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Advance Publication on the web September 1, 2020 doi:10.1246/cl.200530

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# Synthesis of Alkyl Sulfones from Alkenes and Tosylmethylphosphonium Iodide through Photo-Promoted C–C Bond Formation

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1 A new synthetic method of alkyl sulfones through C–C 2 bond formation between alkenes and 3 tosylmethylphosphonium iodide is reported. A tosylmethyl 4 radical is generated from the phosphonium iodide under 5 irradiation of visible light with the aid of *fac*-Ir(ppy)<sub>3</sub>. It 6 undergoes regioselective 1,2-addition across the carbon– 7 carbon double bond to afford an elongated alkyl radical, 8 which abstracts a hydrogen atom from C<sub>6</sub>F<sub>5</sub>SH, producing 9 an alkyl sulfone with one-carbon extension.

#### 10 Keywords: sulfones, phosphonium salts, radicals

11 Sulfones are not only structural motifs prevalent in 12 natural products, pharmaceuticals, and agrochemicals,<sup>1</sup> but also synthetic intermediates versatile and useful in organic 13 chemistry.<sup>2</sup> A recent emergence of sulfone-containing 14 functional materials<sup>3</sup> has sparked a renewed interest in their 15 16 synthetic methods. There are a number of authentic methods 17 available for the synthesis of this important class of 18 compounds.<sup>4</sup> Most frequently used are (i) preliminary 19 preparation of sulfides followed by oxidation through 20 sulfoxides with oxidants such as peracids or hydrogen 21 peroxide [Figure 1(a)],<sup>5</sup> (ii) alkylation of sulfinates [Figure 22 1(b)],<sup>6</sup> (iii) Friedel-Crafts type reaction of sulfonyl 23 chlorides,<sup>7</sup> (iv) radical addition of sulfonyl halides to alkenes [Figure 1(c)],<sup>8</sup> and (v) addition of sulfinic acids<sup>9</sup> or 24  $\alpha$ -sulforyl carbanions to C–C or C–O double bonds.<sup>10</sup> The 25 26 vast majority of those conventional methods rely upon C-S 27 bond formation for construction of the main frameworks. 28 They often require vigorously oxidative, basic, or acidic 29 conditions, which lead to reduced tolerance for functional 30 groups. An increasing demand of sulfones in various fields 31 of miscellaneous relevance has made it highly desirable to 32 develop a new synthetic method of sulfones which uses widely accessible starting materials, readily available 33 34 reagents, and mild reaction conditions that tolerate a diverse range of functional groups. Alkenes are stable, widely 35 accessible, and thus, suitable for the starting materials 36 constituting main structural backbones of sulfones.<sup>5c,d,8</sup> 37 Nonetheless, a 1,2-addition reaction in which a 38 sulfonylmethyl group is appended to alkenes through C-C 39 bond formation remains underdeveloped.<sup>11,12</sup> A notable 40 example was reported by Renaud et al.<sup>13</sup> Tosylmethyl 41 42 chloride undergoes 1,2-hydro(tosylmethylation) of electron-43 rich alkenes such as enamines. It requires the use of an 44 excess amount of tributyltin hydride and high temperature 45 of 80 °C, which are both possibly averted from experimental procedures. We now report a new synthetic method of alkyl 46 47 sulfones through C-C bond formation between alkenes and a sulfone-stabilized phosphonium salt [Figure 1(d)]. The 48 49 reaction occurring at ambient temperature uses visible light,

a) preliminary preparation of sulfides followed by oxidation<sup>5</sup>

R'SH NaOH  

$$(R'S)_2 \xrightarrow{Na} [R'SM] \xrightarrow{R-X} R' \xrightarrow{[ox.]} O O$$
  
R'M  $M = Na, Li, MgX; [ox.] = mCPBA, H_2O_2, etc.$ 

b) alkylation of sulfinates<sup>6</sup>

$$R'SO_2CI \xrightarrow{NaHCO_3 aq.} \xrightarrow{O} \qquad \xrightarrow{R-X} \qquad \xrightarrow{O} \qquad \xrightarrow{O} \qquad \xrightarrow{R-X} \qquad \xrightarrow{O} \qquad$$

c) 1,2-hydrosulfonylation of alkenes<sup>8</sup>

$$R'SO_{2}CI \longrightarrow \begin{bmatrix} 0 & 0 \\ \cdot & R' \end{bmatrix} \xrightarrow{R'} H \xrightarrow{R'} H \xrightarrow{R'} O \xrightarrow{R'} R'$$

d) this work: 1,2-hydro(sulfonylmethylation) of alkenes

$$[Ph_{3}PCH_{2}SO_{2}R']^{+}I^{-} \longrightarrow \begin{bmatrix} & & & \\ &$$

51 **Figure 1.** Various methods for the synthesis of alkyl 52 sulfones.

53 to which a variety of functional groups are transparent, and 54 thus, compatible.

55 Recently, have found that we an (alkoxycarbonyl)methyl radical is generated from an ester-56 57 stabilized phosphorus ylide (Ph<sub>3</sub>P=CHCO<sub>2</sub>R) in the 58 presence of a photoredox catalyst under irradiation of 59 visible light.<sup>14</sup> The radical species substituted by an 60 alkoxycarbonyl electron-withdrawing group is so electrophilic as to add onto an alkene in a regioselective 61 fashion to elongate the carbon chain. A similar chain-62 63 elongation reaction of alkenes was achieved with the use of cyano-stabilized phosphorus ylide (Ph<sub>3</sub>P=CHCN).<sup>15</sup> Next, 64 we examined whether a sulfone-stabilized phosphorus ylide 65 (Ph<sub>3</sub>P=CHSO<sub>2</sub>R) could generate a (sulfonyl)methyl radical, 66 which would be electrophilic as well. Its addition to an 67 68 alkene would elongate it by one carbon to furnish the 69 corresponding alkyl sulfone. Firstly, a reaction of a sulfone-70 stabilized phosphorus ylide with an alkene was attempted 71 under the conditions previously employed for the reaction

with an ester-stabilized phosphorus ylide;<sup>14</sup> a mixture of 4-72 73 phenylbut-1-ene (1a, 1.0 equiv), phosphorus ylide 2 74 (Ph<sub>3</sub>P=CHTs, 2.0 equiv), *fac*-Ir(ppy)<sub>3</sub> (1.0 mol %, ppy = 2-75 phenylpyridinato),  $C_6F_5SH$  (20 mol %), and ascorbic acid 76 (5.0 equiv) in 1,2-dimethoxyethane/H<sub>2</sub>O (1:1, 0.1 M) was 77 irradiated with blue LEDs (470 nm) at ambient temperature for 40 hours. A 1,2-hydro(tosylmethylation) reaction took 78 79 place and the desired elongated alkyl sulfone 3a was 80 obtained in 59% isolated yield. No branched isomeric

1 adduct was observed within the detection limit of <sup>1</sup>H NMR 2 (400 MHz). With this preliminary result in hand, the 3 phosphonium salt 4, the precursory compound of 2, was used in place of 2 under slightly modified reaction 4 conditions.<sup>16</sup> A mixture of **1a**, phosphonium iodide **4** (2.0 5 equiv), fac-Ir(ppy)<sub>3</sub> (1.0 mol %), C<sub>6</sub>F<sub>5</sub>SH (10 mol %), 6 sodium ascorbate (2.0 equiv), and KHSO<sub>4</sub> (3.0 equiv)<sup>17</sup> in 7 acetonitrile/H<sub>2</sub>O (1:1, 0.1 M) was irradiated by blue LEDs at 8 9 ambient temperature (Scheme 1). The reaction proceeded faster finishing in 16 hours, and the yield of **3a** increased to 10 81%.18 11

 $\begin{array}{c|c} Ph & fac-lr(ppy)_{3} (1.0 \text{ mol } \%) \\ & 1a & \\ & + & \\ [Ph_{3}PCH_{2}Ts]^{+}l^{-} & \\ & 4 (2.0 \text{ equiv}) & \\ \end{array} \\ \begin{array}{c} fac-lr(ppy)_{3} (1.0 \text{ mol } \%) \\ C_{6}F_{5}SH (10 \text{ mol } \%) \\ & \\ sodium \text{ ascorbate } (2.0 \text{ equiv}) \\ & \\ KHSO_{4} (3.0 \text{ equiv}) \\ & \\ MeCN/H_{2}O (1:1), \text{ rt, 16 h} \\ & \\ \\ blue \text{ LEDs} \end{array} \end{array} Ph \begin{array}{c} & \\ & 3a 81\% \\ & 3a 81\% \\ & \\ \end{array} \\ \begin{array}{c} Ts \\ & 3a 81\% \\ & \\ & \\ \end{array}$ 

13 Scheme 1. 1,2-Hydro(tosylmethylation) of alkene 1a with 14 phosphonium iodide 4: Ts = p-toluenesulfonyl.

15 We propose a mechanism depicted in Scheme 2, which is analogous to that assumed for the reaction of ester- and 16 cyano-stabilized phosphorus ylides.<sup>14,15</sup> Initially, visible 17 light activates the iridium(III) complex, fac-Ir(ppy)<sub>3</sub> to its 18 excited state [Ir(III)]\*. This excited-state complex is a 19 strong reductant  $(E^{\text{red}}_{1/2}[\text{Ir}(\text{IV})/\text{Ir}(\text{III})^*] = -1.81\text{V}$  versus 20 SCE in acetonitrile).<sup>19</sup> It transfers a single electron to the 21 phosphonium iodide 4 into the  $\sigma^*$  orbital of the C–P<sup>+</sup> bond 22 23 which is energetically low-lying and large in lobe. The C–P 24  $\sigma$  bond accommodating one electron in its  $\sigma^*$  orbital breaks 25 into radical 5 and PPh<sub>3</sub>. The tosylmethyl radical 5 is 26 electrophilic enough to add to the alkene **1a** regioselectively. 27 The resulting secondary alkyl radical 6 is not electrophilic 28 enough to add to another molecule of the alkene. Instead, it 29 accepts a hydrogen atom from C<sub>6</sub>F<sub>5</sub>SH to produce the 30 linearly elongated alkyl sulfone 3a and thiyl radical  $(C_6F_5S^{\bullet})^{20}$  Finally,  $[Ir(IV)]^+$  species and  $C_6F_5S^{\bullet}$  are reduced 31 32 back to [Ir(III)] and C<sub>6</sub>F<sub>5</sub>SH, respectively, by sodium 33 ascorbate (Na<sup>+</sup>[AscH]<sup>-</sup>), which turns into dehydroascorbic 34 acid (DHA).21,22

(i) [Ir(III)]  $hv \downarrow$   $[Ir(III)]^* [Ir(IV)]^{+}I^-$  H  $Ir(III)]^* [Ir(IV)]^{+}I^-$  H aa aa aa aa aa aa aa  $C_6F_5S^{-}$  $C_6F_5SH^{-}$ 

The scope of alkenes was examined in the reaction with the phosphonium iodide **4** (Table 1). Terminal alkenes

41 **Table 1.** Scope of alkenes  $1b-1u^a$ 

entry	alkene <b>1</b>	product 3	yield (%) <sup>b</sup>
1	<i>n</i> -BuO	<i>n</i> -BuO	70
2	Ac 1c	Ac Ts 3c	66
3	2-Np 1d	2-Np	75
4	HO <sub>2</sub> C V 1e	$HO_2C_{4}$ Ts 3e	81
5	NC V3 1f	NC Ts 3f	85
6	HO HO Ig	HO Ts 3g	73
7	AcO	Aco Ts 3h	68
8		Cl Ts 3i	69
9	Et <sub>3</sub> Si 🦄 1j	Et <sub>3</sub> Si Ts <b>3j</b>	77
10	(pin)B 1k	(pin)B Ts <b>3k</b>	71
11	11	Ts 31	68
12	1m	Ts 3m	50
13	<i>n</i> -Pr <i>n</i> -Pr ( <i>Z</i> )-1n	<i>n</i> -Pr Ts <b>3n</b>	44
14	<i>n</i> -Pr <i>n</i> -Pr ( <i>E</i> )-1n	<i>n</i> -Pr Ts <b>3n</b>	22
15	10	<b>5 3 0</b>	60
16	Me Me	Ts 3p dr = 8: Me Me	66 1
17	Me Ph Ph	Me Ts 3q Ph	71
18	Me AcO 1r	Me Ts AcO 3r	87
19	Me 1s	Ts 3s dr = 1:1	79
20	Me Me 1t	Me Me Ts 3t	72





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1 **1b–1i** were all good substrates for the addition reaction (entries 1–8).<sup>23</sup> A wide variety of functional groups 2 including alkoxy, acetyl, carboxy, cyano, free hydroxy, 3 acetoxy, and chloro groups were compatible with the 4 5 reaction conditions, and the corresponding linearly extended 6 alkyl sulfones 3b-3i were obtained in isolated yields 7 ranging from 66% to 85%. Of note was that triethylvinylsilane (1j) and pinacol vinylboronate (1k)<sup>24</sup> 8 9 were also suitable entries although silyl and boryl groups are potentially subject to oxidation with peroxy reagents (entries 10 11 9 and 10). The yields of the reaction of vicinally 12 disubstituted alkenes 11-1n ranged from 22% to 68% 13 (entries 11–14). Geminally disubstituted alkenes **10–1r** were 14 eligible for the reaction with 4 (entries 15-18). 15 Trisubstituted and even tetrasubstituted alkenes 1s-1u successfully participated in the addition reaction (entries 16 17 19–21). It is worth mentioning that the produced alkyl 18 sulfone 3u is difficult to synthesize by conventional 19 methods forming a C–S linkage.

20 The tosyl group was readily removed upon treatment 21 with magnesium turnings in methanol at 50 °C (See the 22 Supporting Information more details). Moreover, the 23 following transformation of the elongated alkyl sulfone 3a 24 demonstrated the synthetic utility of the present one-carbon extension reaction (Scheme 3).<sup>24</sup> Sequential treatment of **3a** 25 26 with *n*-BuLi and ICH<sub>2</sub>MgCl extended the carbon chain by 27 another carbon.<sup>25</sup> In a formal sense, the resulting terminal alkene arises from insertion of an ethylene (-CH<sub>2</sub>CH<sub>2</sub>-) 28 29 group between the alkyl and vinyl groups of the starting 30 terminal alkene. Of note is that the present reaction makes 31 such an extension of a carbon chain available to a 32 hydrocarbon molecule lacking any heteroatom 33 functionalities.



## 34

35 Scheme 3. Synthetic derivatization of 3a achieving formal 36 1,2-hydrovinylation of alkenes.

37 In summary, we have developed a new synthetic method of alkyl sulfones from alkenes and a sulfone-38 39 stabilized phosphonium salt through C–C bond formation. 40 An alkene acts as the main structural backbone, to which a 41 tosylmethyl group is appended in a regioselective fashion. A 42 radical pathway promoted by visible light is compatible

52 Supporting available Information is on 53 http://dx.doi.org/10.1246/cl.\*\*\*\*\*

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43 with a variety of functional groups.

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45 This work was supported by JSPS KAKENHI Grant Numbers 15H05756 (M.M.) [Scientific Research (S)] and 46 20H04816 (T.M.) [Scientific Research on Innovative Areas 47 48 (Hybrid Catalysis)]. We thank Mr. N. Oku (Kyoto University) for his kind help in carrying out additional 49 50 experiments requested by the reviewers. 51

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