Hypervalent Iodine Catalyzed Cyclization of Aryl-Substituted Alkanoic Acids

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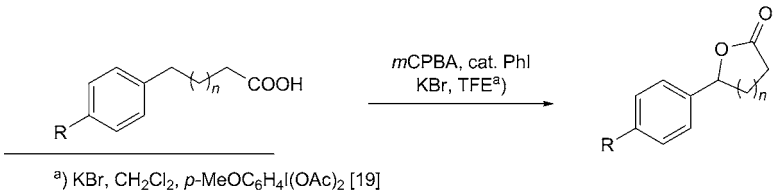
A novel and efficient procedure was developed for direct preparation of aryl-substituted lactones from corresponding aryl-substituted alkanoic acids, catalyzed by the *in situ* generated hypervalent iodine intermediate from iodobenzene (PhI). In this protocol, aryl-substituted alkanoic acids were treated with *m*-chloroperbenzoic acid (*m*CPBA) and KBr in the presence of a catalytic amount of PhI in 2,2,2-trifluoroethanol at room temperature for 24 h, resulting in corresponding aryl lactones in moderate-to-good yields.

Introduction. – The chemistry of hypervalent iodine organic compounds has experienced impressive developments since the early 1990s. Due to their low toxicity, ready availability, easy handling, and reactivity similar to that of heavy-metal reagents or anodic oxidation, they have been found broad application in organic chemistry and frequently used in synthesis [1–9]. They are usually used as stoichiometric oxidants in synthesis and after the oxidations, at least equimolar amounts of iodoarenes are produced as by-products, most of them are disposed of and are not well utilized, restricting further applications. In 2005, *Ochiai et al.* and *Kita et al.* independently reported the first hypervalent iodine-catalyzed oxidative coupling reactions [10][11]. Since then, the catalytic utilization of hypervalent iodine reagents has been increasing in importance, with growing interest in the development of environmentally benign synthetic transformations [12–18]. In these catalyzed reactions, a catalytic amount of an I-containing molecule together with a stoichiometric oxidant is used. The oxidant generates the hypervalent iodine reagent *in situ* and, after the oxidative transformation, the reduced I-containing molecule is re-oxidized.

Recently, *Kita* and co-workers developed a new and metal-free oxidative cyclization for direct formation of aryl lactones from aryl-substituted alkanoic acids, in which a stoichiometric hypervalent iodine reagent, 4-methoxyphenyliodine(III) diacetate (4-$MeOC_6H_4I(OAc)_2$) was used as oxidant, and a benzyl radical formed *via* sp^3 C–H abstraction mechanism was hypothesized [19]. However, the same reaction using a catalytic amount of iodobenzene (PhI) in place of a stoichiometric hypervalent iodine reagent, which is more environmentally benign has not been reported before. Although *Ishihara* and co-workers reported a similar cyclization of keto carboxylic acids to keto lactones catalyzed by hypervalent iodine intermediate in 2009 [20], the protocol is different from the method of *Kita et al.* in substrates and mechanism.

To develop a novel catalytic cyclization for direct preparation of aryl lactones from aryl-substituted alkanoic acids and to extend the scope of catalytic use of hypervalent

Scheme 1. *Catalytic Cyclization of Aryl-Substituted Alkanoic Acid-Catalyzed by Hypervalent Iodine(III)*



iodine reagents for organic synthesis, we first selected a simple protocol to investigate the oxidation of benzylic C–H bonds to the corresponding ketones *via* the benzyl radical intermediate, and found that two systems of PhI/*m*CPBA/KBr and PhI/*m*CPBA/*t*-BuOOH can improve this procedure [21][22]. Based on the success, we have investigated the novel catalytic cyclization of aryl-subsituted alkanoic acids in the presence of a catalytic amount of PhI, and obtained indeed the desired aryl lactones (*Scheme 1*).

Results and Discussion. – First, we investigated the reaction of 4-phenylbutanoic acid with *m*CPBA and KBr in the presence of a catalytic amount of PhI. Generally, the reaction was carried out in organic solvents at room temperature for 12 h, and the desired product, γ-phenyl-γ-lactone, was obtained (*Table 1*).

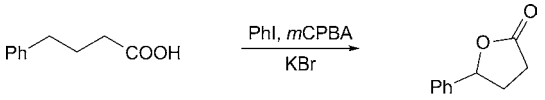
In light of successful formation of the phenyl lactone, the reaction conditions were optimized, and the results are compiled in *Table 1*. It was found that the solvent influenced the reaction distinctly: when 1.0 equiv. of 4-phenylbutanoic acid was mixed with 1.0 equiv. of KBr, 1.2 equiv. of *m*CPBA, and 0.2 equiv. of PhI in CH_2Cl_2 or 2,2,2-trifluoroethanol (TFE) at room temperature and stirred for 12 h, the reaction gave moderate yields; other solvents led to rather poor yields (*Entries 1–8*). Several oxidants were tested under the same conditions, and *m*CPBA turned out to be the most effective one in TFE (*Entries 8–11*). The amount of PhI was checked, and 0.2 equiv. was the best choice (*Entries 8* and *13–15*). However, in the absence of PhI, no desired lactone was observed (*Entry 12*). We examined the amount of KBr, and found that more KBr promoted the reaction, and finally 5.0 equiv. of KBr was selected for the reaction (*Entries 8* and *16–19*). When KCl was used in the reaction in place of KBr, a rather poor yield was obtained. It was observed that the reaction provided a good yield of 77% after 24 h (*Entries 19–21*).

We also checked the cyclization of 4-phenylbutanoic acid under the cyclization conditions of *Ishibara* and co-workers [20]; however, no γ-phenyl-γ-lactone was obtained.

With the optimal conditions in hand, and to check the general applicability of this method, aryl-substituted alkanoic acids were reacted with 0.2 equiv. of PhI, 1.2 equiv. of *m*CPBA, and 5.0 equiv. of KBr in TFE 24 h (*Table 2*).

It is obvious from *Table 2* that the catalytic cyclization was compatible with most of aryl-substituted alkanoic acids and the corresponding aryl lactones were obatined in moderate-to-good yields (*Entries 1–3*, *5*, and *7–10*). Similar treatment of 3-phenylpropanoic acid (**1d**) and 6-phenylhexanoic acid (**1f**), did not furnish the expected product **2d** due to the instability of four-membered lactones (*Entry 4*); another seven-

Table 1. *Optimization of the Catalytic Cyclization of 4-Phenylbutanoic Acid*



1a **2a**

Entry	Solvent	Oxidant (1.2 equiv.)	PhI [equiv.]	KBr [equiv.]	Time [h]	Yield [%][a])
1	CH_2Cl_2	*m*CPBA	0.2	1.0	12	60
2	THF	*m*CPBA	0.2	1.0	12	31
3	MeCN	*m*CPBA	0.2	1.0	12	29
4	MeOH	*m*CPBA	0.2	1.0	12	20
5	EtOH	*m*CPBA	0.2	1.0	12	17
6	AcOEt	*m*CPBA	0.2	1.0	12	29
7	DMF	*m*CPBA	0.2	1.0	12	28
8	CF_3CH_2OH	*m*CPBA	0.2	1.0	12	64
9	CF_3CH_2OH	$NaBO_3 \cdot 4\ H_2O$	0.2	1.0	12	19
10	CF_3CH_2OH	*Oxone*®	0.2	1.0	12	31
11	CF_3CH_2OH	$Na_2S_2O_8$	0.2	1.0	12	20
12	CF_3CH_2OH	*m*CPBA	0	1.0	12	0
13	CF_3CH_2OH	*m*CPBA	0.1	1.0	12	50
14	CF_3CH_2OH	*m*CPBA	0.3	1.0	12	56
15	CF_3CH_2OH	*m*CPBA	0.4	1.0	12	54
16	CF_3CH_2OH	*m*CPBA	0.2	0	12	0
17	CF_3CH_2OH	*m*CPBA	0.2	0.5	12	35
18	CF_3CH_2OH	*m*CPBA	0.2	1.5	12	65
19	CF_3CH_2OH	*m*CPBA	0.2	5.0	12	70
20	CF_3CH_2OH	*m*CPBA	0.2	5.0	8	52
21	CF_3CH_2OH	*m*CPBA	0.2	5.0	24	77

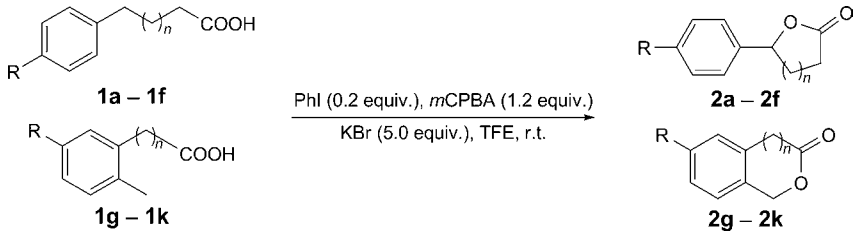
[a]) Yield of isolated **2a**.

membered ring product **2f** was obtained in a yield of only 18% (*Entry 6*). Therefore, the catalytic cyclization was suitable for the preparation of five-membered and six-membered aryl lactones, especially with respect of γ-aryl-γ-lactones.

A plausible reaction pathway for the present reaction is outlined in *Scheme 2*. PhI is first oxidized by *m*CPBA to the hypervalent iodine intermediate, which is then transformed to the active key species by treatment with KBr, followed by a benzyl radical formation *via* sp^3 C–H abstraction. Finally, the intramolecular cyclization takes place to provide the corresponding aryl lactones. The reduced by-product PhI is re-oxidized into hypervalent iodine reagent by *m*CPBA in the recycling reaction.

Conclusions. – Lactones are important and ubiquitous building blocks in organic synthesis, material science, medicine, polymer field, and natural-product chemistry [23–27]. Because some aryl-substituted lactones possess several significant biological features, such as antibiotic and cytotoxic activities [28–33], the methods for their preparation have been of increasing importance. The direct oxidative cyclizations from the corresponding aryl-substituted alkanoic acids is a convenient and attractive route for the synthesis of these lactones [34–38]. However, the use of expensive, unstable, or

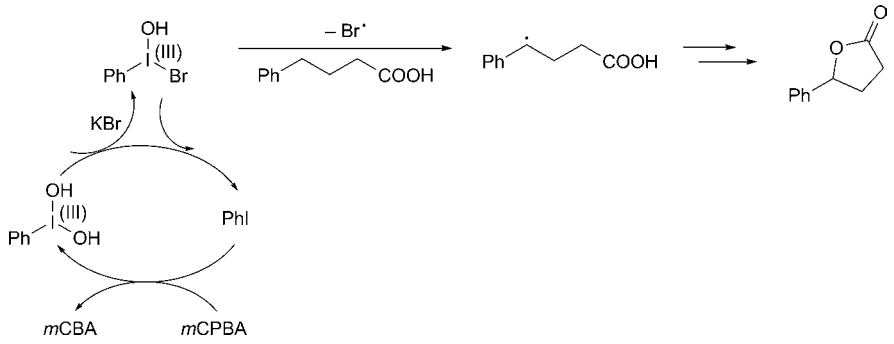
Table 2. *Catalytic Cyclization of Aryl Alkanoic Acids*



Entry	**1**	R	*n*	Lactone (**2**)	Yield [%][a])
1	**1a**	H	1	**2a**	77
2	**1b**	Br	1	**2b**	70
3	**1c**	Me	1	**2c**	60
4	**1d**	H	0	**2d**	0
5	**1e**	H	2	**2e**	58
6	**1f**	H	3	**2f**	18
7	**1g**	H	0	**2g**	72
8	**1h**	Me	0	**2h**	55
9	**1i**	Br	0	**2i**	57
10	**1j**	H	1	**2j**	67
11	**1k**	Me	1	**2k**	43

[a]) Yield of isolated **2**.

Scheme 2. *A Possible Catalytic Cycle for Cyclization of Aryl-Substituted Alkanoic Acids*



toxic metal oxidants and the need of higher reaction temperatures in these procedures has limited their oxidative cyclizations [39–43]. Our novel method using a catalytic amount of PhI and *m*CPBA as the terminal oxidant in the presence of KBr has reduced the amount of expensive hypervalent iodine reagent distinctly, offering a more environmentally benign procedure. It has some advantages such as mild reaction conditions and simple procedure, and it affords moderate-to-good yields. Further investigation of the cyclizations of aryl-substituted alkanioc acids catalyzed by the *in situ* generated hypervalent iodine intermediate will be reported in due course.

Experimental Part

General. Alkanoic acids, *m*CPBA, PhI, and KBr were commercially available. M.p.: *XT-4* melting-point apparatus; uncorrected. IR Spectra: *Thermo-Nicolet 6700* instrument; $\tilde{\nu}$ in cm^{-1}. ^1H- and ^{13}C-NMR spectra: *Bruker AVANCE III* (500 MHz) spectrometer; δ in ppm rel. to Me_4Si as internal standard, *J* in Hz. MS: *Thermo-ITQ 1100* mass spectrometer; in *m*/*z*.

General Procedure for the Catalytic Cyclization of Alkanoic Acids. To TFE (5.0 ml), alkanoic acid **1** (0.5 mmol), *m*CPBA (75%, 0.6 mmol), PhI (0.1 mmol), and KBr (2.5 mmol) were added. The resulting soln. was stirred at r.t. for 24 h. Then, H_2O (10 ml), sat. aq. $Na_2S_2O_3$ (4 ml) and sat. aq. Na_2CO_3 (4 ml) were poured into the mixture. The mixture was extracted with CH_2Cl_2 (3 × 10 ml), and the combined org. layer was washed with brine, dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue was purified by prep. TLC (silica gel; hexane/AcOEt 3:1) to give the pure aryl lactone **2**.

*4,5-Dihydro-5-phenyl-2(3*H*)-furanone* (**2a**). Yield: 62 mg (77%). White solid. M.p. 35–36° ([19]: 35–36°). IR (film): 3070, 1754, 1616, 1493, 1297, 1216, 1176, 1140, 1022, 762, 698. ^1H-NMR: 7.43–7.34 (*m*, 5 H); 5.53 (*t*, *J* = 5.0, 1 H); 2.72–2.64 (*m*, 3 H); 2.24–2.18 (*m*, 1 H). ^{13}C-NMR: 176.87; 139.40; 128.77; 128.45; 125.28; 81.22; 30.95; 28.94. ESI-MS: 162 (47, M^+), 117 (100).

*5-(4-Bromophenyl)-4,5-dihydro-2(3*H*)-furanone* (**2b**). Yield: 84 mg (70%). White solid. M.p. 81–82° ([44]: 81.5–82.5°). IR (film): 1763, 1592, 1490, 1178, 1142, 1008, 828, 804. ^1H-NMR: 7.53 (*d*, *J* = 5.0, 2 H); 7.23 (*d*, *J* = 5.0, 2 H); 5.49–5.46 (*m*, 1 H); 2.68–2.65 (*m*, 3 H); 2.19–2.13 (*m*, 1 H). ^{13}C-NMR: 176.48; 138.44; 131.94; 126.96; 122.40; 80.42; 30.90; 28.83. ESI-MS: 240 (19, $M^+(Br^{79})$), 242 (20, $M^+(Br^{81})$), 161 (100).

*4,5-Dihydro-5-(4-methylphenyl)-2(3*H*)-furanone* (**2c**). Yield: 53 mg (60%). White solid. M.p. 73–74° ([45]: 71–72.5°). IR (film): 3036, 2932, 1721, 1603, 1493, 1165, 1139, 1037, 1008, 852. ^1H-NMR: 7.23 (*d*, *J* = 5.0, 4 H); 5.50 (*dd*, *J* = 10.0, 5.0, 1 H); 2.68–2.63 (*m*, 3 H); 2.37 (*s*, 3 H); 2.22–2.19 (*m*, 1 H). ^{13}C-NMR: 176.96; 138.33; 136.35; 129.41; 125.35; 81.33; 30.94; 29.03; 21.13. ESI-MS: 176 (26, M^+), 91 (100).

*3,4,5,6-Tetrahydro-6-phenyl-2*H*-pyran-2-one* (**2e**). Yield: 51 mg (58%). White solid. M.p. 75–76° ([19]: 76–78°). IR (film): 3043, 2969, 2950, 1727, 1493, 1245, 1035, 764, 705. ^1H-NMR: 7.42–7.33 (*m*, 5 H); 5.38 (*dd*, *J* = 10.0, 5.0, 1 H); 2.76–2.69 (*m*, 1 H); 2.63–2.56 (*m*, 1 H); 2.21–2.17 (*m*, 1 H); 2.03–1.99 (*m*, 2 H); 1.93–1.86 (*m*, 1 H). ^{13}C-NMR: 171.31; 139.75; 128.61; 128.27; 125.70; 81.63; 30.52; 29.52; 18.61. ESI-MS: 176 (5.6, M^+), 104 (100).

7-Phenyloxepan-2-one (**2f**). Yield: 15 mg (18%). White solid. M.p. 67–68° ([19]: 66–67°). IR (film): 2924, 1766, 1616, 1520, 1183, 1147, 1011, 940, 773, 723. ^1H-NMR: 7.41–7.36 (*m*, 5 H); 5.31 (*d*, *J* = 10.0, 1 H); 2.82–2.76 (*m*, 2 H); 2.16–2.02 (*m*, 4 H); 1.81–1.75 (*m*, 2 H). ^{13}C-NMR: 174.84; 140.79; 128.57; 128.10; 125.86; 82.11; 37.49; 34.98; 28.63; 22.86. ESI-MS: 190 (47, M^+), 117 (100).

*2-Benzofuran-1(3*H*)-one* (**2g**). Yield: 48 mg (72%). White solid. M.p. 72–74° ([19]: 73–74°). IR (film): 3023, 2927, 2888, 1742, 1600, 1490, 1457, 1249, 1220, 1144, 1029, 752. ^1H-NMR: 7.93 (*d*, *J* = 10.0, 1 H); 7.71–7.68 (*m*, 1 H); 7.56–7.50 (*m*, 2 H); 5.34 (*s*, 2 H). ^{13}C-NMR: 171.08; 146.54; 134.00; 129.05; 125.79; 122.09; 69.64. ESI-MS: 134 (2.1, M^+), 105 (100).

*6-Methyl-2-benzofuran-1(3*H*)-one* (**2h**). Yield: 41 mg (55%). White solid. M.p. 85–86° ([46]: 88°). IR (film): 3037, 2925, 1759, 1591, 1496, 1457, 1156, 1056, 996, 821, 770. ^1H-NMR: 7.67 (*s*, 1 H); 7.48 (*d*, *J* = 5.0, 1 H); 7.37 (*d*, *J* = 10.0, 1 H); 5.26 (*s*, 2 H); 2.44 (*s*, 3 H). ^{13}C-NMR: 171.16; 143.82; 139.14; 135.10; 125.75; 125.50; 121.73; 69.51; 21.12. ESI-MS: 148 (8.1, M^+), 91(100).

*6-Bromo-2-benzofuran-1(3*H*)-one* (**2i**). Yield: 61 mg (57%). White solid. M.p. 97–98° ([47]: 96–98°). IR (film): 3076, 1769, 1458, 1358, 1212, 1191, 1046, 997, 870, 824, 767. ^1H-NMR: 8.08 (*d*, *J* = 5.0, 1 H); 7.82 (*d*, *J* = 10.0, 1 H); 7.41 (*d*, *J* = 5.0, 1 H); 5.30 (*s*, 2 H). ^{13}C-NMR: 169.42; 145.09; 137.09; 128.83; 127.91; 123.65; 123.05; 69.46. ESI-MS: 212 (11, $M^+(Br^{79})$), 214 (13, $M^+(Br^{81})$), 133 (100).

*1,4-Dihydro-3*H*-2-benzopyran-3-one* (**2j**). Yield: 50 mg (67%). White solid. M.p. 80–81° ([48]: 81–82°). IR (film): 3023, 2889, 1746, 1489, 1458, 1392, 1252, 1224, 1147, 1034, 761. ^1H-NMR: 7.37–7.31 (*m*, 2 H); 7.27–7.23 (*m*, 2 H); 5.33 (*s*, 2 H); 3.73 (*s*, 2 H). ^{13}C-NMR: 170.66; 131.55; 130.97; 128.82; 127.37; 127.07; 124.68; 70.08; 36.18. ESI-MS: 148 (5.0, M^+), 104 (100).

*1,4-Dihydro-6-methyl-3*H*-2-benzopyran-3-one* (**2k**). Yield: 35 mg (43%). White solid. M.p. 70–71° ([48]: 72–73°). IR (film): 2921, 1725, 1616, 1590, 1501, 1461, 1255, 1216, 1157, 1126, 1021, 833, 780.

^{1}H-NMR: 7.13 (*dd*, *J* = 10.0, 5.0, 2 H); 7.05 (*s*, 1 H); 5.29 (*s*, 2 H); 3.68 (*s*, 2 H); 2.38 (*s*, 3 H). ^{13}C-NMR: 170.85; 138.81; 130.93; 128.60; 127.99; 127.70; 124.54; 70.01; 36.20; 21.19. ESI-MS: 162 (53, M^+), 117 (100).

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