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Facile oxidative conversion of alcohols to esters using molecular iodine

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Abstract—A simple, efficient, and high-yield procedure for the oxidative conversion of alcohols to various types of esters and ketones, with molecular iodine and potassium carbonate was successfully carried out.

1. Introduction

1.1. Oxidation of alcohols

Oxidation of alcohols with less toxic reagents is very important in organic synthesis. Therefore, it has been intensively studied and numerous reagents and methods have been developed.[1]

Oxidation with molecular halogens and related reagents is attractive due to the simple operation and low cost. To date, halogen-mediated oxidation of alcohols to aldehydes, esters, and ketones with aqueous NaOCl,[2] $Ca(OCl)_2$/AcOH,[3a,b] *t*-BuOCl/Py,[3c] Cl_2-dimethyl sulfide,[4a,b,e] Cl_2-dimethyl sulfoxide,[4c] Cl_2-pyridine,[4d] Cl_2-HMPT or Br_2-HMPT,[4f,g] Br_2/KBr,[5a] Br_2 and HOBr,[5b] $NaBrO_2$,[5c] $NaBrO_3$,[5d] $PhCH_2N^+Me_3Br_3^-$,[5e] NaBr/CH_3CO_3H,[5f] PhIO/KBr,[5g] and $PyH^+Br_3^-$[5h] has been well studied. However, these methods still have several drawbacks such as strong oxidative conditions, or acidic or basic conditions.

Direct oxidative condensation of primary alcohols to the corresponding esters, where both the acid and the alcohol portion of the esters are derived from the alcohols, is interesting and useful.[6] Therefore, extensive study has been carried out as follows: oxidative condensation with metal catalysts such as ruthenium or palladium; $Ru_3(CO)_{12}$,[7a] $RuH_2(Ph_3P)_4$,[7b,c] $PdCl_2$,[7d,e] $Pd(OAc)_2$,[7f] $PhCH_2N^+Me_3Br_4MoO^-$,[7g] under heating conditions, with chromic acids such as $Na_2Cr_2O_7/H_2SO_4$,[8a] PCC/Al_2O_3,[8b] and with oxoammonium salt.[9] However, these reactions have drawbacks from a practical point of view, that is, the reaction requires high temperature, yield of the esters is not high, or the alcohols used are limited.

1.2. Oxidation of aldehydes

It is often required to transform aldehydes directly into esters during various stages in organic synthesis and natural product synthesis.[10] The oxidation of aldehydes to carboxylic acids or esters is one of the most frequently encountered reactions in organic chemistry. Such a process has been accomplished in a variety of ways. Two-step methods include the oxidation of hemiacetals,[11] acetals,[12] cyanohydrins,[13] etc. Conversion of aldehydes to the methyl esters or acids by NaOCl,[14a] *t*-BuOCl,[14b] and $Ca(OCl)_2$[14c] is effective; however, activated aromatic aldehydes gave lower yield of the products, because of the ring chlorination. Generally, one-pot or one-step methods reported require the use of heavy-metal oxidants such as $KMnO_4$,[15] PDC,[16] or the very expensive silver,[17] rhodium,[18] ruthenium,[19] or vanadium[20] catalysts. Oxidation using $NaClO_2/H_2O_2$[21] is the most common method. NIS-mediated oxidation,[22] electrochemical oxidation,[23] as well as very recently using cat. V_2O_5/H_2O_2,[24a] cat. $QHSO_4/H_2O_2$,[24b] cat. $S{\cdot}SnO_2/H_2O_2$,[24c] and the conversion of aldehydes into glycol monoesters with $Al_2O_3/MeSO_3H$,[25] also have been reported.

Most of the reported methods are useful for oxidation of aldehydes into the corresponding esters. However, conversion of aldehydes with a stoichiometrical amount of

Keywords: Molecular iodine; Oxidation; Alcohol; Aldehyde; 2,2,2-Trifluoroethyl ester; Methyl ester; Condensed ester; Ketone.
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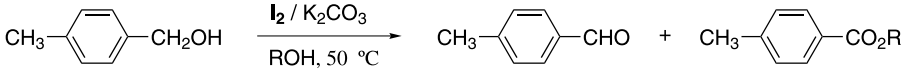
Table 1. Oxidation of *p*-methylbenzyl alcohol with I_2/K_2CO_3 in protic polar solvents



1f **2f** **3f (R = CH_2CF_3)** **4f (R = CH_3)**

Entry	I_2/K_2CO_3 (equiv)	ROH (pK_a)	Time (h)	Yields (%)		
				If	**2f**	**Xf**
1	5/5	$(CH_3)_3COH$ (18.0)	19	0	87	7 (R=H)
2	5/5	CH_3CH_2OH (15.9)	14	90	10	0
3	5/5	CH_3OH (16.0)	24	30	4	66 (**4f**)
4	5/5	H_2O (15.7)	21	0	47	53 (R=H)
5	3/3	CF_3CH_2OH (12.4)	18	0	16	83 (**3f**)
6	5/0	CF_3CH_2OH	18	100	0	0
7	5/5	CF_3CH_2OH	14	0	6	85 (**3f**)
8	5/5	CF_3CH_2OH	18[a]	0	5	91 (**3f**)
9	5/5	$(CF_3)_2CHOH$ (9.2)	14	76	24	0

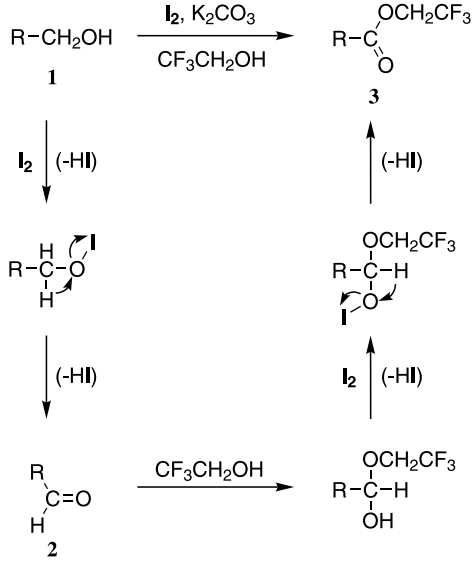
[a] Half amount each of I_2 and K_2CO_3 was added at first, and the rest was added later.

alcohols into esters with molecular iodine has been never studied.

1.3. Oxidation with molecular iodine

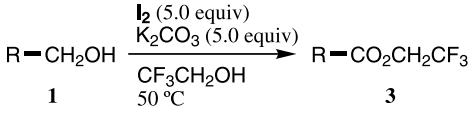
Molecular iodine is a mild, cheap and easily available oxidizing reagent, and, moreover, it is useful because of its solid form and it is less toxic than molecular bromine or chlorine. Previously, simple and effective conversion of aldehydes to the corresponding methyl esters,[26a,b] carboxylic acids,[26c] and nitriles[26d] in the presence of molecular iodine in methanol, in water/acetonitrile, and in ammonia water, respectively, was reported. The Lieben iodoform reaction, which is the reaction of methyl ketones with molecular iodine in aqueous basic solution, is known for the detection of the methyl ketone group.[27]

Here, as a part of our basic study of molecular iodine for organic synthesis,[28] we would like to report a useful oxidation of alcohols to the corresponding esters and ketones by a simple procedure using molecular iodine and potassium carbonate in protic polar solvents (i.e., 2,2,2-trifluoroethanol, methanol, and *t*-butyl alcohol). To the best of our knowledge, reports on the direct oxidative esterification of primary alcohols with molecular iodine in such alcoholic solvents have not yet appeared,[29] while the

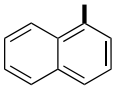


Scheme 1. Plausible reaction pathway for 2,2,2-trifluoroethyl ester.

Table 2. Oxidation of primary alcohols to 2,2,2-trifluoroethyl esters with I_2/K_2CO_3 in CF_3CH_2OH

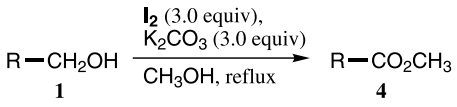


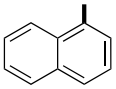
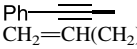
1 **3**

Entry	R–	Time (h)	Yield (%)
1	3-$O_2NC_6H_4$–	1	80 (**3a**)
2	3,4-$Cl_2C_6H_3$–	2[a]	91 (**3b**)
3	4-$CF_3C_6H_4$–	4	87 (**3c**)
4	4-ClC_6H_4–	5[a]	93 (**3d**)
5		5[b]	78
6	Ph–	5[a]	91 (**3e**)
7	4-$CH_3C_6H_4$–	18[a]	91 (**3f**)
8		18[b]	83
9	4-$CH_3OC_6H_4$–	24[a]	85 (**3g**)
10		24[b]	63
11	2,5-$(CH_3)_2C_6H_3$–	24[a]	85 (**3h**)
12	3,4,5-$(CH_3O)_3C_6H_2$–	16	93 (**3i**)
13		14	95 (**3j**)
14		1	78 (**3k**)
15		24[a]	67 (**3l**)
16	$CH_3(CH_2)_{11}$–	84	60 (**3III**)
17		15[b]	91 (**3VI**)
18		5[b]	100 (**3X**)

[a] Half amount each of I_2 and K_2CO_3 was added at first, and the rest was added later.
[b] Molar ratio of I_2/K_2CO_3 is 3.0/3.0.

Table 3. Oxidation of primary alcohols to methyl esters with I_2/K_2CO_3 in CH_3OH

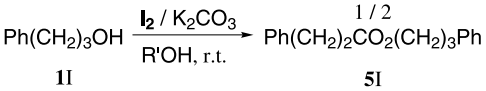


Entry	R–	Time (h)	Yield[a] (%)
1	3-$O_2NC_6H_4$–	20	86
2	4-ClC_6H_4–	21	93
3	Ph–	14	86
4	4-$CH_3C_6H_4$–	15	87
5	4-$CH_3OC_6H_4$–	28	74
6	3,4,5-$(CH_3O)_3C_6H_2$–	19	77
7		30	82
8		24	82
9		39	91
10	$PhCH_2CH_2$–	40[b]	76
11	$CH_3(CH_2)_6$–	5	73
12	$CH_3(CH_2)_{11}$–	7	70
13	4-$CH_3OC_6H_4(CH_2)_2$–	80[b]	82
14		23	70
15		8	90
16		27	76
17	CH_2=$CH(CH_2)_8$–	20[c]	75

[a] Method A. Alcohol (1 mmol) in MeOH (0.5 ml).
[b] Method B. Alcohol (2 mmol) in MeOH (0.6 ml).
[c] After the reaction, zinc powder was added to regenerate the olefinic group.

electrochemical conversion of alcohols to the corresponding ketones or esters using potassium iodide via the formation of iodonium ion species generated by electroxidation,[30] and oxidative conversion of secondary alcohols into α-iodoketones with NaI, H_2O_2, and acid,[31a] and oxidation of alcohols using IPy_2BF_4/I_2[31b] were reported.

Table 4. Oxidative condensation of 3-phenylpropanol with I_2/K_2CO_3 in protic polar solvents



Entry	I_2/K_2CO_3 (equiv)	R′OH	Time (h)	Yields (%)
1	1.2/5	CH_3OH	21	0 (26,[a] 70[b])
2	1.2/5	CH_3CH_2OH	21	0 (8,[c] 89[b])
3	1.2/5	$(CH_3)_2CHOH$	21	8 (92[b])
4	1.2/5	CF_3CH_2OH	21	34 (50[b])
5	1.2/5	$(CH_3)_3COH$	21	96
6	1.2/1.2	$(CH_3)_3COH$	66	42 (58[b])
7	1.2/2.4	$(CH_3)_3COH$	66	84 (16[b])
8	1.2/3.6	$(CH_3)_3COH$	66	93 (7[b])
9	3.0/3.0	$(CH_3)_3COH$	45	92 (7[b])

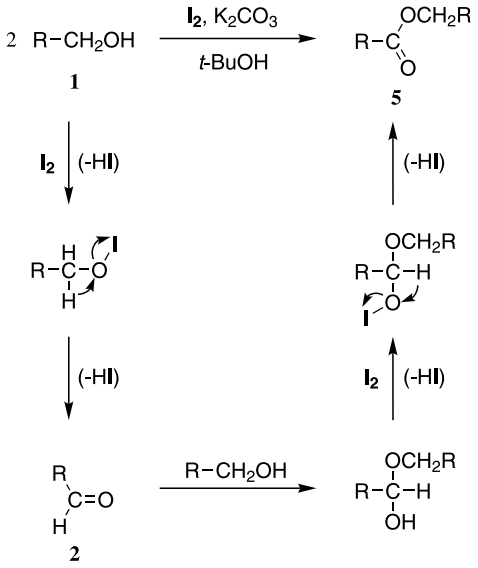
[a] Yield of methyl 3-phenylpropanoate.
[b] Yield of recovered 3-phenylpropanol.
[c] Yield of ethyl 3-phenylpropanoate.

2. Results and discussion

2.1. Preparation of 2,2,2-trifluoroethyl esters

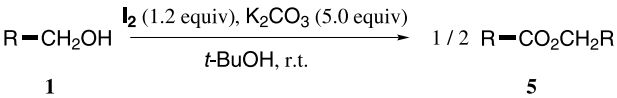
At first, direct oxidative conversion of alcohols to the esters with molecular iodine in the presence of potassium carbonate was carried out. Table 1 shows the effect of a protic polar solvent (5 ml for 1 mmol of alcohol), that is, *t*-butyl alcohol, ethanol, methanol, water, and 2,2,2-trifluoroethanol in the oxidative conversion of *p*-methylbenzyl alcohol to the corresponding esters or acid (with water). It indicates 2,2,2-trifluoroethanol is the best solvent among them, to provide the corresponding 2,2,2-trifluoroethyl ester in high yield (entry 8). Thus, less acidic solvents such as water, methanol, ethanol, *t*-butyl alcohol gave the ester or acid products in low yield under the present conditions. Since it is known that the pK_a value of 2,2,2-trifluoroethanol is ca. 12.4,[32] the result suggests that the acidic polar solvent promotes the present reaction. Probably, 2,2,2-trifluoroethanol activates molecular iodine as an oxidant through a hydrogen bond between its OH proton and molecular iodine. Recently, it has been known that 2,2,2-trifluoroethanol sometimes promotes the reactions; therefore, synthetic use of 2,2,2-trifluoroethanol as a solvent is interesting.[33] For example, 2,2,2-trifluoroethanol activates hydrogen peroxide for the oxidation of thiols to disulfides,[33a] epoxidation of olefins,[33b–d] Mn(III)/Cu(II)-mediated oxidative radical cyclization of α-(methylthio) acetamides,[33e] Et_3N-induced β-cleavage reaction,[33f] etc. The addition of a small amount of methanesulfonic acid to the methanol or ethanol solvent was not effective. The present reaction requires potassium carbonate as a base (entry 6). Though 1,1,1,3,3,3-hexafluoro-2-propanol (pK_a 9.2) is more acidic than 2,2,2-trifluoroethanol, the yield was much reduced due to the poor solubility of molecular iodine (entry 9).

In the present oxidative conversion of alcohols in 2,2,2-trifluoroethanol with molecular iodine and potassium carbonate, it was found that the solvent, 2,2,2-trifluoroethanol, was not oxidized at all by the NMR measurement of the reaction mixture. Here, exactly, aldehyde is formed at

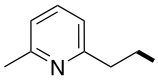


Scheme 2. Plausible reaction pathway for condensed ester.

Table 5. Oxidative condensation of primary alcohols with I_2/K_2CO_3 in *t*-BuOH



1 **5**

Entry	R–	Time (h)	Yields (%)	Entry	R–	Time (h)	Yields (%)
1	3-$O_2NC_6H_4$–	16	95 (**5a**)	10	4-$CH_3OC_6H_4(CH_2)_2$–	27	98 (**5IV**)
2	3,4-$Cl_2C_6H_3$–	48	99 (**5b**)	11	$PhOCH_2$–	42[a]	78 (**5V**)
3	4-$CF_3C_6H_4$–	48	99 (**5c**)	12		27	93 (**5VI**)
4		19	99 (**5k**)	13		19	88 (5VII)
5	4-$CHDOC_6H_4$–	31	3 (97[b])	14		33	76 (**5VIII**)
6	$Ph(CH_2)_2$–	21	96 (**5I**)	15		50	72 (**5IX**)
7		48[c]	97	16	$Br(CH_2)_5$–	42	94[d]
8	$CH_3(CH_2)_6$–	33	97 (**5II**)	17		26	32 (41[b])
9	$CH_3(CH_2)_{11}$–	41	98 (**5III**)				

[a] I_2 (2.4 equiv) was used.
[b] Yield of aldehyde.
[c] Reaction was carried out with 3 g (22 mmol) of alcohol.
[d] Bromine atom was partly substituted by iodine atom (ca. 12%).

first, and, therefore, it can be obtained in high yield when the reaction is carried out at room temperature. For example, the treatment of *p*-methoxybenzyl alcohol, 2,4,6-trimethylbenzyl alcohol, and α-thienylmethanol with molecular iodine (2.5 equiv) and potassium carbonate (2.5 equiv) in 2,2,2-trifluoroethanol at room temperature for 19, 83, and 70 h provided the corresponding aldehydes in 99, 85, and 87% yields together with trace amounts of the corresponding 2,2,2-trifluoroethyl esters, respectively. Though TEMPO-mediated oxidation of alcohols to aldehydes with molecular iodine was reported recently,[34] aldehydes can be formed by the reaction of alcohols with molecular iodine alone in 2,2,2-trifluoroethanol. Once aldehydes are formed, then oxidative conversion to 2,2,2-trifluoroethyl esters proceeds smoothly via the hemiacetals, as shown in Scheme 1.

Based on these results, a variety of primary alcohols were directly oxidized to the corresponding 2,2,2-trifluoroethyl esters in good yields, as shown in Table 2. Benzylic alcohols (entries 1–15), aliphatic primary alcohols (entries 16 and 17), and neopentyl-typed 1-adamantanemethanol (entry 18) could be oxidized to the corresponding 2,2,2-trifluoroethyl esters in good to moderate yields. In benzylic alcohols, formation of the esters with high yields requires 5 equiv each of molecular iodine and potassium carbonate, while in aliphatic primary alcohols, 3 equiv each was required. The present reaction is clean and the esters are obtained in quite good yields from the simple primary alcohols.

2.2. Preparation of methyl esters

When methanol was used as a solvent, instead of 2,2,2-trifluoroethanol, under the same conditions with molecular iodine and potassium carbonate, the formation of methyl esters was observed. However, the yields were not high. When a mixture of alcohol with I_2 and K_2CO_3 in a small amount of methanol was refluxed, the corresponding methyl esters were obtained in high yields, as shown in Table 3. In the present oxidative conversion of alcohols to methyl esters with methanol solvent, methanol is partly oxidized competitively. Therefore, the amount of solvent was quite reduced, and conditions were concentrated (0.3–0.5 ml for 1 mmol of alcohol) to provide the corresponding esters in the best yield.

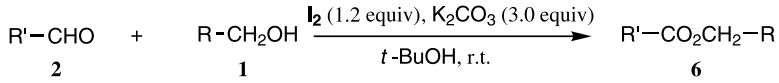
2.3. Preparation of oxidative condensed esters

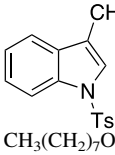
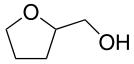
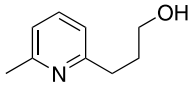
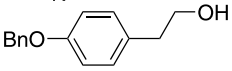
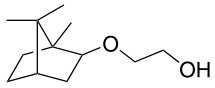
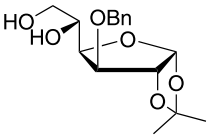
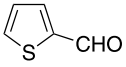
3-Phenylpropanol was treated with molecular iodine and potassium carbonate in various alcoholic solvents (0.5 ml for 1 mmol alcohol), such as methanol, ethanol, isopropyl alcohol, 2,2,2-trifluoroethanol, and *t*-butyl alcohol, at room temperature, and the results are shown in Table 4. Thus, the results indicate that *t*-butyl alcohol is the best solvent, with 1.2 equiv of molecular iodine and 5.0 equiv of potassium carbonate, to provide 3-phenylpropyl 3-phenylpropanoate in high yields (entries 5–8).

The reaction probably proceeds as follows: oxidation of alcohol to aldehyde by molecular iodine, formation of hemiacetal by the reaction of the formed aldehyde and alcohol, and then, oxidation of the hemiacetal to an ester by molecular iodine, as shown in Scheme 2.

Based on these results, various benzylic alcohols (entries 1–4) and primary alcohols (entries 6–16) could be directly converted to the corresponding oxidatively condensed esters in high yields in *t*-butyl alcohol at room temperature, except for *p*-methoxybenzyl alcohol which has an electron-donating group (entry 5), and 1-adamantanemethanol which is a neopentyl-type alcohol (entry 17), as shown in Table 5. When the reaction was carried out with 3 g

Table 6. Stoichiometrically oxidative condensation of aldehydes and alcohols with I_2/K_2CO_3 in *t*-BuOH



Entry	R′-CHO (1.0 equiv)	R-CH_2OH (1.05 equiv)	Time (h)	Yields (%)
1	$PhCH_2CH_2CHO$	4-$ClC_6H_4CH_2OH$	22	90 (**6d**)
2	$PhCH_2CH_2CHO$	4-$CH_3C_6H_4CH_2OH$	22	87 (**6f**)
3	$PhCH_2CH_2CHO$		22[a]	61 (**6m**)
4	$PhCH_2CH_2CHO$	$CH_3(CH_2)_7OH$	22	91 (**6II**)
5	$PhCH_2CH_2CHO$	$CH_3(CH_2)_{12}OH$	24	84 (**6III**)
6	$PhCH_2CH_2CHO$	$PhO(CH_2)_2OH$	22	89 (**6V**)
7	$PhCH_2CH_2CHO$		22	91 (**6VIII**)
8	$PhCH_2CH_2CHO$		22	76 (**6IX**)
9	$PhCH_2CH_2CHO$		22	**80** (**6XI**)
10	$PhCH_2CH_2CHO$		25	84 (**6XII**)
11	$PhCH_2CH_2CHO$		23	63[b] (**6XIII**)
12	$CH_3(CH_2)_3CHO$	$CH_3(CH_2)_7OH$	22	87 (**6II′**)
13	$PhCH(CH_3)CHO$	$CH_3(CH_2)_7OH$	22	62
14	PhCHO	$CH_3(CH_2)_7OH$	22	47 (46[c])
15		$CH_3(CH_2)_7OH$	22	20 (45[c])

[a] Aldehyde/alcohol = 1.5/1.0 (equiv), and reaction temperature was 45 °C.
[b] Yield of mono ester.
[c] Yield of octyl octanoate (**5II**).

(22 mmol) of 3-phenylpropanol, instead of 1 mmol scale, 3-phenylpropyl 3-phenylpropanoate was obtained again in high yield (entry 7). Thus, this reaction can be carried out easily for large-scale preparation of oxidatively condensed esters from primary alcohols.

2.4. Stoichiometrically oxidative condensation of aldehydes and alcohols to esters

At first, esterification of 3-phenylpropanal with various alcohols was carried out using iodine and potassium carbonate in *t*-butyl alcohol at room temperature (entries 1–11). Next, some of the aldehydes with 1-octanol were examined (entries 12–15). The results are summarized in Table 6, and indicate that the oxidative esterification of aldehydes bearing a primary alkyl group, with primary alcohols proceeded smoothly to provide the corresponding esters in high yields. The reactions of aldehydes bearing a primary alkyl group, with secondary alcohols, or aldehydes bearing a secondary alkyl group, with alcohols, gave the corresponding esters in moderate to low yields. Thus the present oxidative condensation of aldehydes with alcohols is rather sensitive to steric hindrance and electronic effect, since the formation of hemiacetal is the key step.

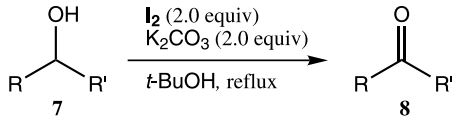
2.5. Preparation of ketones

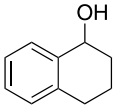
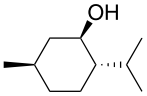
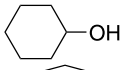
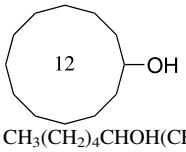
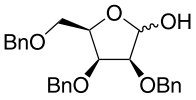
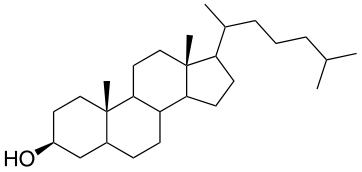
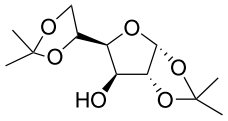
Benzylic (entries 1–5) and aliphatic (entries 6–12) alcohols could be also oxidized in *t*-butyl alcohol with molecular iodine and potassium carbonate under the refluxing conditions smoothly, to the corresponding ketones in high yields, as shown in Table 7. 2,3,5-Tri-*O*-benzyl-D-ribofuranose and cholestanol were oxidized to 2,3,5-tri-*O*-benzyl-D-ribofuranone and cholestanone in good yields, respectively (entries 11 and 12). Generally, purine-like *C*-nucleosides such as formycin A and formycine B were synthesized from 2,3,5-tri-*O*-benzyl-D-ribofuranone.[35] However, diacetone-D-glucose was not oxidized at all, even for long reaction time. Probably, molecular iodine cannot approach the hydroxyl group, due to the steric hindrance (entry 13).

3. Conclusion

The present method is a simple, efficient, and high-yield procedure for the oxidative conversion of primary alcohols to the corresponding esters, condensed esters, and secondary alcohols to the corresponding ketones, with molecular

Table 7. Oxidation of secondary alcohols to ketones with I_2/K_2CO_3 in *t*-BuOH



Entry	Secondary alcohol	Time (h)	Yield (%)
1	$PhCHOHC_2H_5$	8	99
2	$4\text{-}ClC_6H_4CHOHCH_3$	6	86
3	$4\text{-}CH_3C_6H_4CHOHCH_3$	5	99
4	$PhCHOH(CH_2)_7CH_3$	6	99
5		7	99
6		16	99
7		4	78
8		6	99
9	$CH_3(CH_2)_4CHOH(CH_2)_4CH_3$	5	99
10	$Ph(CH_2)_3CHOHCH_3$	6	99
11		8	79
12		40	90
13		20	0 (88[a])

[a] Yield of starting alcohol.

iodine and potassium carbonate in 2,2,2-trifluoroethanol, methanol, and *t*-butyl alcohol. The advantages of the present reactions are the operational simplicity, elimination of the use of toxic oxidants or solvents, generality of the reactions, and high yield of the products.

4. Experimental

4.1. General

^1H NMR and ^{13}C NMR spectra were obtained with JEOL-JNM-LA-400, JEOL-JNM-LA-400s, and JEOL-JNM-LA-500 spectrometers. Chemical shifts are expressed in ppm downfield from tetramethylsilane (TMS) in δ units. IR spectra were measured with JASCO FT/IR-200 and FT/IR-4100 spectrometers. Mass spectra were recorded on JEOL-HX-110 and JEOL-JMS-ATII15 spectrometers. Melting points were determined on Yamato melting points apparatus Model MP-21. Silica Gel 60 (Kanto Kagaku Co.) was used for column chromatography and Wakogel B-5F was used for preparative TLC.

Most of the organic chemical substrates are commercially available. All the compounds gave satisfactory spectroscopic data, most methyl esters and ketones were identified with commercially available authentic materials.[36]

4.2. Typical procedure for oxidative conversion of primary alcohols to 2,2,2-trifluoroethyl esters

To a solution of benzyl alcohol (1 mmol) in CF_3CH_2OH (5 ml) were added I_2 (2.5 mmol) and K_2CO_3 (2.5 mmol) under an argon atmosphere. The mixture obtained was stirred for 2 h at 50 °C, then I_2 (2.5 mmol) and K_2CO_3 (2.5 mmol) were added to the mixture again. After 3 h at the same temperature, the mixture was quenched with satd aq Na_2SO_3 (3–5 ml) at 0 °C, and was extracted with Et_2O three times. The organic layer was washed with brine and dried over Na_2SO_4 to provide 2,2,2-trifluoroethyl benzoate in 91% yield in an almost pure state. If necessary, the product was purified by flash column chromatography on silica gel (hexane–EtOAc=4:1) to give pure 2,2,2-trifluoroethyl benzoate as an oil.

4.2.1. 2,2,2-Trifluoroethyl 3-nitrobenzoate (3a). Colorless solid; mp 46–47 °C; IR (KBr): 1750, 1300, 1250, 1170, 1130 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.78 (2H, q, J=8.4 Hz), 7.73 (1H, t, J=8.1 Hz), 8.42 (1H, dt, J=8.1, 1.2 Hz), 8.49 (2H, ddd, J=8.1, 2.2, 1.2 Hz), 8.91 (1H, t, J=2.2 Hz); HRMS (FAB): obsd M+H=250.0320, calcd for $C_9H_7F_3NO_4$ M+H=250.0327.

4.2.2. 2,2,2-Trifluoroethyl 3,4-dichlorobenzoate (3b). Colorless solid; mp 32–33 °C; IR (KBr): 1740, 1300, 1265, 1235, 1170, 1110 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.71 (2H, q, J=8.4 Hz), 7.57 (1H, d, J=8.4 Hz), 7.90 (1H, dd, J=8.4, 2.0 Hz), 8.15 (1H, d, J=2.0 Hz); HRMS (EI): obsd M+=271.9624, calcd for $C_9H_5Cl_2F_3O_2$ M+=271.9619.

4.2.3. 2,2,2-Trifluoroethyl 4-trifluoromethylbenzoate (3c). Oil; IR (neat): 1745, 1165, 1130, 1100, 1065 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.74 (2H, q, J=8.3 Hz), 7.75 (2H, d, J=8.1 Hz), 8.20 (2H, d, J=8.1 Hz); HRMS (EI): obsd M+=272.0293, calcd for $C_{10}H_6F_6O_2$ M+=272.0298.

4.2.4. 2,2,2-Trifluoroethyl 4-chlorobenzoate (3d). Oil; IR (neat): 1740, 1295, 1255, 1170, 1105 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.70 (2H, q, J=8.4 Hz), 7.46 (2H, d, J=8.8 Hz), 8.02 (2H, d, J=8.8 Hz); HRMS (EI): obsd M+=238.0005, calcd for $C_9H_6ClF_3O_2$ M+=238.0008.

4.2.5. 2,2,2-Trifluoroethyl benzoate (3e). Oil; bp 84–86 °C/19 mm Hg (lit.[37] bp 77 °C/13 mm Hg); IR (neat): 1740, 1295, 1255, 1170, 1115 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.70 (2H, q, J=8.4 Hz), 7.48 (2H, t, J=7.7 Hz), 7.62 (1H, tt, J=7.7, 1.4 Hz), 8.09 (2H, dt, J=7.7, 1.4 Hz); HRMS (EI): obsd M+=204.0413, calcd for $C_9H_7F_3O_2$ M+=204.0398.

4.2.6. 2,2,2-Trifluoroethyl *p*-toluate (3f). Oil; IR (neat): 1740, 1295, 1260, 1170, 1110 cm^{-1}; ^1H NMR ($CDCl_3$) δ=2.43 (3H, s), 4.68 (2H, q, J=8.4 Hz), 7.27 (2H, d,

J=8.1 Hz), 7.97 (2H, d, J=8.1 Hz); HRMS (EI): obsd M+=218.0561, calcd for $C_{10}H_9F_3O_2$ M+=218.0555.

4.2.7. 2,2,2-Trifluoroethyl 4-methoxybenzoate (3g). Oil; IR (neat): 1735, 1255, 1165 cm^{-1}; ^1H NMR ($CDCl_3$) δ=3.88 (3H, s), 4.67 (2H, q, J=8.4 Hz), 6.95 (2H, d, J=9.0 Hz), 8.03 (2H, d, J=9.0 Hz); HRMS (EI): obsd M+=234.0494, calcd for $C_{10}H_9F_3O_3$ M+=234.0504.

4.2.8. 2,2,2-Trifluoroethyl 2,5-dimethylbenzoate (3h). Oil; ^1H NMR ($CDCl_3$) δ=2.36 (3H, s), 2.56 (3H, s), 4.67 (2H, q, J=8.4 Hz), 7.16 (1H, d, J=8.0 Hz), 7.26 (1H, d, J=8.0 Hz), 7.77 (1H, s); HRMS (EI): obsd M+=232.0708, calcd for $C_{11}H_{11}F_3O_2$ M+=232.0711.

4.2.9. 2,2,2-Trifluoroethyl 3,4,5-trimethoxybenzoate (3i). Colorless solid; mp 67–68 °C; IR (KBr): 1735, 1160, 1130 cm^{-1}; ^1H NMR ($CDCl_3$) δ=3.92 (6H, s), 3.93 (3H, s), 4.70 (2H, q, J=8.4 Hz), 7.32 (2H, s); HRMS (FAB): obsd M+=294.0698, calcd for $C_{12}H_{13}F_3O_5$ M+=294.0715.

4.2.10. 2,2,2-Trifluoroethyl 1-naphthoate (3j). Colorless solid; mp 28–29 °C; IR (KBr): 1720, 1160, 1125 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.79 (2H, q, J=8.4 Hz), 7.55 (2H, m), 7.66 (1H, td, J=7.8, 1.4 Hz), 7.91 (1H, d, J=7.8 Hz), 8.09 (1H, dd, J=7.8, 1.4 Hz), 8.29 (1H, d, J=7.8 Hz), 8.92 (1H, d, J=7.8 Hz); HRMS (FAB): obsd M+=254.0549, calcd for $C_{13}H_9F_3O_2$ M+=254.0555.

4.2.11. 2,2,2-Trifluoroethyl nicotinate (3k). Oil; IR (neat): 1745, 1300, 1260, 1180, 1120 cm^{-1}; ^1H NMR ($CDCl_3$) δ= 4.75 (2H, q, J=8.3 Hz), 7.45 (1H, dd, J=8.0, 4.9 Hz), 8.34 (1H, dt, J=8.0, 1.9 Hz), 8.85 (1H, dd, J=4.9, 1.9 Hz), 9.28 (1H, d, J=1.9 Hz); HRMS (EI): obsd M+=205.0348, calcd for $C_8H_6F_3NO_2$ M+=205.0351.

4.2.12. 2,2,2-Trifluoroethyl thiophene-2-carboxylate (3l). Oil; IR (neat): 1725, 1250, 1160, 1095 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.68 (2H, q, J=8.4 Hz), 7.15 (1H, dd, J=5.0, 3.9 Hz), 7.66 (1H, dd, J=5.0, 1.2 Hz), 7.89 (1H, dd, J=3.9, 1.2 Hz); HRMS (EI): obsd M+=209.9967, calcd for $C_7H_5F_3O_2S$ M+=209.9962.

4.2.13. 2,2,2-Trifluoroethyl tridecanoate (3III). Oil; ^1H NMR ($CDCl_3$) δ=0.88 (3H, t, J=6.9 Hz), 1.22–1.32 (18H, m), 1.61 (2H, q, J=7.5 Hz), 2.41 (2H, t, J=7.5 Hz), 4.46 (2H, q, J=8.4 Hz); HRMS (EI): obsd M+=296.1957, calcd for $C_{15}H_{27}F_3O_2$ M+=296.1963.

4.2.14. 2,2,2-Trifluoroethyl cyclohexanecarboxylate (3VI). Oil; ^1H NMR ($CDCl_3$) δ=1.28 (3H, m), 1.47 (2H, m), 1.65 (1H, m), 1.75 (2H, m), 1.94 (2H, m), 2.42 (1H, tt, J=11.1, 3.6 Hz), 4.47 (2H, q, J=8.5 Hz); HRMS (EI): obsd M+=210.0860, calcd for $C_9H_{13}F_3O_2$ M+=210.0868.

4.2.15. 2,2,2-Trifluoroethyl adamantanecarboxylate (3X). Oil; bp 90–100 °C/1 mm Hg; IR (neat): 1745, 1160, 1080 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.73 (6H, m), 1.93 (6H, m), 2.04 (3H, m), 4.45 (2H, q, J=8.4 Hz); HRMS (EI): obsd M+=262.1178, calcd for $C_{13}H_{17}F_3O_2$ M+=262.1181.

4.3. Typical procedure for oxidative conversion of primary alcohols to methyl esters

To a solution of 4-chlorobenzyl alcohol (1 mmol) in CH_3OH (0.5 ml) were added I_2 (3 mmol) and K_2CO_3 (3 mmol) under an argon atmosphere. The mixture obtained was stirred at 70 °C. After 21 h at the same temperature, the mixture was quenched with satd aq Na_2SO_3 (3–5 ml) at 0 °C, and was extracted with Et_2O three times. The organic layer was washed with brine and dried over Na_2SO_4 to provide methyl 4-chlorobenzoate in 93% yield in an almost pure state. If necessary, the product was purified by flash column chromatography on silica gel (hexane–EtOAc= 8:1) to give pure methyl 4-chlorobenzoate as a colorless solid. Mp 43 °C (lit.[36] mp 43 °C). ^1H NMR ($CDCl_3$): δ=3.92 (3H, s), 7.42 (2H, d, J=8.8 Hz), 7.98 (2H, d, J=8.8 Hz).

4.4. Typical procedure for oxidative condensation of primary alcohols to esters

To a solution of 3-phenylpropanol (1 mmol) in *t*-butyl alcohol (0.5 ml) were added I_2 (1.2 mmol) and K_2CO_3 (5.0 mmol) under an argon atmosphere. The obtained mixture was stirred at room temperature, until the iodine color almost disappeared. After 21 h at the same temperature, the mixture was quenched by the addition of water (20 ml), Et_2O (5 ml), and satd aq Na_2SO_3 (0.5 ml) at 0 °C. Then the mixture was extracted with Et_2O three times. The organic layer was washed with brine and dried over Na_2SO_4 to provide 3-phenylpropyl 3-phenylpropanoate in 96% yield in an almost pure state. If necessary, the product was purified by flash column chromatography on silica gel (hexane–EtOAc=10:1) to give pure 3-phenylpropyl 3-phenylpropanoate as a colorless oil.

4.4.1. 3-Nitrobenzyl 3-nitrobenzoate (5a). Colorless solid; mp 146–147 °C; IR (KBr): 1720, 1280, 1265, 1140 cm^{-1}; ^1H NMR ($CDCl_3$) δ=5.52 (2H, s), 7.62 (1H, t, J=8.1 Hz), 7.70 (1H, t, J=8.1 Hz), 7.82 (1H, m), 8.24 (1H, m), 8.34 (1H, m), 8.41 (1H, m), 8.45 (1H, m), 8.89 (1H, m); ^{13}C NMR ($CDCl_3$) δ=66.1 (s), 123.2 (t), 123.3 (t), 123.6 (t), 124.7 (t), 127.8 (t), 129.8 (t), 131.3 (q), 134.3 (t), 135.4 (t), 137.3 (q), 148.3 (q), 148.4 (q), 164.1 (q); HRMS (EI): obsd M+=302.0541, calcd for $C_{14}H_{10}N_2O_6$ M+=302.0539.

4.4.2. 3,4-Dichlorobenzyl 3,4-dichlorobenzoate (5b). Colorless solid; mp 90–91 °C; IR (KBr): 1725, 1295, 1270, 1245, 1110 cm^{-1}; ^1H NMR ($CDCl_3$) δ=5.30 (2H, s), 7.27 (1H, dd, J=8.3, 2.0 Hz), 7.47 (1H, d, J=8.3 Hz), 7.53 (2H, m), 7.88 (1H, dd, J=8.3, 2.0 Hz), 8.12 (1H, d, J=2.0 Hz); ^{13}C NMR ($CDCl_3$) δ=65.7 (s), 127.6 (t), 128.7 (t), 129.5 (q), 130.3 (t), 130.6 (t), 130.7 (t), 131.6 (t), 132.8 (q), 132.9 (q), 133.1 (q), 135.6 (q), 138.0 (q), 164.3 (q); HRMS (EI): obsd M+=347.9285, calcd for $C_{14}H_8Cl_4O_2$ M+=347.9278.

4.4.3. 4-(Trifluoromethyl)benzyl 4-(trifluoromethyl)-benzoate (5c). Colorless solid; mp 57–58 °C (lit.[38] mp 61–62 °C); IR (KBr): 1720, 1325, 1270, 1105, 1065 cm^{-1}; ^1H NMR ($CDCl_3$) δ=5.45 (2H, s), 7.57 (2H, d, J=8.2 Hz), 7.66 (2H, d, J=8.0 Hz), 7.72 (2H, d, J=8.2 Hz), 8.19 (2H, d, J=8.0 Hz); ^{13}C NMR ($CDCl_3$) δ=66.2 (s), 125.5 (t),

125.7 (t), 128.3 (t), 130.1 (t), 130.6 (q), 133.0 (q), 134.7 (q), 139.5 (q), 165.0 (q); HRMS (FAB): obsd M+H= 349.0661, calcd for $C_{16}H_{11}O_2F_6$ M+H=349.0663.

4.4.4. 3-Pyridinylmethyl nicotinate (5k). Oil; IR (neat): 1720, 1275, 1110 cm^{-1}; ^1H NMR ($CDCl_3$) δ=5.42 (2H, s), 7.35 (1H, ddd, J=7.9, 4.9, 0.8 Hz), 7.41 (1H, ddd, J=7.9, 4.9, 0.8 Hz), 7.81 (1H, dt, J=7.9, 2.0 Hz), 8.32 (1H, dt, J= 7.9, 2.0 Hz), 8.62 (1H, dd, J=4.9, 1.5 Hz), 8.74 (1H, d, J= 1.5 Hz), 8.80 (1H, dd, J=4.9, 2.0 Hz), 9.25 (1H, dd, J=2.0, 0.8 Hz); ^{13}C NMR ($CDCl_3$) δ=64.5 (s), 123.3 (t), 123.5 (t), 125.6 (q), 131.1 (q), 136.1 (t), 137.1 (t), 149.7 (t), 149.8 (t), 149.9 (t), 150.9 (t), 153.7 (t), 164.9 (q); HRMS (FAB): obsd M+H=215.0814, calcd for $C_{12}H_{11}N_2O_2$ M+H= 215.0821.

4.4.5. 3-Phenylpropyl 3-phenylpropanoate (5I). Oil; bp 210 °C/1 mm Hg (lit.[8b] bp 146–150 °C/0.8 mm Hg); IR (neat): 1730, 1160, 1030 cm^{-1}; ^1H NMR ($CDCl_3$) δ= 1.93 (2H, m), 2.63 (2H, t, J=7.7 Hz), 2.64 (2H, t, J= 7.7 Hz), 2.95 (2H, t, J=7.7 Hz), 4.09 (2H, t, J=6.5 Hz), 7.18 (6H, m) 7.28 (4H, m); ^{13}C NMR ($CDCl_3$) δ=30.2 (s), 31.0 (s), 32.1 (s), 35.9 (s), 63.8 (s), 126.0 (t), 126.3 (t), 128.3 (t), 128.4 (t), 128.5 (t), 140.5 (q), 141.2 (q), 173.0 (q); HRMS (FAB): obsd M+H=269.1552, calcd for $C_{18}H_{21}O_2$ M+H=269.1542.

4.4.6. Octyl octanoate (5II). Oil; IR (neat): 1735, 1460, 1165 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.88 (3H, t, J=7.0 Hz), 0.88 (3H, t, J=7.0 Hz), 1.25–1.40 (18H, m), 1.62 (4H, t, J=7.5 Hz), 2.29 (2H, t, J=7.5 Hz), 4.06 (2H, t, J=6.7 Hz); ^{13}C NMR ($CDCl_3$) δ=14.0 (p), 22.6 (s), 25.0 (s), 25.9 (s), 28.6 (s), 28.9 (s), 29.1 (s), 29.2 (s), 31.6 (s), 31.7 (s), 34.4 (s), 64.3 (s), 173.9 (q).

4.4.7. Tridecyl tridecanoate (5III). Colorless solid; mp 35–36 °C; IR (KBr): 1735, 1370, 1230, 1205, 1185 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.88 (6H, t, J=6.9 Hz), 1.25–1.38 (38H, m), 1.63 (4H, m), 2.29 (2H, t, J=7.6 Hz), 4.05 (2H, t, J=6.7 Hz); ^{13}C NMR ($CDCl_3$) δ=14.1 (p), 22.7 (s), 25.0 (s), 26.0 (s), 28.7 (s), 29.2 (s), 29.3 (s), 29.5 (s), 29.6 (s), 29.7 (s), 31.9 (s), 34.4 (s), 64.4 (s), 174.0 (q); HRMS (FAB): obsd M+H=397.4037, calcd for $C_{26}H_{53}O_2$ M+H= 397.4046.

4.4.8. 3-(4′-Methoxyphenyl)propyl 3-(4′-methoxyphenyl) propanoate (5IV). Colorless solid; mp 38–39 °C; IR (KBr): 1730, 1510, 1245, 1030 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.89 (2H, tt, J=7.9, 6.5 Hz), 2.58 (2H, t, J=7.9 Hz), 2.60 (2H, t, J=7.9 Hz), 2.89 (2H, t, J=7.9 Hz), 3.77 (3H, s), 3.78 (3H, s), 4.07 (2H, t, J=6.5 Hz), 6.83 (4H, dd, J=7.8, 2.1 Hz), 7.06 (2H, d, J=8.7 Hz), 7.13 (2H, d, J=8.7 Hz); ^{13}C NMR ($CDCl_3$) δ=30.1 (s), 30.3 (s), 31.1 (s), 36.1 (s), 55.2 (p), 63.7 (s), 113.7 (t), 113.8 (t), 129.2 (t), 132.5 (q), 133.2 (q), 157.8 (q), 158.0 (q), 173.0 (q); HRMS (FAB): obsd M+ H=328.1671, calcd for $C_{20}H_{24}O_4$ M+H=328.1675.

4.4.9. 2-Phenoxyethyl 2-phenoxyacetate (5V). Colorless solid; mp 82–83 °C (lit.[39] mp 83–84 °C); IR (KBr): 1740, 1365, 1215 cm^{-1}; ^1H NMR ($CDCl_3$) δ=4.19 (2H, t, J= 4.7 Hz), 4.57 (2H, t, J=4.7 Hz), 4.67 (2H, s), 6.90 (4H, m), 6.98 (2H, m), 7.28 (4H, m); ^{13}C NMR ($CDCl_3$) δ=63.5 (s), 65.2 (s), 65.6 (s), 114.6 (t), 114.7 (t), 121.3 (t), 121.8 (t), 129.5 (t), 157.7 (q), 158.3 (q), 168.9 (q); HRMS (FAB): obsd M+ =272.1059, calcd for $C_{16}H_{16}O_4$ M+ =272.1049.

4.4.10. Cyclohexylmethyl cyclohexanecarboxylate (5VI). Oil; IR (neat): 1730, 1170, 1130 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.97 (2H, m), 1.12–1.32 (6H, m), 1.44 (2H, m), 1.57–1.78 (9H, m), 1.90 (2H, m), 2.29 (1H, tt, J=11.3, 3.6 Hz), 3.87 (2H, d, J=6.4 Hz); ^{13}C NMR ($CDCl_3$) δ=25.5 (s), 25.7 (s), 25.8 (s), 26.4 (s), 29.1 (s), 29.7 (s), 37.2 (t), 43.3 (t), 69.2 (s), 176.2 (q); HRMS (FAB): obsd M+H=225.1858, calcd for $C_{14}H_{25}O_2$ M+H=225.1855.

4.4.11. Cyclopentylmethyl cyclopentanecarboxylate (5VII). Oil; IR (neat): 1730, 1180, 1155 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.25 (2H, m), 1.50–1.64 (6H, m), 1.65–1.84 (6H, m), 1.88 (2H, m), 2.20 (1H, septet, J=7.5 Hz), 2.73 (1H, quint, J=7.9 Hz), 3.96 (2H, d, J=7.5 Hz); ^{13}C NMR ($CDCl_3$) δ=25.3 (s), 25.8 (s), 29.3 (s), 30.0 (s), 38.6 (t), 44.0 (t), 68.2 (s), 176.9 (q); HRMS (FAB): obsd M+H= 197.1531, calcd for $C_{12}H_{21}O_2$ M+H=197.1542.

4.4.12. (Tetrahydrofuran-2-yl)methyltetrahydrofuran-2-carboxylate (5VIII). Oil; IR (neat): 1745, 1200, 1175, 1080 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.62 (1H, m), 1.80–2.10 (6H, m), 2.26 (1H, m), 3.80 (1H, m), 3.90 (2H, m), 4.00–4.18 (3H, m), 4.22 (1H, m), 4.51 (1H, m); ^{13}C NMR ($CDCl_3$) δ=25.2 (s), 25.6 (s), 25.6 (s), 27.9 (s), 30.2 (s), 66.6 (s), 68.4 (s), 69.3 (s), 76.3 (t), 76.4 (t), 76.6 (t), 173.4 (q); HRMS (FAB): obsd M+H=201.1145, calcd for $C_{10}H_{17}O_4$ M+H=201.1127.

4.4.13. 3-(6′-Methylpyridin-2′-yl)propyl 3-(6′-methylpyridin-2′-yl)propanoate (5IX). Oil; IR (neat): 1730, 1455, 1160 cm^{-1}; ^1H NMR ($CDCl_3$) δ=2.04 (2H, tt, J= 7.6, 6.5 Hz), 2.51 (3H, s), 2.52 (3H, s), 2.78 (4H, tt, J=7.6, 3.1 Hz), 3.07 (2H, t, J=7.6 Hz), 4.11 (2H, t, J=6.5 Hz), 6.91 (1H, d, J=7.6 Hz), 6.97 (3H, m), 7.47 (2H, t, J= 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ=24.5 (p), 28.7 (s), 33.0 (s), 33.8 (s), 34.6 (s), 63.9 (s), 119.6 (t), 119.7 (t), 120.7 (t), 120.8 (t), 136.6 (t), 157.9 (q), 159.4 (q), 160.3 (q), 173.1 (q); HRMS (EI): obsd M+ =298.1678, calcd for $C_{18}H_{22}N_2O_2$ M+ =298.1681.

4.5. Typical procedure for stoichiometrically oxidative condensation of aldehydes with primary alcohols to esters

To a solution of 3-phenylpropanal (1 mmol) and 1-octanol (1.05 mmol) in *t*-butyl alcohol (0.5 ml) were added I_2 (1.2 mmol) and K_2CO_3 (3.0 mmol) under an argon atmosphere. The obtained mixture was stirred at room temperature, until the iodine color almost disappeared. After 22 h at the same temperature, the mixture was quenched by the addition of water (20 ml), Et_2O (5 ml), and satd aq Na_2SO_3 (0.5 ml) at 0 °C. Then the mixture was extracted with Et_2O three times. The organic layer was washed with brine and dried over Na_2SO_4 to provide octyl 3-phenylpropanoate in 91% yield. If necessary, the product was purified by flash column chromatography on silica gel (hexane–EtOAc=10:1) to give pure octyl 3-phenylpropanoate as a colorless oil.

4.5.1. 4-Chlorobenzyl 3-phenylpropanoate (6d). Oil; IR (neat): 1735, 1495, 1145, 1090 cm^{-1}; ^1H NMR ($CDCl_3$)

δ=2.68 (2H, t, J=7.8 Hz), 2.96 (2H, t, J=7.8 Hz), 5.06 (2H, s), 7.19 (5H, m), 7.28 (4H, m); ^{13}C NMR ($CDCl_3$) δ= 30.9 (s), 35.8 (s), 65.4 (s), 126.3 (t), 128.3 (t), 128.5 (t), 128.7 (t), 129.5 (t), 134.1 (q), 134.4 (q), 140.3 (q), 172.6 (q); HRMS (FAB): obsd M+H=275.0839, calcd for $C_{16}H_{16}O_2Cl$ M+H=275.0839.

4.5.2. 4-Methylbenzyl 3-phenylpropanoate (6f). Oil; IR (neat): 1730, 1150 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=2.34 (3H, s), 2.66 (2H, t, J=7.8 Hz), 2.95 (2H, t, J=7.8 Hz), 5.06 (2H, s), 7.13–7.22 (7H, m), 7.26 (2H, m); ^{13}C NMR ($CDCl_3$) δ=21.1 (p), 30.9 (s), 35.9 (s), 66.2 (s), 126.2 (t), 128.3 (t), 128.4 (t), 128.5 (t), 129.2 (t), 132.9 (q), 138.0 (q), 140.4 (q), 172.7 (q); HRMS (FAB): obsd M+H=255.1392, calcd for $C_{17}H_{19}O_2$ M+H=255.1385.

4.5.3. (*N*-Tosyl-indol-3-yl)methyl 3-phenylpropanoate (6m). Oil; ^{1}H NMR ($CDCl_3$) δ=2.34 (3H, s), 2.65 (2H, t, J=7.8 Hz), 2.94 (2H, t, J=7.8 Hz), 5.22 (2H, s), 7.12–7.27 (7H, m), 7.34 (1H, t, 7.6 Hz), 7.47 (1H, d, 7.6 Hz), 7.60 (1H, s), 7.78 (2H, d, 7.6 Hz), 7.97 (1H, d, 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ=21.5 (p), 30.8 (s), 35.7 (s), 57.7 (s), 113.6 (t), 117.2 (q), 119.7 (t), 123.4 (t), 125.0 (t), 125.6 (t), 126.2 (t), 126.9 (t), 128.2 (t), 128.4 (t), 129.4 (q), 129.9 (t), 135.0 (q), 135.1 (q), 140.2 (q), 145.1 (q), 172.7 (q); HRMS (FAB): obsd M+ =433.1339, calcd for $C_{25}H_{23}NO_4S$ M+ = 433.1348.

4.5.4. Octyl 3-phenylpropanoate (6II). Oil; IR (neat): 1735, 1160 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.88 (3H, t, J= 7.0 Hz), 1.22–1.38 (10H, m), 1.59 (2H, m), 2.63 (2H, t, J= 7.9 Hz), 2.95 (2H, t, J=7.9 Hz), 4.06 (2H, t, J=6.8 Hz), 7.20 (3H, m), 7.29 (2H, m); ^{13}C NMR ($CDCl_3$) δ=14.1 (p), 22.6 (s), 25.9 (s), 28.6 (s), 29.1 (s), 29.2 (s), 31.0 (s), 31.8 (s), 35.9 (s), 64.6 (s), 126.2 (t), 128.3 (t), 128.4 (t), 140.5 (q), 173.0 (q); HRMS (FAB): obsd M+H=263.2017, calcd for $C_{17}H_{27}O_2$ M+H=263.2011.

4.5.5. Tridecyl 3-phenylpropanoate (6III). Oil; IR (neat): 1735, 1455, 1160 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.88 (3H, t, J=7.0 Hz), 1.22–1.38 (20H, m), 1.63 (2H, m), 2.62 (2H, t, J=7.9 Hz), 2.95 (2H, t, J=7.9 Hz), 4.06 (2H, t, J=6.7 Hz), 7.20 (3H, m), 7.28 (2H, m); ^{13}C NMR ($CDCl_3$) δ=14.2 (p), 22.8 (s), 26.0 (s), 28.7 (s), 29.3 (s), 29.4 (s), 29.6 (s), 29.7 (s), 29.8 (s), 31.1 (s), 32.0 (s), 36.0 (s), 64.7 (s), 126.3 (t), 128.4 (t), 128.5 (t), 140.7 (q), 173.1 (q).

4.5.6. 2-Phenoxyethyl 3-phenylpropanoate (6V). Colorless solid; mp 52–53 °C; IR (KBr): 1730, 1240, 1160 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=2.67 (2H, t, J=7.8 Hz), 2.95 (2H, t, J=7.8 Hz), 4.12 (2H, t, J=4.7 Hz), 4.42 (2H, t, J=4.7 Hz), 6.90 (2H, d, J=7.5 Hz), 6.96 (1H, t, J=7.5 Hz) 7.19 (3H, m), 7.26 (4H, m); ^{13}C NMR ($CDCl_3$) δ=30.8 (s), 35.7 (s), 62.8 (s), 65.8 (s), 114.6 (t), 121.1 (t), 126.2 (t), 128.3 (t), 128.5 (t), 129.5 (t), 140.3 (q), 158.4 (q), 172.8 (q); HRMS (FAB): obsd M+H=271.1324, calcd for $C_{17}H_{19}O_3$ M+ H=271.1334.

4.5.7. (Tetrahydrofuran-2-yl)methyl 3-phenyl-propanoate (6VIII). Oil; IR (neat): 1730, 1160, 1075 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=1.55 (2H, m), 1.90 (3H, m), 2.68 (2H, t, J=8.0 Hz), 2.96 (2H, t, J=8.0 Hz), 3.79 (1H, m), 3.87 (1H, m), 4.01 (1H, m), 4.09 (1H, m), 4.16 (1H, m), 7.23 (3H, m), 7.28 (2H, m); ^{13}C NMR ($CDCl_3$) δ=25.6 (s), 27.9 (s), 30.9 (s), 35.7 (s), 66.5 (s), 68.4 (s), 76.4 (t), 126.2 (t), 128.2 (t), 128.4 (t), 140.4 (q), 172.8 (q); HRMS (FAB): obsd M+H= 235.1314, calcd for $C_{14}H_{19}O_3$ M+H=235.1334.

4.5.8. 3-(6′-Methylpyridin-2′-yl)propyl 3-phenyl-propanoate (6IX). Oil; IR (neat): 1730, 1455, 1155 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=2.04 (2H, m), 2.52 (3H, s), 2.63 (2H, t, J=7.7 Hz), 2.78 (2H, t, J=7.7 Hz), 2.95 (2H, t, J=7.7 Hz), 4.12 (2H, t, J=6.6 Hz), 6.90 (1H, d, J=7.6 Hz), 6.96 (1H, d, J=7.6 Hz), 7.20 (3H, m), 7.28 (2H, m), 7.47 (1H, t, J= 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ=24.5 (p), 28.7 (s), 30.9 (s), 34.7 (s), 35.8 (s), 64.0 (s), 119.6 (t), 120.7 (t), 126.2 (t), 128.3 (t), 128.5 (t), 136.6 (t), 140.5 (q), 157.9 (q), 160.3 (q), 172.9 (q); HRMS (FAB): obsd M+H=284.1634, calcd for $C_{18}H_{22}NO_2$ M+H=284.1651.

4.5.9. 4-(Benzyloxy)phenethyl 3-phenylpropanoate (6XI). Colorless solid; mp 61–62 °C; IR (KBr): 1725, 1240, 1170 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=2.60 (2H, t, J= 7.8 Hz), 2.83 (2H, t, J=7.0 Hz), 2.91 (2H, t, J=7.8 Hz), 4.23 (2H, t, J=7.0 Hz), 5.02 (2H, s), 6.89 (2H, d, J= 8.7 Hz), 7.08 (2H, d, J=8.7 Hz), 7.17 (3H, m), 7.28 (3H, m), 7.38 (4H, m); ^{13}C NMR ($CDCl_3$) δ=30.9 (s), 34.2 (s), 35.9 (s), 65.2 (s), 70.0 (s), 114.8 (t), 126.2 (t), 127.5 (t), 127.9 (t), 128.3 (t), 128.5 (t), 128.6 (t), 129.9 (t), 130.1 (q), 137.1 (q), 140.5 (q), 157.5 (q), 172.8 (q); HRMS (FAB): obsd M+ =360.1721, calcd for $C_{24}H_{24}O_3$ M+ =360.1725.

4.5.10. 2-(1′,7′,7′-Trimethyl-bicyclo[2,2,1] hept-2′-yloxy)ethyl 3-phenylpropanoate (6XII). Oil; IR (neat): 1735, 1165, 1120 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.80 (3H, s), 0.87 (3H, s), 0.96 (3H, s), 0.97 (2H, m), 1.45–1.75 (5H, m), 2.63 (2H, t, J=7.8 Hz), 2.95 (2H, t, J=7.8 Hz), 3.20 (1H, dd, J=7.5, 3.5 Hz), 3.47 (1H, m), 3.58 (1H, m), 4.17 (2H, m), 7.20 (3H, m), 7.28 (2H, m); ^{13}C NMR ($CDCl_3$) δ=11.7 (p), 20.1 (p), 20.2 (p), 27.3 (s), 30.9 (s), 34.4 (s), 35.9 (s), 38.5 (s), 45.0 (t), 46.4 (q), 49.2 (q), 63.8 (s), 66.9 (s), 87.4 (t), 126.2 (t), 128.3 (t), 128.5 (t), 140.6 (q), 172.8 (q); HRMS (FAB): obsd M+ =330.2214, calcd for $C_{21}H_{30}O_3$ M+ = 330.2195.

4.5.11. (*R*)-2-((3a*R*,5*R*,6*R*,6a*S*)-6-(Benzyloxy)-tetrahydro-2,2-dimethylfuro[2,3-*d*][1,3]dioxol-5-yl)-2-hydroxyethyl 3-phenylpropanoate (6XIII). Oil; IR (neat): 1735, 1215, 1165, 1070, 1025 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ= 1.30 (3H, s), 1.46 (3H, s), 2.46 (1H, broad), 2.65 (2H, t, J= 7.8 Hz), 2.94 (2H, t, J=7.8 Hz), 4.09 (2H, m), 4.14 (2H, m), 4.38 (1H, dd, J=14.4, 5.5 Hz), 4.55 (1H, d, J=11.9 Hz), 4.60 (1H, d, J=3.9 Hz), 4.69 (1H, d, J=11.9 Hz), 5.91 (1H, d, J=3.9 Hz), 7.18 (3H, t, J=3.9 Hz), 7.25 (2H, m), 7.33 (5H, m); ^{13}C NMR ($CDCl_3$) δ=26.2 (p), 26.7 (p), 30.8 (s), 35.6 (s), 66.5 (s), 67.4 (t), 72.0 (s), 79.3 (t), 81.6 (t), 82.0 (t), 105.1 (t), 111.7 (q), 126.2 (t), 127.7 (t), 128.1 (t), 128.4 (t), 128.6 (t), 137.1 (q), 140.2 (q), 173.0 (q); HRMS (FAB): obsd M+H=443.2065, calcd for $C_{25}H_{31}O_7$ M+H= 443.2070.

4.5.12. Octyl pentanoate (6II′). Oil; IR (neat): 1735, 1170 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.88 (3H, t, J=7.0 Hz), 0.92 (3H, t, J=7.4 Hz), 1.22–1.38 (12H, m), 1.61 (4H, m), 2.30 (2H, t, J=7.4 Hz), 4.06 (2H, t, J=6.7 Hz); ^{13}C NMR ($CDCl_3$) δ=13.7 (p), 14.0 (p), 22.3 (s), 22.6 (s), 25.9 (s),

27.1 (s), 28.6 (s), 29.2 (s), 31.8 (s), 34.1 (s), 64.4 (s), 174.0 (q); HRMS (EI): obsd M+ =214.1940, calcd for $C_{13}H_{26}O_2$ M+ =214.1933.

4.6. Typical procedure for oxidation of secondary alcohols to ketones

To a solution of (−)-menthol (1 mmol) in *t*-butyl alcohol (1 ml) were added I_2 (2 mmol) and K_2CO_3 (2 mmol) under an argon atmosphere. The mixture obtained was stirred at 90 °C. After 16 h at the same temperature, the mixture was quenched with satd aq Na_2SO_3 (3–5 ml) at 0 °C, and was extracted with Et_2O three times. The organic layer was washed with brine and dried over Na_2SO_4 to provide (−)-menthone in 99% yield in an almost pure state. If necessary, the product was purified by flash column chromatography on silica gel (hexane–EtOAc=4:1) to give pure (−)-menthone as an oil.[36] ^{1}H NMR ($CDCl_3$) δ=0.85 (3H, d, J= 6.8 Hz), 0.92 (3H, d, J=6.8 Hz), 1.01 (3H, d, J=6.1 Hz), 1.28–1.45 (2H, m), 1.79–2.20 (6H, m), 2.35 (1H, m).

4.6.1. 2,3,5-Tri-*O*-benzyl-D-ribofuranone. Oil; ^{1}H NMR ($CDCl_3$) δ=3.52 (1H, dd, J=11.1, 2.7 Hz), 3.62 (1H, dd, J=11.1, 2.7 Hz), 4.08 (1H, dd, J=5.6, 1.7 Hz), 4.41 (3H, m), 4.53 (2H, m), 4.67 (1H, d, J=11.9 Hz), 4.72 (1H, d, J= 11.9 Hz), 4.91 (1H, d, J=11.9 Hz), 7.18 (2H, m), 7.25–7.37 (13H, m); ^{13}C NMR ($CDCl_3$) δ=68.6 (s), 72.2 (s), 72.6 (s), 73.4 (s), 73.6 (t), 75.2 (t), 81.6 (t), 127.4 (t), 127.8 (t), 127.9 (t), 128.0 (t), 128.1 (t), 128.3 (t), 128.4 (t), 136.8 (q), 137.0 (q), 137.1 (q), 173.7 (q).

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