Supporting Information For

## **Enantioselective C-C Bond Formation during the Oxidation of 5-Phenylpent-2-enyl Carboxylates with Hypervalent Iodine(III)**

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**Figure S1**. GLC traces of racemic **3a** (top) and optically active **3a** (bottom), Chirasil-DEX-CB ( $25 \text{ m} \times 0.25 \text{ mm} \times 0.25 \mu \text{m}$  film thickness), at column temperature of 170 °C.



**Figure S2**. GLC traces of racemic **3c** (top) and optically active **3c** (bottom), Chirasil-DEX-CB ( $25 \text{ m} \times 0.25 \text{ mm} \times 0.25 \mu \text{m}$  film thickness), at column temperature of 160 °C.

X ray crystallographic data for **3b** have been deposited at the Cambridge Crystallographic Centre: Deposition number: CCDC 1544511. The data were summarized in Table S 1 and Figure S3.

$C_{25}H_{22}O_4$	$V(\text{\AA}^3)$	1950.46(19)	
386.45	Ζ	4	
monoclinic	$D_{\rm calcd}  ({\rm g/cm}^{-3})$	1.316	
<i>P</i> 2 <sub>1</sub> /n	$\mu$ (cm <sup>-1</sup> )	0.88	
14.2906(7)	$2 heta_{ m max}$ (°)	54.9	
5.9928(4)	no. of data collected	4448	
22.8149(12)	no. of parameters	262	
90.000	R	0.0874	
93.389(7)	Rw	0.1455	
90.000			
	$C_{25}H_{22}O_4$ 386.45 monoclinic $P2_1/n$ 14.2906(7) 5.9928(4) 22.8149(12) 90.000 93.389(7) 90.000	$C_{25}H_{22}O_4$ $V(Å^3)$ 386.45 $Z$ monoclinic $D_{calcd} (g/cm^{-3})$ $P2_1/n$ $\mu (cm^{-1})$ 14.2906(7) $2\theta_{max} (^{\circ})$ 5.9928(4)no. of data collected22.8149(12)no. of parameters90.000 $R$ 93.389(7) $Rw$ 90.000 $R$	

Table S1. Crystal data and structure refinement for 3b.



Figure S3. An ORTEP view (50% probability level) of 3b























