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Benzylic-acetoxylation of alkylbenzenes with $PhI(OAc)_2$ in the presence of catalytic amounts of $TsNH_2$ and I_2

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ABSTRACT

Treatment of alkylbenzenes with (diacetoxyiodo)benzene in the presence of catalytic amounts of *p*-toluenesulfonamide or *p*-nitrobenzenesulfonamide, and molecular iodine in 1,2-dichloroethane at 60 °C gave the corresponding (α-acetoxy)alkylbenzenes in good to moderate yields. The present reaction is a simple method for the introduction of an acetoxy group to the benzylic position of alkylbenzenes.

(Diacetoxyiodo)benzene (DIB) is one of the most useful hypervalent iodine reagents for organic synthesis because it can serve as an alternative to toxic heavy-metal reagents.[1] The advantages of DIB are as follows: it is a non-metal oxidant and it can be used for not only polar reactions, but also radical reactions to generate oxygen-centered radicals, nitrogen-centered radicals, and carbon-centered radicals.[2] For the radical reactions, a DIB-molecular iodine (I_2) system is used and the initial formation of acetyl hypoiodite (CH_3CO_2I) from the reaction of DIB and I_2 is the key step.[3] Synthetic studies of alkoxyl radicals derived from substrates, such as steroidal alcohols and sugars, with DIB and I_2 have been well carried out by Suarez et al.[2] We have also examined the synthetic utility of nitrogen-centered radicals for the construction of tetrahydroquinolines, benzosultams, and saccharins from sulfonamides with DIB and I_2 under irradiation with a tungsten lamp,[4] and oxygen-centered radicals for the construction of chromans from 3-arylpropanols with DIB and I_2 under irradiation with a tungsten lamp.[5] The formation of tetrahydroquinolines, benzosultams, and chromans proceeds through the intramolecular cyclization of the formed sulfonamidyl radicals and alkoxyl radicals onto their aromatic rings. Recently, the α-sulfonylamidation of alkylbenzenes with DIB, I_2, and *p*-toluenesulfonamide at 50 °C without solvent was reported to provide α-(*p*-toluenesulfonylamido)alkylbenzenes in moderate to good yields.[6] In those reactions, the reaction of ethylbenzene with DIB, I_2, and *p*-toluenesulfonamide in 1,2-dichloroethane gave a mixture of α-(*p*-toluenesulfonylamido)ethylbenzene and α-(acetoxy)ethylbenzene in 5% and 4% yields, respectively.[6] We have also reported the preparation of α-(*p*-toluenesulfonylamido)alkylbenzenes from alkylbenzenes with *p*-toluenesulfonamide and 1,3-diiodo-5,5-dimethylhydantoin (DIH), which works as synthon of a DIB and I_2 system.[7] Here, as part of our synthetic study of DIB for organic synthesis,[4,5] we would like to report the benzylic-acetoxylation of alkylbenzenes with DIB in the presence of catalytic amounts of *p*-toluenesulfonamide and I_2. To the best of our knowledge, the following methods for the practical preparation of α-(acetoxy)alkylbenzenes from alkylbenzenes are known, such as $K_2S_2O_8$ with transition metal salts in AcOH at 115 °C,[8a] $K_2S_2O_8$/LiBr (cat.)/AcONa in AcOH at 115 °C,[8b] CAN (cerium ammonium nitrate, cat.)/$KBrO_3$ in AcOH at 90–110 °C,[8c] NHPI (*N*-hydroxyphthalimide, cat.)/$Co(OAc)_2$ (cat.)/HNO_3 (cat.) under air in AcOH at 80 °C,[8d] DDQ (2,3-dichloro-5,6-dicyano-1,4-benzoquinone) in AcOH under ultrasonic irradiation,[8e] $NaIO_4$/LiBr(cat.) in AcOH at 90–110 °C,[8f] and related reactions.[8g,h] The $Pd(OAc)_2$-catalyzed α-acetoxylation of alkylbenzenes with DIB in AcOH at 100 °C, and the $Pd(OAc)_2$/CuI-catalyzed α-acetoxylation of alkylbenzenes with oxygen in AcOH were also reported.[9] However, there are several drawbacks such as toxicity and/or the high cost of some transition metals.

The present reaction was carried out as follows: ethylbenzene **1a**, DIB, I_2, and *p*-toluenesulfonamide were added to 1,2-dichloroethane under an argon atmosphere. The mixture was warmed at 60 °C for 2 h. After the reaction, the mixture was poured into satd aq sodium

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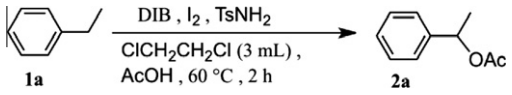
sulfite solution and extracted with diethyl ether. After removal of the ether solvent, the residue was subjected to preparative TLC on silica gel to give α-(acetoxy)ethylbenzene **2a** in 63% yield as shown in Table 1 (entry 5).[10] In the absence of either I_2 or *p*-toluenesulfonamide under the same conditions, α-(acetoxy)ethylbenzene was not formed at all (entries 8 and 11). This suggests that I_2 and *p*-toluenesulfonamide, respectively, work as catalysts in the present reaction. Instead of using DIB as the oxidant, [bis(trifluoroacetoxy)iodo]benzene was employed. However, [bis(trifluoroacetoxy)iodo]benzene did not work at all, whereas *p*-chloro[(diacetoxy)iodo]benzene and *p*-methyl[(diacetoxy)iodo]benzene worked to generate α-(acetoxy)ethylbenzene **2a** in moderate yields (entries 13–15). Oxone®, ^{t}BuOCl, and DIH (1,3-diiodo-5,5-dimethylhydantoin) also did not work at all as an oxidant, respectively (entries 16∼18). When acetonitrile and carbon tetrachloride instead of 1,2-dichloroethane as solvent were used, the yield of α-(acetoxy)ethylbenzene was decreased to ∼30% yield (entries 19 and 20). The same treatment of ethylbenzene with DIB, I_2, and *p*-toluenesulfonamide in 1,2-dichloroethane under irradiation conditions with a tungsten lamp provided α-(acetoxy)ethylbenzene in almost the same yield as that with warming conditions at 60 °C (entries 5 and 21). When [bis(benzoyloxy)iodo]benzene was used instead of DIB, α-(benzoyloxy)ethylbenzene **3a** was obtained in good yield (entry 22). These results indicate that the acetoxy group of α-(acetoxy)ethylbenzene **2a** comes from the acetoxy group of DIB. Based on these results, other

Table 1
α-Acetoxylation of ethylbenzene with a DIB·I_2·$TsNH_2$ system



1a **2a**

Entry	DIB (equiv)	I_2 (equiv)	$TsNH_2$ (equiv)	AcOH (mL)	Yield (%)
1[a]	2.0	0.2	0.2	1	25
2[b]	2.0	0.2	0.2	1	50
3[b]	1.5	0.2	0.2	1	42
4	2.0	0.2	0.2	0	57
5	2.5	0.2	0.2	0	63
6	3.0	0.2	0.2	0	59
7	2.5	0.1	0.2	0	47
8	2.5	0	0.2	0	0
9	2.5	0.2	0.3	0	61
10	2.5	0.2	0.1	0	43
11	2.5	0.2	0	0	0
12	2.5	0.5	0.5	0	24
13	2.5[c]	0.2	0.2	1	0
14	2.5[d]	0.2	0.2	0	56
15	2.5[e]	0.2	0.2	0	50
16	2.0[f]	0.2	0.2	1	0
17	2.0[g]	0.2	0.2	1	0
18	2.0[h]	0.2	0.2	1	3
19[i]	2.5	0.2	0.2	0	33
20[j]	2.5	0.2	0.2	0	29
21[k]	2.5	0.2	0.2	0	61
22	2.5[l]	0.2	0.2	0	67[m]

[a] The reaction time was 10 min.
[b] The reaction time was 1 h.
[c] Instead of DIB, [bis(trifluoroacetoxy)iodo]benzene was used.
[d] Instead of DIB, *p*-chloro(diacetoxyiodo)benzene was used.
[e] Instead of DIB, *p*-methyl(diacetoxyiodo)benzene was used.
[f] Instead of DIB, oxone was used.
[g] Instead of DIB, ^{t}BuOCl was used.
[h] Instead of DIB, DIH was used.
[i] CH_3CN was used as solvent.
[j] CCl_4 was used as solvent.
[k] Irradiated with a tungsten lamp.
[l] Instead of DIB, [bis(benzoyloxy)iodo]benzene was used.
[m] Yield of **3a**.

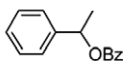
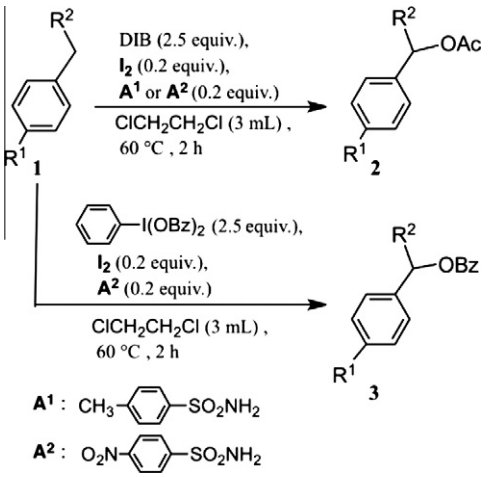
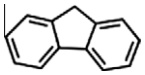
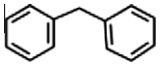
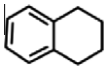
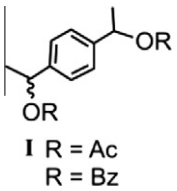
3a: 

Table 2
α-Acetoxylation of alkylbenzenes with a DIB·I_2·$ArSO_2NH_2$ system



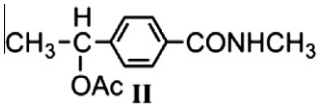
Entry	R^1	R^2	Yield[a] (%)		
			2		3
			A^1	A^2	A^1
1	H	CH_3	63	73	67
2	Br	CH_3	66	72	61
3	$C(CH_3)_3$	CH_3	66	68	58
4	C_2H_5	CH_3	38 (44)[b]	29 (51)[b]	43 (27)[b]
5	CO_2CH_3	CH_3	48	59	37
6	NO_2	CH_3	27	35	29
7	$CON(CH_3)_2$	CH_3	52 (25)[c]	28	35
8	H	CH_2CH_3	42	54	39
9	H	$(CH_2)_2CH_3$	48	60	57
10	H	$(CH_2)_2OAc$	27	31	45
11	H	$(CH_2)_2Br$	36	42	30
12	H	$(CH_2)_6CH_3$	52	57	48
13	Ph	$(CH_2)_3CH_3$	64	81	61
14			61 (9)[d]	48 (13)[d]	51 (11)[e]
15			61	37	51
16			55 (1:0.7)[f]	63 (1:0.7)[f]	58 (1:0.8)[g]

[a] Isolated yield.
[b] Yield of compound **I**.



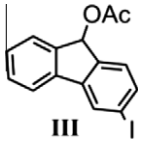
I R = Ac
R = Bz

[c] Yield of compound **II**.



II

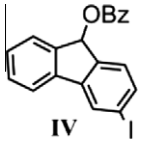
[d] Yield of compound **III**.



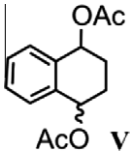
III

Table 2 (*continued*)

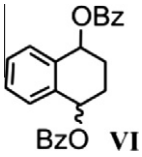
[e] Yield of compound **IV**.



[f] Yield of compound **V**.



[g] Yield of compound **VI**.

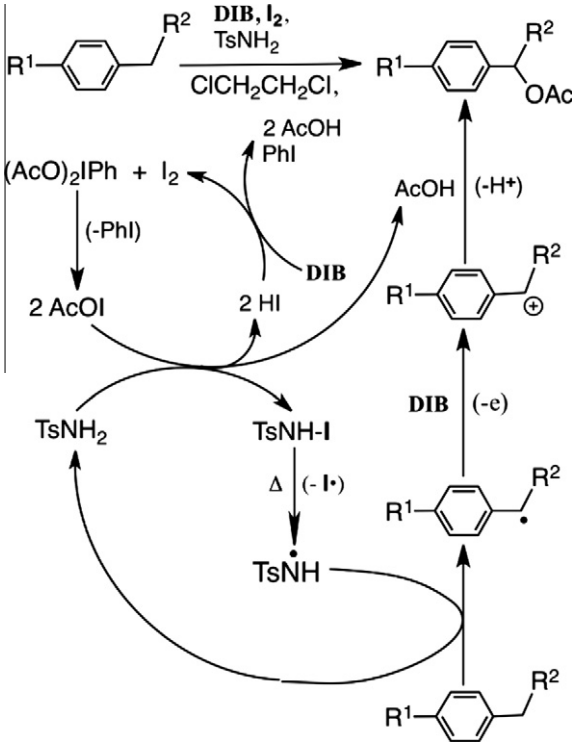


alkylbenzenes, such as 4-bromo-1-ethylbenzene, 4-*t*-butyl-1-ethylbenzene, methyl 4-ethylbenzoate, 4-ethyl-*N*,*N*-dimethylbenzamide, *n*-propylbenzene, *n*-butylbenzene, *n*-octylbenzene, and 4-pentyl-1-biphenyl were treated with DIB, I_2, and *p*-toluenesulfonamide at 60 °C to provide the corresponding α-(acetoxy)alkylbenzenes in good to moderate yields as shown in Table 2 (entries 2, 3, 5, 7–9, 12, and 13). When 1,4-diethylbenzene was used 1-α-(acetoxyethyl)-4-ethylbenzene was obtained in 38% yield together with 1,4-di(α-acetoxy)ethylbenzene in 44% yield (entry 4). The yields of α-acetoxy compounds were low when *p*-nitroethylbenzene was used (entry 6). Treatment of 4-ethyl-*N*,*N*-dimethylbenzamide with DIB in the presence of I_2 and *p*-toluenesulfonamide generated 4-(α-acetoxy)ethyl-*N*,*N*-dimethylbenzamide in 52% yield together with 4-(α-acetoxy)ethyl-*N*-methylbenzamide in 25% yield, which was an N-demethylated compound (entry 7). The same treatment of fluorene and diphenylmethane with DIB, I_2, and *p*-toluenesulfonamide provided the corresponding α-acetoxy compounds in good to moderate yields (entries 14 and 15). When 1,2,3,4-tetrahydronaphthalene was used, a mixture of *cis*- and *trans*-1,4-diacetoxy-1,2,3,4-tetrahydronaphthalenes was obtained in 55% yield. Moreover, when *p*-nitrobenzenesulfonamide ($\mathbf{A^2}$), which may have stronger N–H bond energy than that of *p*-toluenesulfonamide due to the presence of an electron-withdrawing group, was used instead of *p*-toluenesulfonamide ($\mathbf{A^1}$), the yield of α-acetoxylated compounds was slightly increased (from column $\mathbf{A^1}$ to column $\mathbf{A^2}$). The same treatment of alkylbenzenes with [bis(benzoyloxy)iodo]benzene instead of DIB, generated the corresponding α-benzoyloxy compounds **3**. However, there is not so much difference in yield between DIB and [bis(benzoyloxy)iodo]benzene.

A plausible reaction mechanism is shown in Scheme 1. The initial formation of *N*-iodo-*p*-toluenesulfonamide from the reaction of *p*-toluenesulfonamide with DIB in the presence of I_2 occurs, and this is followed by homolytic N–I bond cleavage to generate a sulfonamidyl radical.

The sulfonamidyl radical abstracts a benzylic hydrogen atom from alkylbenzene to provide a benzyl radical that may be oxidized to benzylic cation with DIB. Once a benzylic cation is formed, it smoothly reacts with acetic acid to give α-(acetoxy)alkylbenzene. Practically, the addition of galvinoxyl free radical completely retarded the present reaction and ethylbenzene was obtained with high recovery under the same conditions.

In conclusion, treatment of alkylbenzenes with DIB in the presence of catalytic amounts of I_2 and *p*-toluenesulfonamide at 60 °C gave the corresponding (α-acetoxy)alkylbenzenes in good to moderate yields. The present reaction is a simple method for the α-acetoxylation at the benzylic position of alkylbenzenes. Further synthetic studies using the present reaction are under way in this laboratory.



Scheme 1. Plausible reaction mechanism.

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10. *Typical procedure for preparation of α-(acetoxy)ethylbenzene with ethylbenzene, DIB, I_2, and p-toluenesulfonamide*: (Diacetoxyiodo)benzene (5 mmol, 1.61 g), I_2 (0.4 mmol, 102 mg), *p*-toluenesulfonamide (0.4 mmol, 68.4 mg), and ethylbenzene (2 mmol, 184 mg) were added to dichloroethane (3 mL). The mixture was warmed at 60 °C for 2 h under an argon atmosphere. Then, the mixture was poured into saturated aqueous sodium sulfite solution and extracted with diethyl ether (3 × 20 mL). The organic layer was dried over Na_2SO_4. After filtration and removal of the solvent under reduced pressure, the residue was subjected to preparative TLC on silica gel using a mixture of hexane and ethyl acetate (5:1) as an eluent to give α-(acetoxy)ethylbenzene in 63% yield.

(α-Acetoxy)ethylbenzene ($C_{10}H_{12}O_2$): IR (Neat): 1065, 1241, 1743 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.53 (d, J = 6.6 Hz, 3H), 2.07 (s, 3H), 5.88 (q, J = 6.6 Hz, 1H), 7.26–7.36 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS) δ = 21.3, 22.1, 72.2, 126.0, 128.0, 128.4, 141.6, 170.2; HRMS (ESI) calcd for $C_{10}H_{12}O_2Na$ [M+Na]$^+$: m/z 187.0730, found m/z 187.0728.

(α-Benzoyloxy)ethylbenzene ($C_{15}H_{14}O_2$): IR (Neat): 1109, 1267, 1713 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.67 (d, J = 6.6 Hz, 3H), 6.13 (q, J = 6.6 Hz, 1H), 7.29 (t, J = 7.3 Hz, 1H), 7.36 (t, J = 7.5 Hz, 2H), 7.41–7.44 (m, 4H), 7.55 (t, J = 7.5 Hz, 1H), 8.07 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 22.4, 72.9, 126.0, 127.8, 128.3, 128.5, 129.6, 130.5, 132.9, 141.7, 165.7; HRMS (ESI) calcd for $C_{15}H_{14}O_2Na$ [M+Na]$^+$: m/z 249.0886, found m/z 249.0882.

4-(α-Acetoxy)ethyl-1-bromobenzene ($C_{10}H_{11}O_2Br$): IR (Neat): 599, 1071, 1241, 1735 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.51 (d, J = 6.7 Hz, 3H), 2.06 (s, 3H), 5.82 (q, J = 6.7 Hz, 1H), 7.22 (d, J = 8.3 Hz, 2H), 7.47 (d, J = 8.3 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 21.2, 22.1, 71.6, 121.7, 127.8, 131.6, 140.7, 170.1; HRMS(APPI) calcd for $C_{10}H_{10}O_2Br$ [M−H]$^+$: m/z 240.9859, found m/z 240.9854.

4-(α-Benzoyloxy)ethyl-1-bromobenzene ($C_{15}H_{13}O_2Br$): IR (Neat): 711, 1109, 1270, 1718 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.65 (d, J = 6.6 Hz, 3H), 6.07 (q, J = 6.6 Hz, 1H), 7.32 (d, J = 8.5 Hz, 2H), 7.44 (t, J = 7.5 Hz, 2H), 7.48 (d, J = 8.5 Hz, 2H), 7.56 (t, J = 7.5 Hz, 1H), 8.06 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 22.2, 72.2, 121.7, 127.7, 128.3, 129.6, 130.2, 131.6, 133.0, 140.8, 165.6; HRMS(APPI) calcd for $C_{15}H_{12}O_2Br$ [M−H]$^+$: m/z 303.0015, found m/z 303.0010.

1-(α-Acetoxy)ethyl-4-*t*-butylbenzene ($C_{14}H_{20}O_2$): IR (Neat): 1064, 1242, 1743 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.33 (s, 9H), 1.53 (d, J = 6.6 Hz, 3H), 2.07 (s, 3H), 5.88 (q, J = 6.6 Hz, 1H), 7.28 (d, J = 8.6 Hz, 2H), 7.37 (d, J = 8.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 21.3, 21.9, 31.3, 34.4, 72.1, 125.3, 125.8, 138.5, 150.7, 170.3; HRMS (ESI) calcd for $C_{14}H_{20}O_2Na$ [M+Na]$^+$: m/z 243.1356, found m/z 243.1353.

1-(α-Benzoyloxy)ethyl-4-*t*-butylbenzene ($C_{19}H_{22}O_2$): IR (Neat): 1110, 1270, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.31 (s, 9H), 1.66 (d, J = 6.6 Hz, 3H), 6.13 (q, J = 6.6 Hz, 1H), 7.36–7.39 (m, 4H), 7.42 (t, J = 7.4 Hz, 2H), 7.54 (t, J = 7.4 Hz, 1H), 8.07 (d, J = 7.4 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 22.2, 31.3, 34.5, 72.7, 125.4, 125.8, 128.3, 129.6, 130.6, 132.8, 138.6, 150.8, 165.8; HRMS (ESI) calcd for $C_{19}H_{22}O_2Na$ [M+Na]$^+$: m/z 305.1512, found m/z 305.1509.

1-(α-Acetoxy)ethyl-4-ethylbenzene ($C_{12}H_{16}O_2$): IR (Neat): 1064, 1240, 1738 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.23 (t, J = 7.8 Hz, 3H), 1.53 (d, J = 6.6 Hz, 3H), 2.06 (s, 3H), 2.64 (q, J = 7.8 Hz, 2H), 5.86 (q, J = 6.6 Hz, 1H), 7.18 (d, J = 7.9 Hz, 2H), 7.27 (d, J = 7.9 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 15.4, 21.3, 22.0, 28.5, 72.1, 126.1, 127.9, 138.8, 143.9, 170.3; HRMS (ESI) calcd for $C_{12}H_{16}O_2Na$ [M+Na]$^+$: m/z 215.1043, found m/z 215.1040.

1-(α-Benzoyloxy)ethyl-4-ethylbenzene ($C_{17}H_{18}O_2$): IR (Neat): 1113, 1270, 1715 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.24 (t, J = 7.7 Hz, 3H), 1.66 (d, J = 6.6 Hz, 3H), 2.64 (q, J = 7.7 Hz, 2H), 6.11 (q, J = 6.6 Hz, 1H), 7.20 (d, J = 8.1 Hz, 2H), 7.37 (d, J = 8.1 Hz, 2H), 7.42 (t, J = 7.5 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 8.07 (d, J = 7.5 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 15.4, 22.2, 28.5, 72.8, 126.1, 127.9, 128.2, 129.6, 130.5, 132.8, 138.9, 143.9, 165.7; HRMS (ESI) calcd for $C_{17}H_{18}O_2Na$ [M+Na]$^+$: m/z 277.1199, found m/z 277.1196.

1,4-Bis(α-acetoxyethyl)benzene ($C_{14}H_{18}O_4$): IR (Neat): 1065, 1240, 1739 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.52 (d, J = 6.3 Hz, 6H), 2.07 (s, 6H), 5.87 (q, J = 6.3 Hz, 2H), 7.33(s, 4 H); ^{13}C NMR ($CDCl_3$, TMS) δ = 21.3, 22.1, 72.0, 126.3, 141.3, 170.3; HRMS (ESI) calcd for $C_{14}H_{18}O_4Na$ [M+Na]$^+$: m/z 273.1097, found m/z 273.1093.

1,4-Bis(α-benzoyloxyethyl)benzene ($C_{24}H_{22}O_4$): IR (Neat): 1065, 1268, 1713 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.66 (d, J = 6.6 Hz, 6H), 6.13 (q, J = 6.6 Hz, 2H), 7.43 (t, J = 7.3 Hz, 4H), 7.45 (s, 4H), 7.55 (t, J = 7.3 Hz, 2H), 8.07 (d, J = 7.3 Hz, 4H); ^{13}C NMR($CDCl_3$, TMS) δ = 22.3, 72.6, 126.3, 128.3, 129.6, 130.4, 132.9, 141.4, 165.8; HRMS (ESI) calcd for $C_{24}H_{22}O_4Na$ [M+Na]$^+$:m/z 397.1410, found m/z 397.1404.

Methyl 4-(α-acetoxy)ethylbenzoate ($C_{12}H_{14}O_4$): IR (Neat): 1067, 1236, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.53 (d, J = 6.6 Hz, 3H), 2.09 (s, 3H), 3.91 (s, 3H), 5.90 (q, J = 6.6 Hz, 1H), 7.41 (d, J = 8.2 Hz, 2H), 8.02 (d, J = 8.2 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 21.1, 22.1, 52.0, 71.7, 125.8, 129.5, 129.8, 146.7, 166.6, 170.1; HRMS (ESI) calcd for $C_{12}H_{14}O_4Na$ [M+Na]$^+$: m/z 245.0784, found m/z 245.0781.

Methyl 4-(α-benzoyloxy)ethylbenzoate ($C_{17}H_{16}O_4$): IR (Neat): 1110, 1263, 1713 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.68 (d, J = 6.5 Hz, 3H), 3.91 (s, 3H), 6.15 (q, J = 6.5 Hz, 1H), 7.45 (t, J = 7.5 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 8.04 (d, J = 8.4 Hz, 2H), 8.08 (d, J = 7.5 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 22.3, 52.0, 72.3, 125.8, 128.3, 129.6, 129.9, 130.0, 130.1, 133.0, 146.8, 165.6, 166.6; HRMS (ESI) calcd for $C_{17}H_{16}O_4Na$ [M+Na]$^+$: m/z 307.0941, found m/z 307.0934.

4-(α-Acetoxy)ethyl-1-nitrobenzene ($C_{10}H_{11}O_4N$): IR (Neat): 1069, 1238, 1348, 1524, 1739 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.55 (d, J = 6.7 Hz, 3H), 2.11 (s, 3H), 5.92 (q, J = 6.7 Hz, 1H), 7.51 (d, J = 8.7 Hz, 2H), 8.22 (d, J = 8.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 21.1, 22.2, 71.2, 123.8, 126.7, 147.4, 149.0, 170.0; HRMS(APPI) calcd for $C_{10}H_{10}O_4N$ [M−H]$^+$: m/z 208.0604, found m/z 208.0604.

4-(α-Benzoyloxy)ethyl-1-nitrobenzene ($C_{15}H_{13}O_4N$):IR (Neat): 1069, 1270, 1348, 1522, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.70 (d, J = 6.6 Hz, 3H), 6.18 (q, J = 6.6 Hz, 1H), 7.48 (t, J = 7.0 Hz, 2H), 7.56 (t, J = 7.0 Hz, 1H), 7.61 (d, J = 8.8 Hz, 2H), 8.08 (d, J = 7.0 Hz, 2H), 8.23 (d, J = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 22.4, 71.8, 123.9, 126.7, 128.5, 129.7, 129.8, 133.3, 147.5, 149.1, 165.6; HRMS(APPI) calcd for $C_{15}H_{12}O_4N$ [M−H]$^+$: m/z 270.0761, found m/z 270.0754.

4-(α-Acetoxy)ethyl-*N*,*N*-dimethylbenzamide ($C_{13}H_{17}O_3N$): IR (Neat): 1065, 1241, 1633, 1733, 3354, 3460 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.53 (d, J = 6.6 Hz, 3H), 2.09 (s, 3H), 3.05 (br, 6H), 5.89 (q, J = 6.6 Hz, 1H), 7.39 (q, J = 8.5 Hz, 4H); ^{13}C NMR($CDCl_3$, TMS) δ = 21.3, 22.2, 35.3, 39.6, 71.8, 126.0, 127.3, 135.8, 143.1, 170.2, 171.2; HRMS (ESI) calcd for $C_{13}H_{17}O_3NNa$ [M+Na]$^+$: m/z 258.1101, found m/z 258.1095.

4-(α-Benzoyloxy)ethyl-*N*,*N*-dimethylbenzamide ($C_{18}H_{19}O_3N$): IR (Neat): 1112, 1270, 1633, 1715, 3471 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.67 (d, J = 6.6 Hz, 3H), 3.04 (br, 6H), 6.14 (q, J = 6.6 Hz, 1H), 7.41 (t, J = 7.1 Hz, 2H), 7.44 (t, J = 7.4 Hz, 4H), 7.56 (t, J = 7.1 Hz, 1H), 8.08 (d, J = 7.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 22.4, 35.3, 39.6, 72.4, 126.0, 127.3, 128.4, 129.6, 130.3, 135.8, 143.2, 165.7, 171.2; HRMS (ESI) calcd for $C_{18}H_{19}O_3NNa$ [M+Na]$^+$: m/z 320.1257, found m/z 320.1261.

(α-Acetoxy)propylbenzene ($C_{11}H_{14}O_2$): IR (Neat): 1072, 1236, 1736 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.87 (t, J = 7.4 Hz, 3H), 1.74–1.96 (m, 2H), 2.07 (s, 3H), 5.66 (t, J = 6.9 Hz, 1H), 7.27–7.36 (m, 5H); ^{13}C NMR($CDCl_3$, TMS) δ = 9.8, 21.2, 29.2, 77.3, 126.5, 127.8, 128.3, 140.5, 170.4; HRMS (ESI) calcd for $C_{11}H_{14}O_2Na$ [M+Na]$^+$: m/z 201.0886, found m/z 201.0887.

(α-Benzoyloxy)propylbenzene ($C_{16}H_{16}O_2$): IR (Neat): 1111, 1271, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.97 (t, J = 7.4 Hz, 3H), 1.91–2.01 (m, 1H), 2.01–2.12 (m, 1H), 5.92 (t, J = 6.9 Hz, 1H), 7.28 (t, J = 7.3 Hz, 1H), 7.34 (t, J = 7.3 Hz, 2H), 7.42 (d, J = 7.3 Hz, 2H), 7.44 (t, J = 7.4 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 8.09 (d, J = 7.4 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 10.0, 29.5, 77.9, 126.4, 127.8, 128.3, 128.4, 129.6, 130.5, 132.9, 140.6, 165.9; HRMS (ESI) calcd for $C_{16}H_{16}O_2Na$ [M+Na]$^+$: m/z 263.1043, found m/z 263.1046.

(α-Acetoxy)butylbenzene ($C_{12}H_{16}O_2$): IR (Neat): 1025, 1237, 1736 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.92 (t, J = 7.4 Hz, 3H), 1.21–1.41 (m, 2H), 1.70–1.80 (m, 1H), 1.86–1.93 (m, 1H), 2.07 (s, 3H), 5.74 (t, J = 6.3 Hz, 1H), 7.25–7.36 (m, 5H); ^{13}C NMR($CDCl_3$, TMS) δ = 13.8, 18.8, 21.3, 38.4, 75.9, 126.5, 127.8, 128.4, 140.8, 170.4; HRMS (ESI) calcd for $C_{12}H_{16}O_2Na$ [M+Na]$^+$: m/z 215.1043, found m/z 215.1041.

(α-Benzoyloxy)butylbenzene ($C_{17}H_{18}O_2$): IR (Neat): 1111, 1272, 1718 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.95 (t, J = 7.4 Hz, 3H), 1.31–1.51 (m, 2H), 1.83–1.92 (m, 1H), 2.02–2.11 (m, 1H), 5.99 (t, J = 6.1 Hz, 1H), 7.28 (t, J = 7.3 Hz, 1H), 7.35 (t, J = 7.3 Hz, 2H), 7.42 (d, J = 7.3 Hz, 2H), 7.44 (t, J = 7.5 Hz, 2H), 7.58 (t, J = 7.5 Hz, 1H), 8.08 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS) δ = 13.8, 18.8, 38.7, 76.5, 126.4, 127.8, 128.3, 128.4, 129.6, 130.5, 132.9, 140.9, 165.8; HRMS (ESI) calcd for $C_{17}H_{18}O_2Na$ [M+Na]$^+$: m/z 277.1199, found m/z 277.1202.

1,3-Diacetoxy-1-phenylpropane ($C_{13}H_{16}O_4$): IR (Neat): 1043, 1236, 1743 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.04 (s, 3H), 2.06 (s, 3H), 2.06–2.15 (m, 1H), 2.21–2.30 (m, 1H), 3.98–4.04 (m, 1H), 4.10–4.19 (m, 1H), 5.85 (dd, J = 8.3 and 5.7 Hz, 1H), 7.27–7.36 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS) δ = 20.7, 21.0, 35.1, 60.5, 72.7, 126.3, 128.0, 128.5, 139.8, 170.0, 170.1; HRMS (ESI) calcd for $C_{13}H_{16}O_4Na$ [M+Na]$^+$: m/z 259.0941, found m/z 259.0938.

3-Acetoxy-1-benzoyloxy-1-phenylpropane ($C_{18}H_{18}O_4$): IR (Neat): 1111, 1272, 1720 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.00 (s, 3H), 2.20–2.30 (m, 1H), 2.38–2.47 (m, 1H), 4.09–4.16 (m, 1H), 4.19–4.28 (m, 1H), 6.12 (dd, J = 8.1 and 5.4 Hz, 1H), 7.31 (t, J = 7.2 Hz, 1H), 7.36 (t, J = 7.2 Hz, 2H), 7.40–7.48 (m, 4H), 7.60 (t, J = 7.3 Hz, 1H), 8.07 (d, J = 7.3 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 20.9, 35.4, 60.8, 73.5, 126.3, 128.2, 128.4, 128.6, 129.6, 130.1, 133.1, 139.9, 165.6, 171.0; HRMS (ESI) calcd for $C_{18}H_{18}O_4Na$ $[M+Na]^+$:m/z 321.1097, found m/z 321.1092.

1-Acetoxy-4-bromo-1-phenylbutane ($C_{12}H_{15}O_2Br$): IR (Neat): 1026, 1235, 1735 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.77–1.86 (m, 1H), 1.86–2.01 (m, 2H), 2.01–2.08 (m, 1H), 2.09 (s, 3H), 3.40 (t, J = 6.5 Hz, 2H), 5.76 (dd, J = 7.6 and 5.6 Hz, 1H), 7.28–7.39 (m, 5H); ^{13}C NMR($CDCl_3$, TMS) δ = 21.2, 28.7, 33.1, 34.8, 75.0, 126.4, 128.1, 128.5, 140.1, 170.3; HRMS(APPI) calcd for $C_{12}H_{14}O_2Br$ $[M-H]^+$: m/z 269.0172, found m/z 269.0196.

1-Benzoyloxy-4-bromo-1-phenylbutane ($C_{17}H_{17}O_2Br$): IR (Neat): 1109, 1271, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.87–2.29 (m, 4H), 3.43 (t, J = 6.6 Hz, 2H), 6.03 (dd, J = 7.8 and 5.5 Hz, 1H), 7.30 (t, J = 7.0 Hz, 1H), 7.36 (t, J = 7.0 Hz, 2H), 7.42 (d, J = 7.0 Hz, 2H), 7.45 (t, J = 7.6 Hz, 2H), 7.57 (t, J = 7.6 Hz, 1H), 8.08 (d, J = 7.6 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 28.7, 33.2, 35.1, 75.6, 126.3, 128.1, 128.4, 128.6, 129.6, 130.2, 133.1, 140.2, 165.7; HRMS(APPI) calcd for $C_{17}H_{16}O_2Br$ $[M-H]^+$: m/z 331.0328, found m/z 331.0351.

(α-Acetoxy)octylbenzene ($C_{16}H_{24}O_2$): IR (Neat): 1022, 1237, 1738 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.86 (t, J = 7.0 Hz, 3H), 1.15–1.38 (m, 10H), 1.71–1.79 (m, 1H), 1.84–1.94 (m, 1H), 2.06 (s, 3H), 5.72 (t, J = 6.5 Hz, 1H), 7.26–7.35 (m, 5H); ^{13}C NMR($CDCl_3$, TMS) δ = 14.0, 21.3, 22.6, 25.5, 29.1, 29.3, 31.7, 36.3, 76.1, 126.5, 127.8, 128.4, 140.8, 170.4; HRMS (ESI) calcd for $C_{16}H_{24}O_2Na$ $[M+Na]^+$: m/z 271.1669, found m/z 271.1666.

(α-Benzoyloxy)octylbenzene ($C_{21}H_{26}O_2$): IR (Neat): 1109, 1271, 1720 cm^{-1};^1H NMR ($CDCl_3$, TMS) δ = 0.86 (t, J = 6.9 Hz, 3H), 1.17–1.46 (m, 10H), 1.86–1.92 (m, 1H), 2.01–2.10 (m, 1H), 5.97 (t, J = 6.9 Hz, 1H), 7.29 (t, J = 7.2 Hz, 1H), 7.35 (t, J = 7.2 Hz, 2H), 7.42 (d, J = 7.2 Hz, 2H), 7.45 (t, J = 7.3 Hz, 2H), 7.56 (t, J = 7.3 Hz, 1H), 8.08 (d, J = 7.3 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 14.1, 22.6, 25.5, 29.1, 29.3, 31.8, 36.5, 76.7, 126.4, 127.8, 128.3, 128.4, 129.6, 130.5, 132.9, 140.9, 165.9; HRMS (ESI) calcd for $C_{21}H_{26}O_2Na$ $[M+Na]^+$: m/z 333.1825, found m/z 333.1819.

4-(α-Acetoxy)pentylbiphenyl ($C_{19}H_{22}O_2$): IR (Neat): 1108, 1271, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.89 (t, J = 7.1 Hz, 3H), 1.20–1.30 (m, 1H), 1.30–1.39 (m, 3H), 1.76–1.84 (m, 1H), 1.90–1.98 (m, 1H), 2.09 (s, 3H), 5.76 (t, J = 7.0 Hz, 1H), 7.35 (t, J = 7.5 Hz, 1H), 7.40 (d, J = 7.5 Hz, 2H), 7.44 (t, J = 7.5 Hz, 2H), 7.58 (t, J = 7.9 Hz, 4H); ^{13}C NMR($CDCl_3$, TMS) δ = 13.9, 21.3, 22.4, 27.7, 36.0, 75.9, 127.0, 127.1, 127.2, 127.3, 128.7, 137.8, 139.8, 140.7, 170.4; HRMS (ESI) calcd for $C_{19}H_{22}O_2Na$ $[M+Na]^+$: m/z 305.1512, found m/z 305.1510.

4-(α-Benzoyloxy)pentylbiphenyl ($C_{24}H_{24}O_2$): IR (Neat): 1108, 1271, 1719 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.91 (t, J = 7.1 Hz, 3H), 1.30–1.48 (m, 4H), 1.90–1.99 (m, 1H), 2.06–2.15 (m, 1H), 6.02 (t, J = 6.1 Hz, 1H), 7.34 (t, J = 7.3 Hz, 1H), 7.40–7.51 (m, 6H), 7.54–7.60 (m, 5H), 8.10 (d, J = 7.3 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 14.0, 22.5, 27.7, 36.3, 76.5, 126.9, 127.0, 127.1, 127.2, 127.3, 128.3, 128.7, 129.6, 130.5, 132.9, 140.0, 140.8, 165.9; HRMS (ESI) calcd for $C_{24}H_{24}O_2Na$ $[M+Na]^+$: m/z 367.1669, found m/z 367.1667.

9-Acetoxy-9*H*-fluorene ($C_{15}H_{12}O_2$): IR (Neat): 1024, 1234, 1737 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.19 (s, 3H), 6.80 (s, 1H), 7.30 (t, J = 7.6 Hz, 2H), 7.41 (t, J = 7.6 Hz, 2H), 7.55 (d, J = 7.6 Hz, 2H), 7.67 (d, J = 7.6 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 21.2, 75.1, 120.1, 125.8, 127.8, 129.4, 141.0, 142.0, 171.7; HRMS (ESI) calcd for $C_{15}H_{12}O_2Na$ $[M+Na]^+$: m/z 240.0730, found m/z 247.0733.

9-Benzoyloxy-9*H*-fluorene ($C_{20}H_{14}O_2$): IR (Neat): 1262, 1451, 1724 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 7.05 (s, 1H), 7.31 (t, J = 7.8 Hz, 2H), 7.41–7.47 (m, 4H), 7.57 (t, J = 7.4 Hz, 1H), 7.63 (d, J = 7.8 Hz, 2H), 7.71 (d, J = 7.8 Hz, 2H), 8.09 (d, J = 7.4 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 75.7, 120.2, 126.2, 128.0, 128.5, 128.6, 129.6, 130.1, 133.3, 141.2, 142.3, 167.4; HRMS (ESI) calcd for $C_{20}H_{14}O_2Na$ $[M+Na]^+$: m/z 309.0886, found m/z 309.0888.

Diphenylmethyl acatate ($C_{15}H_{14}O_2$): IR (Neat): 1023, 1232, 1741 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.16 (s, 3H), 6.88 (s, 1H), 7.26–7.36 (m, 10H); ^{13}C NMR($CDCl_3$, TMS) δ = 21.3, 76.8, 127.0, 127.9, 128.5, 140.2, 170.0; HRMS (ESI) calcd for $C_{15}H_{14}O_2Na$ $[M+Na]^+$:m/z 249.0886, found m/z 249.0886.

Diphenylmethyl benzoate ($C_{20}H_{16}O_2$): IR (Neat): 1107, 1265, 1712 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 7.12 (s, 1H), 7.29 (t, J = 7.3 Hz, 2H), 7.32–7.38 (m, 4H), 7.42–7.47 (m, 6 H), 7.57 (t, J = 7.3 Hz, 1H), 8.14 (d, J = 7.3 Hz, 2H); ^{13}C NMR($CDCl_3$, TMS) δ = 77.3, 127.0, 127.8, 128.3, 128.4, 129.6, 129.9, 133.0, 140.2, 165.4; HRMS (ESI) calcd for $C_{20}H_{16}O_2Na$ $[M+Na]^+$: m/z 311.1043, found m/z 311.1041.