gave the picolinic acid derivative (**7**), which was decarboxylated at 120 °C to yield the trisubstituted pyridine (**8**). ^1H NMR spectra of **6** and **8** contained peaks near δ 8.3, in satisfactory agreement with a value of δ 8.5 reported[9] for α-protons in pyridine derivatives. These data indicate tentatively that the bromine atom in **6** is bonded not to C(6) but to C(5). Hence the bromine atoms in **2** and **4** also must be bonded to C(5). A preliminary study of selective dehalogenation of the 4-chloro atom of **8** was attempted,[10] but insufficient **8** was available for thorough study.

In Table I are presented ^{13}C NMR data for bromomaltol (**2**) and 2-bromopyromeconic acid (2-bromo-3-hydroxy-4*H*-pyran-4-one).[11] Chemical shifts for these two bro-

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