## Novel phosphoryloxylactonisation of pentenoic acids mediated by ammonium iodide

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Using ammonium iodide as a catalyst and *m*-chloroperbenzoic acid as the oxidant, a novel and efficient procedure has been developed for phosphoryloxylactonisation of alkenoic acids in  $CH_3CN$  at room temperature, which provides the corresponding phosphoryloxylactones in good yields. In this protocol, it is proposed that ammonium iodide catalyst is first oxidised to hypoiodous acid, which reacts with the alkenoic acid to afford the corresponding iodolactone. Then, the iodolactone is further transformed into a hypervalent iodine intermediate by continuing oxidation, and finally the *in situ* generated active iodine species reacts with a diaryl phosphate to furnish the phosphoryloxylactone end product.

Keywords: phosphoryloxylactonisation, hypervalent iodine intermediate, ammonium iodide, catalytic cyclisation, pentenoic acids

Hypervalent iodine reagents have found broad application in organic chemistry and are frequently used in synthesis as they are non-metallic oxidation reagents and avoid the issues of toxicity of many transition metals commonly involved in such processes.<sup>1-10</sup> Using hypervalent iodine reagents, some special compounds such as phosphoryloxylactones which are important in organic chemistry as well as biochemistry have been prepared. In 1988, Koser's group utilised a novel hypervalent iodine reagent, [hydroxyl((bis(phenyloxy) phosphoryl)oxy)iodo]-benzene, to react with pent-4-enoic acids and prepared some new phosphoryloxylactones.<sup>11</sup> However, as this method was a two-step procedure and the yields are low, the number of prepared phosphoryloxylactones is only three. Other methods for synthesis of phosphoryloxylactones, except direct phosphorylation of sugar lactones, are less.<sup>12,13</sup> In order to develop more efficient procedures for the preparation of phosphoryloxylactones, we recently improved Koser's method using (diacetoxyiodo)benzene and phosphates in place of [hydroxy((bis(phenyloxy)phosphoryl)oxy)iodo]benzene and reported a "one pot" procedure.14

The catalytic utilisation of hypervalent iodine reagents as catalysts has been increasing in importance, with growing interest in the development of environmentally benign synthetic transformations.<sup>15-19</sup> In these reactions, a substoichiometric amount of an iodine-containing compound together with a stoichiometric amount of oxidant are used. Recently, Togo and co-workers have investigated a new method for the  $\alpha$ -tosyloxylation of ketones using molecular iodine as catalyst, which has been shown to involve a hypervalent iodine intermediate. <sup>20</sup> This method avoids the need for expensive aryl iodides and gives good yields. It is well known that molecular iodine can be easily accessed from iodides, but the use of the one-pot reaction mediated by hypervalent iodine species generated in situ from iodides, especially inorganic iodides, a method which is more environmentally benign has been quite limited.21-23

In order to extend the scope of hypervalent iodine reagents for organic synthesis, we have developed a method for phosphoryloxylactonisation of alkenoic acids using iodobenzene as catalyst, and a series of phosphoryloxylactones has been prepared.<sup>24</sup> Based on this success, we have now investigated the more environmentally benign catalytic phosphoryloxylactonisation of alkenoic acids in the presence of a sub-stoichiometric amount of ammonium iodide (NH<sub>4</sub>I), and obtained the desired phosphoryloxylactones in good yields. We now report this novel phosphoryloxylactonisation of alkenoic acids catalysed by NH<sub>4</sub>I.

Firstly, we examined the reaction of equivalent amounts of pent-4-enoic acid, diphenyl phosphate and m-chloroperbenzoic acid (mCPBA) in the presence of 0.1 equiv. of NH<sub>4</sub>I in CH<sub>2</sub>CN at room temperature and found that this reaction occurred readily and provided the desired product 5-(bis(phenyloxy) phosphory)oxy-4-pentanolactone (Table 1). Encouraged by the result, the reaction conditions were optimised. As shown in Table 1, solvent influenced the reaction greatly and CH<sub>3</sub>CN was superior to other solvents (entries 1-6). 2.0 Equiv. of mCPBA and 1.2 equiv. of diphenyl phosphate were optimal for the reaction (entries 5, 7-13). The amount of NH<sub>4</sub>I was also investigated, and 0.1 equiv. of it was optimal (entries 11, 14-15). However, if NH,I was absent, no desired product was observed (entry 16). The reaction was completed in several hours and 8 h was selected as the best suitable reaction time (entries 11, 17-19). Other oxidants, such as Oxone, NaBO, and  $Na_{3}S_{2}O_{8}$ , when they were in place of *m*CPBA, usually resulted in bad yields (entries 20-22).

Under the optimal reaction conditions, we investigated the phosphoryloxylactonisation of 1.0 equiv. of a range of alkenoic acids (1), 1.2 equiv. of a selection of diarylphosphate (2) and 2.0 equiv. of *m*CPBA with 0.1 equiv. of  $\text{NH}_4\text{I}$  in CH<sub>3</sub>CN at room temperature in 8 h (Scheme 1). The results are summarised in Table 2.



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 Table 1
 Optimisation of the NH<sub>4</sub>I catalysed phosphoryloxylactonisation of pent-4-enoic acid

$\bigcirc 0 \\ \bigcirc 0 \\ 0 \\$	$\xrightarrow{\text{NH}_{4}\text{I}}_{\text{RT}} \xrightarrow{\text{O}}_{\text{O}} \xrightarrow{\text{O}}_{\text{O}} \xrightarrow{\text{O}}_{\text{OP(OPh)}_2}$
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Entry	NH <sub>4</sub> I/equiv.	Oxidant/equiv. <sup>b</sup>	Diphenyl phosphate/equiv.	Time/h	Solvent	Yield/%ª
1	0.1	1.5	1.0	8	THF	21
2	0.1	1.5	1.0	8	CH₃OH	25
3	0.1	1.5	1.0	8	DMF	3
4	0.1	1.5	1.0	8	CF <sub>3</sub> CH <sub>2</sub> OH	54
5	0.1	1.5	1.0	8	CH₃CN	61
6	0.1	1.5	1.0	8	CH <sub>2</sub> Cl <sub>2</sub>	32
7	0.1	2.0	1.0	8	CH₃CN	68
8	0.1	2.5	1.0	8	CH <sub>3</sub> CN	65
9	0.1	1.2	1.0	8	CH₃CN	45
10	0.1	1.0	1.0	8	CH <sub>3</sub> CN	34
11	0.1	2.0	1.2	8	CH <sub>3</sub> CN	71
12	0.1	2.0	1.5	8	CH <sub>3</sub> CN	72
13	0.1	2.0	2.0	8	CH₃CN	73
14	0.2	2.0	1.2	8	CH <sub>3</sub> CN	72
15	0.05	2.0	1.2	8	CH <sub>3</sub> CN	52
16	0	2.0	1.2	8	CH <sub>3</sub> CN	0
17	0.1	2.0	1.2	4	CH <sub>3</sub> CN	55
18	0.1	2.0	1.2	12	CH <sub>3</sub> CN	73
19	0.1	2.0	1.2	15	CH <sub>3</sub> CN	74
20	0.1	(2.0) <sup>c</sup>	1.2	8	CH <sub>3</sub> CN	24
21	0.1	(2.0) <sup>d</sup>	1.2	8	CH₃CN	5
22	0.1	(2.0) <sup>e</sup>	1.2	8	CH <sub>3</sub> CN	13

alsolated yield.

<sup>b</sup>mCPBA unless otherwise indicated: <sup>c</sup>Oxone; <sup>d</sup>NaBO<sub>3</sub>; <sup>e</sup>Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub>.

Table 2 shows that the studied alkenoic acids readily reacted with phosphates (2a and 2b) catalysed by NH<sub>4</sub>I, to provide 5-phosphoryloxy-4-pentanolactones the corresponding were obtained in moderate to good yields (Table 2, entries 1-7). Phosphoryloxylactones 3b, 3c and 3f were mixtures of diastereoisomers, the ratios were 2.8:1 for 3b, 1:1 for 3c and 2.4:1 for 3f respectively, which were determined by examination of the <sup>1</sup>H NMR spectra of phosphoryloxylactones. Bis(4-nitrophenyl)phosphate (2c) also reacted with pent-4enoic acid, but the product (3h) was unstable and decomposed during purification; the yield was measured on the crude product by <sup>1</sup>H NMR (entry 8). In order to prepare the related sixmembered and four-membered phosphoryloxylactones, hex-5enoic acid and but-3-enoic acid were treated under the same conditions, but neither of the desired phosphoryloxylactones were obtained. It may be that they were not formed or were unstable and decomposed during purification, which was as we have observed before.<sup>11,14,24</sup> Therefore, our new method is only suitable for preparation of five-membered phosphoryloxylactones.

A plausible reaction pathway for the present reaction is shown in Scheme 2, which is different from that we reported previously.<sup>24</sup> Thus,  $NH_4I$  is first oxidised by *mCPBA* to HOI, which reacts with the alkenoic acid to afford the iodolactone.<sup>25</sup> Then, this is transformed into the hypervalent iodine intermediate *in situ* by continuing oxidation.<sup>26,27</sup> The hypervalent iodine intermediate is generally highly unstable and when it reacts with phosphate, the phosphoryloxylactone is formed by nucleophilic substitution.<sup>2-4</sup> To support the proposed mechanism, pent-4-enoic acid was first treated with HOI,<sup>25</sup> which provided the iodolactone in nearly quantitative yield. Then, the reaction of this iodolactone with phosphate was examined. In the absence of oxidant *m*CPBA, the desired product 5-phosphoryloxy-4-pentanolactone was not observed even after a long reaction time. However, when *m*CPBA was added to the reaction mixture, the desired product



Entry	Alkenoic acids (1)	Phosphates ( <b>2</b> )	Phosphoryloxylactones ( <b>3</b> )	Yield/% <sup>a</sup>
1	R <sup>1</sup> , R <sup>2</sup> , R <sup>3</sup> =H <b>1a</b>	R=Ph <b>2a</b>	O O 3a	71
2	R¹=Me R², R³=H <b>1b</b>	2a	Me O O O O O O O O O O O O O O O O O O O	74
3	R²=Me R¹, R³=H <b>1c</b>	2a	$Me \xrightarrow{O} 3c^{\flat}$	63
4	R¹, R²=Me R³=H <b>1d</b>	2a	Me O 3d O O 3d OP(OPh) <sub>2</sub>	74
5	1a	R=Bn <b>2b</b>	O O OP(OBn) <sub>2</sub> 3e	71
6	16	2b	$ \begin{array}{c} Me \\ O \\ O \\ W \\ OP(OBn)_2 \end{array} $	68
7	1d	2b	Me O <b>3g</b>	76
8	1a	R=C <sub>6</sub> H <sub>4</sub> NO <sub>2</sub> - <i>p</i> <b>2c</b>	$\bigcup_{0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\$	47 <sup>c</sup>

 Table 2
 The catalytic phosphoryloxylactonisation of alkenoic acids

<sup>a</sup>lsolated yields.

<sup>b</sup>See text (diastereoisomeric mixture).

<sup>c</sup> <sup>1</sup>H NMR analysis.

was obtained in good yield. Therefore, an *in situ* generated hypervalent iodine intermediate is responsible for effecting the reaction.

In summary, we have developed a novel and efficient procedure for phosphoryloxylactonisation of alkenoic acids using a sub-stoichiometric amount of  $NH_4I$  with *mCPBA* in CH<sub>3</sub>CN at room temperature. The results highlight the advantageous role of inorganic iodide as the catalyst as a replacement for expensive aryl iodides. Moreover, it extends the scope of hypervalent iodine reagents in organic synthesis, as well as being more environmentally benign.

## Experimental

Melting points were determined on a digital apparatus and were not corrected. IR spectra were recorded on a Thermo-Nicolet 6700 instrument, NMR spectra were measured on a Bruker Avance III (500 MHz) spectrometer using TMS as internal standard for <sup>1</sup>H and <sup>13</sup>C spectra and 80% phosphoric acid as standard for <sup>31</sup>P spectra. Mass spectra were determined on a Thermo-ITQ 1100 mass spectrometer. Alkenoic acids, phosphates, *m*CPBA, NH<sub>4</sub>I and solvents were commercially available. *NH*<sub>4</sub>*I* catalytic phosphoryloxylactonisation of alkenoic acids; typical procedure

To CH<sub>3</sub>CN (5 mL), pent-4-enoic acid (0.030 g, 0.3 mmol), diphenyl phosphate (0.090 g, 0.36 mmol), mCPBA (75%, 0.138 g, 0.60 mmol) and NH<sub>4</sub>I (0.0043 g, 0.030 mmol) were added. The mixture was stirred at room temperature for 8 h. Then H<sub>2</sub>O (5 mL), sat.aq Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (2 mL) and sat. aq Na<sub>2</sub>CO<sub>3</sub> (2 mL) were added. The mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×5 mL) and the combined organic extract was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. The residue was purified on silica gel plate using (3:2 hexane-ethyl acetate) as eluent to afford 5-(bis(phenyloxy) phosphoryl)oxy-4-pentanolactone in 71% yield.

5-(*Bis(phenyloxy)phosphoryl)oxy-4-pentanolactone* (**3a**): White solid, m.p. 77–78 °C (lit.<sup>14</sup> 77–78 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.37–7.34 (m, 4H), 7.23–7.20 (m, 6H), 4.72–4.70 (m, 1H), 4.47–4.43 (m, 1H), 4.32–4.27 (m, 1H), 2.49–2.45 (m, 2H), 2.33–2.26 (m, 1H), 2.10–2.03 (m, 1H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 176.2, 150.3 (dd, *J*=6.3, 2.5 Hz), 129.9, 125.6, 120.0 (d, *J*=5.0 Hz), 77.3, 69.2 (d, *J*=5.0 Hz), 27.9, 23.2. <sup>31</sup>P NMR (202 MHz, CDCl<sub>3</sub>): δ –12.1. IR (neat, cm<sup>-1</sup>): 1771, 1593, 1489, 1293, 1221, 1189, 1044, 947. MS (EI, *m/z*, %): 348 (M<sup>+</sup>, 62.3), 249 (100). HRMS: *m/z* [M]<sup>+</sup> calcd for C<sub>17</sub>H<sub>17</sub>O<sub>6</sub>P: 338.0763; found: 348.0751.

5-(Bis(phenyloxy)phosphoryl)oxy-2-methyl-4-pentanolactone (3b) (2:8:1 diastereoisomeric mixture):<sup>24</sup> Light yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): *δ* 7.38–7.34 (m, 4H), 7.24–7.19 (m, 6H), 4.68–4.65 (m, 0.26H), 4.59-4.54 (m, 0.74H), 4.48-4.40 (m, 1H), 4.31-4.26 (m, 1H), 2.73-2.63 (m, 0.74H), 2.59-2.54 (m, 0.26H), 2.45-2.39 (m, 0.74H), 2.33-2.28 (m, 0.26H), 2.01-1.95 (m, 0.26H), 1.71-1.61 (m, 0.74H), 1.23 (d, J=7.5 Hz, 2.22H), 1.21 (d, J=7.5 Hz, 0.78H). <sup>13</sup>C NMR  $(125 \text{ MHz}, \text{CDCl}_{2})$ :  $\delta$  179.1, 178.3, 150.3 (dd, J=7.5, 3.8 Hz), 129.8 (d, J=5.0 Hz), 125.5, 120.0 (dd, J=7.5, 3.8 Hz), 75.4 (d, J=7.5 Hz), 74.9 (d, J=8.8 Hz), 69.5 (d, J=5.0 Hz), 68.5 (d, J=5.0 Hz), 35.1, 33.6, 31.8, 31.4, 16.0, 15.0. <sup>31</sup>P NMR (202 MHz, CDCl<sub>2</sub>):  $\delta$  –12.1, –12.0. IR (neat, cm<sup>-1</sup>): 3070, 1775, 1590, 1489, 1294, 1190, 1024, 955. MS (EI, m/z, %): 362  $(M^+, 34.8), 249 (100).$  HRMS:  $m/z [M]^+$  calcd for  $C_{18}H_{10}O_6P$ : 362.0919; found: 362,0901

5-(Bis(phenyloxy)phosphoryl)oxy-3-methyl-4-pentanolactone (3c) (1:1 diastereoisomeric mixture):<sup>24</sup> Light yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>2</sub>): δ 7.38-7.34 (m, 4H), 7.23-7.20 (m, 6H), 4.63-4.60 (m, 0.5H), 4.48-4.43 (m, 1H), 4.41-4.37 (m, 0.5H), 4.34-4.29 (m, 0.5H), 4.23-4.20 (m, 0.5H), 2.77-2.72 (m, 0.5H), 2.66-2.55 (m, 1H), 2.46-2.40 (m, 0.5H), 2.22-2.14 (m, 1H), 1.15 (d, J=7.0 Hz, 1.5H), 1.09 (d, J=7.5 Hz, 1.5H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 175.7, 175.4, 150.2 (d, J=7.5 Hz), 129.9, 125.6, 120.0 (d, J=5.0 Hz), 83.9 (d, J=7.5 Hz), 79.7 (d, J=7.5 Hz), 67.8 (d, J=6.3 Hz), 67.1 (d, J=6.3 Hz), 36.2 (d, J=23.8 Hz), 31.8, 31.3, 18.0, 13.5. IR (neat, cm<sup>-1</sup>): 3068, 1786, 1592, 1489, 1293, 1190, 1029, 956. MS (EI, m/z, %): 362 (M<sup>+</sup>, 11.2), 249 (100). HRMS: m/z [M]<sup>+</sup> calcd for C<sub>18</sub>H<sub>19</sub>O<sub>6</sub>P: 362.0919; found: 362.0893.

5-(Bis(phenyloxy)phosphoryl)oxy-2,2-dimethyl-4-pentanolactone (**3d**):<sup>24</sup> Yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.37–7.34 (m, 4H), 7.24-7.19 (m, 6H), 4.65-4.58 (m, 1H), 4.47-4.43 (m, 1H), 4.29-4.24 (m, 1H), 2.09-2.05 (m, 1H), 1.92-1.87 (m, 1H), 1.25 (s, 3H), 1.23 (s, 3H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  180.8, 150.3 (t, *J*=6.3 Hz), 129.9, 125.6, 120.1 (d, J=3.8 Hz), 120.0 (d, J=5.0 Hz), 74.1 (d, J=7.5 Hz), 68.8 (d, J=5.0 Hz), 39.9, 38.2, 24.7 (d, J=3.8 Hz). IR (neat, cm<sup>-1</sup>): 3069, 1776, 1592, 1489, 1293, 1190, 1025, 954. MS (EI, m/z, %): 376 (M<sup>+</sup>, 100). HRMS: m/z [M]<sup>+</sup> calcd for C<sub>19</sub>H<sub>21</sub>O<sub>6</sub>P: 376.1076; found: 376.1047.

5-(Bis(benzyloxy)phosphoryl)oxy-4-pentanolactone (3e):24 Yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>2</sub>): δ 7.36-7.35 (m, 10H), 5.09-5.01 (m, 4H), 4.61-4.58 (m, 1H), 4.16-4.12 (m, 1H), 4.02-3.97 (m, 1H), 2.49-2.43 (m, 2H), 2.27-2.19 (m, 1H), 2.03-1.95 (m, 1H). <sup>13</sup>C NMR  $(125 \text{ MHz}, \text{ CDCl}_3)$ :  $\delta$  176.4, 135.5 (dd, J=6.3, 3.8 Hz), 128.7 (d, J=7.5 Hz), 128.1 (d, J=3.8 Hz), 77.5 (d, J=8.8 Hz), 69.6 (d, J=6.3 Hz), 68.0 (d, J=5.0 Hz), 28.0, 23.2. IR (neat, cm<sup>-1</sup>): 3064, 1779, 1498, 1456, 1278, 1013. MS (EI, m/z, %): 376 (M<sup>+</sup>, 22.3), 277 (100). HRMS: m/z  $[M]^+$  calcd for  $C_{19}H_{21}O_6P$ : 376.1076; found: 376.1075.

5-(Bis(benzoyloxy)phosphoryl)oxy-2-methyl-4-pentanolactone (**3f**):<sup>24</sup> Yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>2</sub>): δ 7.37–7.34 (m, 10H),5.10-5.02 (m, 4H), 4.58-4.52 (m, 0.29H), 4.48-4.45 (m, 0.71H), 4.18-4.13 (m, 0.71H), 4.10-4.08 (m, 0.29H), 4.02-3.97 (m, 1H), 2.70-2.60 (m, 1H), 2.39-2.33 (m, 0.71H), 2.23-2.18 (m, 0.29H), 1.95-1.90 (m, 0.29H), 1.63-1.59 (m, 0.71H), 1.25 (d, J=7.0 Hz, 2.13H), 1.22 (d, J=7.5 Hz, 0.87H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>2</sub>):  $\delta$  179.3, 178.5,

135.5 (d, J=6.3 Hz), 128.7, 128.6 (d, J=3.8 Hz), 128.0 (d, J=2.5 Hz), 75.6 (d, J=7.5 Hz), 75.1 (d, J=7.5 Hz), 69.6 (d, J=5.0 Hz), 68.2 (d, J=6.3 Hz), 67.5 (d, J=5.0 Hz), 35.1, 33.6, 31.9, 31.5, 16.0, 15.1. IR (neat, cm<sup>-1</sup>): 3034, 1774, 1597, 1456, 1278, 1171, 1012. MS (EI, *m/z*, %): 390  $(M^+, 18.8), 277 (100).$  HRMS:  $m/z [M]^+$  calcd for  $C_{20}H_{22}O_cP$ : 390.1232; found: 390.1201

5-(Bis(benzovloxy)phosphoryl)oxy-2,2-dimethyl-4-pentanolactone (**3g**):<sup>24</sup>Yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>2</sub>): δ 7.37–7.35 (m, 10H), 5.10-5.02 (m, 4H), 4.53-4.49 (m, 1H), 4.16-4.12 (m, 1H), 4.01-3.96 (m, 1H), 2.03-1.99 (m, 1H), 1.85-1.80 (m, 1H), 1.24 (s, 6H). 13C NMR (125 MHz, CDCl<sub>2</sub>): δ 181.0, 135.6 (d, J=6.3 Hz), 128.7 (d, J=3.8 Hz), 128.1 (d, J=2.5 Hz), 74.3 (d, J=7.5 Hz), 69.6 (d, J=5.0 Hz), 67.8 (d, J=5.0 Hz), 39.9, 38.3, 24.8 (d, J=8.8 Hz). IR (neat, cm<sup>-1</sup>): 1764, 1460, 1267, 1024, 991. MS (EI, m/z, %): 404 (M<sup>+</sup>, 50.1), 277 (100). HRMS: m/z [M]<sup>+</sup> calcd for C<sub>21</sub>H<sub>25</sub>O<sub>6</sub>P: 404.1389; found: 404.1377.

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