Supplementary Material for:

Mechanism of Oxygen Transfer in the Epoxidation of an Olefin by Molecular Oxygen in the Presence of an Aldehyde

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Experimental

General Information: All reactions using KH were performed in oven-dried glassware under nitrogen with freshly distilled solvents. Tetrahydrofuran (THF) was distilled from sodium and benzophenone. The activity of 3-chloro-peroxybenzoic acid and trimethylacylperoxy acid was determined by a starch-indicated titration of liberated iodine by thiosulfate. All other reagents were obtained from commercial sources and used without purification.

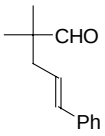
Flash column chromatography was performed using 230-400 mesh silica gel or 150 mesh basic alumina. Merck silica gel plates with QF-254 indicator were used for thin layer chromatography and detection was done by UV light or $KMnO_4$.

^{1}H and ^{13}C NMR spectra were acquired on either a Varian U400 (400 MHz ^{1}H, 100.6 MHz ^{13}C) or U500 (500 MHz ^{1}H, 125.7 MHz ^{13}C). All spectra were recorded in chloroform-*d* ($CDCl_3$). Chemical shifts (δ) are reported in ppm and the following abbreviations are used in peak description: s(singlet), d(doublet), t(triplet), q(quartet), m(multiplet), and b(broadened). Coupling constants (J) are reported in Hz.

Elemental analysis was performed at the University of Illinois Microanalytical Service Laboratory. In cases where microanalytical data was not available, the purity of the compound was assigned greater than 95% by ^{1}H and ^{13}C NMR, with composition confirmed by HRMS. Gas chromatographic data was acquired on an HP5890 with Alltech SE-54 column (30m, 0.25mm ID, 0.25μm film). Unless noted otherwise, all GC data was obtained with the following temperature settings: 100 ºC initial temperature for 2 minutes, temperature ramp to 250 ºC at 20ºC per minute. Mass spectrometric data was obtained by the University of Illinois Mass Spectrometry Center. All melting points were determined by Thomas-Hoover capillary melting point apparatus and reported uncorrected. Isotopic enrichments were calculated by the standard matrix method. ^{18}O-Labelled oxygen gas was purchased from Isotec, Inc.

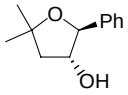
Standard workup for reactions includes dilution with Et_2O and H_2O, separation of phases, $MgSO_4$ drying of the organic phase, filtering, and concentration by rotary evaporation.

Representative oxygen transfer reaction: To a stirring solution of aldehyde (0.80 mmol) and olefin (0.70 mmol) in 5 mL of acetonitrile which had been flushed and filled with O_2 at room temperature, 5 μL of initiator (5 mol %) is added and stirring continued under 1 atmosphere of oxygen for 18 h. The crude reaction mixture is then washed with 20 % aqueous sodium bisulfite solution. Standard workup procedure provided the oxidized olefins, all of which were identified through GC, TLC, and ^{1}H NMR comparisons to commercially available standards.



4

2,2-Dimethyl-5-phenyl-4-pentenal (**4**)[1] Following procedure of Groenewegen et al.,[2] 1.1 eq. of KH suspension (1.75 g, 35 % by weight, 15 mmol) was washed with 2 X 10 mL of hexane to remove mineral oil, and the resultant KH was stirred in 25 mL of freshly distilled THF under N_2 for 15 min at room temperature. To this mixture under N_2, 1 eq. of isobutyraldehyde (1.26 mL, 14 mmol) dissolved in 5 mL of THF was added dropwise over 5 min, producing first a bright lemon-yellow opaque solution which turned a pale straw color after 15 min of stirring. After addition of 1 eq *trans*-cinnamyl bromide (2.12 mL, 14 mmol) in 5 mL of THF, the reaction was stirred for 1 h, producing a white opaque suspension. Careful addition of 10 mL of H_2O and standard workup procedure afforded 3.5 g crude product as a yellow oil. The product was purified by silica gel flash chromatography (10:1 Pet Eth : EtOAc, R_f = 0.80) to provide **4** (1.64 g, 61 %) as a clear, colorless oil. GC: R_t = 7.34 minutes; ^{1}H NMR ($CDCl_3$, 400MHz): δ 1.12 (s, 6H, C$\underline{H}_3$), 2.28 (dd, 2H, J = 7.5, 1.3, C$\underline{H}_2$), 6.11 (dt, 1H, J = 15.8, 7.5, PhCHC$\underline{H}$), 6.42 (d, 1H, J = 15.7, PhC$\underline{H}$), 7.3 (m, 5H, Ar$\underline{H}$), 9.54 (s, 1H, C(O)$\underline{H}$); ^{13}C NMR ($CDCl_3$, 125MHz): δ 21.4 ($\underline{C}H_3$), 40.6 ($\underline{C}H_2$), 46.3 ($\underline{C}(CH_3)_2$), 124.9 (PhCH$\underline{C}$H), 126.2 (Ph$\underline{C}$H), 127.4 (Ar$\underline{C}$H),128.6 (Ar$\underline{C}$H), 133.6 (Ar$\underline{C}$H), 137.2 (Ar$\underline{C}_q$), 205.9 ($\underline{C}$(O)H); HRMS (HR EI, 70 eV): calc: 188.120115, found: 188.119449.

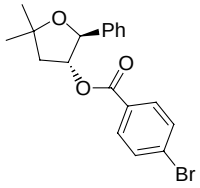


6

5,5-Dimethyl-2-phenyltetrahydrofuran-3-ol (**6**) A 3-necked round-bottomed flask was equipped with a septum, connected to a Schlenk vacuum line, and a Hg-filled gas transfer apparatus. The system was evacuated and filled with N_2 three times, before being evacuated and filled with O_2 to a pressure just greater than 1 atm. The eneal **4** (230.6 mg, 1.2 mmol) in 8 mL of acetonitrile was injected and stirred for 15 min, followed by 25 μL (ca. 10 mol %) *t*-butyl hydroperoxide. Stirring was continued for 18 h at room temperature. Dilution with Et_2O was followed by 1 X 10 mL wash with 20 % aqueous

[1] Armesto, D.; Austin, M. A.; Griffiths, O. J.; Horspool, W. M.; Carpintero, M. *Chem. Commun.* **1996**, 2715.

$NaHSO_3$ and standard workup procedure. Silica gel flash chromatography (3:1 Pet Eth : EtOAc, R_f = 0.30) afforded **6** (206 mg, 87 %). Recrystallization from hexane provided the product as thin white needles. Mp = 44 ºC; GC: R_f = 4.70 min; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.38 (s, 3H, C<u>H</u>$_3$), 1.43 (s, 3H, C<u>H</u>$_3$), 1.88 (dd, 1H, J = 12.8, 7.1, <u>H</u>CH), 2.12 (bs, 1H, O<u>H</u>), 2.17 (dd, 1H, J = 12.6, 7.5, HC<u>H</u>), 4.12 (q, 1H, J = 7, C<u>H</u>OH), 4.65 (d, 1H, J = 6.8, C<u>H</u>Ph), 7.25-7.38 (m, 5H, Ar<u>H</u>); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 29.0 (<u>C</u>H$_3$), 29.9 (<u>C</u>H$_3$), 47.1 (<u>C</u>H$_2$), 79.2 (PhCH<u>C</u>H), 80.1 (<u>C</u>(CH$_3$)$_2$), 86.0 (Ph<u>C</u>H), 126.0 (Ar<u>C</u>H), 127.8 (Ar<u>C</u>H), 128.5 (Ar<u>C</u>H), 140.7 (Ar<u>C</u>$_q$); HRMS (HR CI): (M^+-1) calc: 191.107205, found: 191.107200.



7

3-*para*-Bromobenzoyl-5,5-dimethyl-2-phenyltetrahydrofuran (**7**) To the alcohol **6** (66.2 mg, 0.34 mmol, 1 eq) and *p*-bromobenzoyl chloride (80.0 mg, 0.36 mmol, 1.05 eq) stirring at room temperature in 5 mL of CH_2Cl_2, Et_3N (50 µL, 0.36 mmol, 1.05 eq) and DMAP (5.1 mg, 0.04 mmol, 0.1 eq.) were added. The reaction mixture was stirred at room temperature for 18 h. The crude reaction mixture was washed 1 X 10 mL with 5 % HCl, 3 X 10 mL with saturated aqueous $NaHCO_3$, and standard workup procedures to afford **7** (101 mg, 79 %). Slow solvent evaporation method of recrystallization was used to provide a crystal suitable for X-ray crystallographic analysis. GC: R_f = 9.81 min; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.51 (s, 3H, C<u>H</u>$_3$), 1.52 (s, 3H, C<u>H</u>$_3$), 2.08 (dd, 1H, J = 13.7, 3.2, <u>H</u>CH), 2.34 (dd, 1H, J = 13.9, 7.1, HC<u>H</u>), 5.19 (d, 1H, J = 3.9, PhC<u>H</u>), 5.37 (dt, 1H, J = 7.1, 3.4, C<u>H</u>OCO), 7.26-7.49 (m, 5H, Ar<u>H</u>), 7.59-7.63 (m, 2H, BrAr<u>H</u>), 7.90-7.94 (m, 2H, BrAr<u>H</u>); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 28.2 (<u>C</u>H$_3$), 29.6 (<u>C</u>H$_3$), 44.1 (<u>C</u>H$_2$), 81.9 (<u>C</u>$_q$(CH$_3$)$_2$), 82.6 (ArCO$_2$<u>C</u>H), 84.6 (Ph<u>C</u>H), 125.8 (Ar<u>C</u>H), 127.7 (Ar<u>C</u>H), 128.3 (Ar<u>C</u>$_q$Br), 128.4 (Ar<u>C</u>H), 129.9 (BrAr<u>C</u>$_q$), 131.1 (Ar<u>C</u>H), 131.8 (Ar<u>C</u>H), 140.4 (Ar<u>C</u>$_q$), 165.2 (<u>C</u>=O); HRMS (HR CI): (M^+-2) calc: 373.043931, found: 373.044035.

Details of crystallographic investigation may be obtained from the Cambridge Crystallographic Database (deposit@ccdc.cam.ac.uk): Registry Number 137495.

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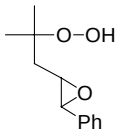
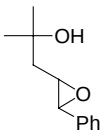
[2] Groenewegen, P.; Kallenberg, H.; van der Gen, A. *Tetrahedron Letters* **1978**, *5*, 491.



8

2-Hydroperoxy-2-methyl-5-phenyl-3,4-epoxy-pentene (**8**) This compound was isolated as an intermediate in the formation of **6**, produced by the same procedure without any workup, and tentatively assigned. ^{1}H NMR ($CDCl_3$, 400MHz): δ 1.32 (s, 3H, C$\underline{H}_3$), 1.37 (s, 3H, C$\underline{H}_3$), 1.89 (dd, 1H, J = 14.8, 8.6, $\underline{H}$CH), 2.10 (dd, 1H, J = 14.8, 3.1, HC$\underline{H}$), 3.14 (dt, 1H, J = 8.4, 2.4, PhCH(O)C$\underline{H}$), 3.68 (d, 1H, J = 2.0, PhC$\underline{H}$), 7.27-7.39 (m, 5H, Ar$\underline{H}$), 8.26 (s, 1H, OO$\underline{H}$).



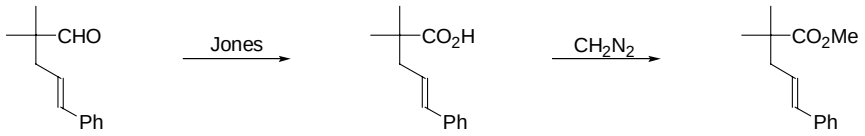
9

18

4-Methyl-1,2-epoxy-1-phenyl-4-pentanol (**9**) and **Triphenylphosphine oxide** (**18**) Following the procedure for the synthesis of **6**, aldehyde **4** (94.3 mg, 0.50 mmol) was stirred under O_2 for 18 h. Triphenylphosphine (137 mg, 0.52 mmol, 1.04 eq) was added and the reaction mixture was stirred for 3 h. Normal workup followed by silica gel flash chromatography (3:1 Pet Eth : EtOAc to pure EtOAc) provided **9** (51 mg, 53 %, R_f = 0.25) as a colorless oil, and **18** (100 mg, 72 %, R_f = 0.08) as a white solid. **9**: ^{1}H NMR ($CDCl_3$, 500MHz): δ 1.34 (s, 3H, C$\underline{H}_3$), 1.35 (s, 3H, C$\underline{H}_3$), 1.78 (dd, 1H, J = 14.2, 7.1, $\underline{H}$CH), 1.91 (dd, 1H, J = 14.2, 4.6, HC$\underline{H}$), 2.13 (bs, 1H, O$\underline{H}$), 3.15 (ddd, 1H, J = 6.8, 4.6, 2.0, PhCH(O)C$\underline{H}$), 3.62 (d, 1H, J = 2.0, PhC$\underline{H}$), 7.25-7.36 (m, 5H, Ar$\underline{H}$); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 29.5 (C$\underline{H}_3$), 29.8 (C$\underline{H}_3$), 45.6 (C$\underline{H}_2$), 58.3 (PhCH(O)C$\underline{H}$), 60.1 (PhC$\underline{H}$), 70.7 ($\underline{C}_q(CH_3)_2$), 125.5 (ArC$\underline{H}$), 128.1 (ArC$\underline{H}$), 128.4 (ArC$\underline{H}$), 137.3 (Ar$\underline{C}_q$); HRMS (HR EI, 70 eV): calc: 192.115030, found: 192.115000. **18**: This compound was chromatographically and spectroscopically identical to a standard sample. ^{1}H NMR ($CDCl_3$, 500MHz): δ 7.42 (td, 2H, J = 7.8, 2.7, ArC$\underline{H}$), 7.50 (td, 1H, J = 7.6, 1.5, ArC$\underline{H}$), 7.65 (m, 2H, ArC$\underline{H}$).

Labeled Synthesis of 9 and 18. The same procedure for the synthesis of **9** and **18** was used, stirring **4** (92.7 mg, 0.49 mmol) under a 50/50 mixture of ^{18}O–^{18}O and ^{16}O–^{16}O for 40 h. Triphenylphoshine (138 mg, 0.53 mmol, 1.08 eq) was added and stirred another 2 h. Silica gel flash

chromatography provided **9** (43.4 mg, 46 %) and **18** (108 mg, 79 %). These products were analyzed by FI/MS and FD/MS respectively.



4 **22** 75% **16** 55%

2,2-Dimethyl-5-phenyl-4-pentenoic Acid (**22**)[3] Excess Jones reagent was added dropwise to a stirring solution of **4** (372 mg, 2 mmol) in 5 mL of acetone at 0 °C. After 30 min, 10 more drops of Jones reagent were added. After stirring for 1 h under N_2, the mixture was warmed to room temperature and stirred for an additional 2 h. Excess isopropanol was added to quench residual Jones reagent. After stirring for 1 h, the mixture was diluted with H_2O and Et_2O, and normal workup procedure was followed, including a 1 X 10 mL 2 % HCl wash, to afford **22** (304 mg, 75 %) as a white solid to be used without further purification. This compound possessed identical spectroscopic characteristics as reported values.[3] Mp = 58-60 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.23 (s, 6H, C$\underline{H}_3$), 2.48 (dd, 2H, J = 7.6, 1.2, C$\underline{H}_2$), 6.20 (dt, 1H, J = 15.9, 7.3, PhChC$\underline{H}$), 6.45 (d, 1H, J = 15.6, PhC$\underline{H}$), 7.28-7.40 (m, 5H, Ar$\underline{H}$), 9.0 (bs, 1H, O$\underline{H}$); ^{13}C NMR ($CDCl_3$, 125MHz): δ 24.7 ($\underline{C}H_3$), 42.6 ($\underline{C}(CH_3)_2$), 43.5 ($\underline{C}H_2$), 125.6 (PhCH$\underline{C}$H), 126.1 (Ar$\underline{C}$H), 127.2 (Ph$\underline{C}$H), 128.4 (Ar$\underline{C}$H), 133.3 (Ar$\underline{C}$H), 137.3 (Ar$\underline{C}_q$), 184.4 ($\underline{C}$(O)H).

Methyl 2,2-dimethyl-5-phenyl-4-pentenoate (**16**)[4] Using an Aldrich Diazomethane Generator, Diazald® (1.356 g, 6 mmol, 4.6 eq) was added dropwise to a 1:1 mixture of EtOH / 35 % KOH at 60 °C. Fresh CH_2N_2 was distilled into a solution of **22** (269 mg, 1.3 mmol) in Et_2O. Three hours after the addition was complete, glacial acetic acid was added to the reaction mixture. The entire apparatus was washed with acetic acid as well and allowed to sit for 1 h. After solvent evaporation, silica gel flash chromatography (10:1 Pet Eth : EtOAc, R_f = 0.69) provided **16** (156 mg, 55 %) as a yellow oil. This compound possessed identical spectroscopic characteristics as reported values.[4] ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.25 (s, 6H, C$\underline{H}_3$), 2.45 (dd, 2H, J = 7.6, 1.2, C$\underline{H}_2$), 3.70 (s, 3H, OC$\underline{H}_3$), 6.16 (dt, 1H, J = 15.9, 7.6, PhCHC$\underline{H}$), 6.42 (d, 1H, J = 15.9, PhC$\underline{H}$), 7.20-7.38 (m, 5H, Ar$\underline{H}$). ^{13}C NMR ($CDCl_3$, 125MHz): δ 24.9 ($\underline{C}H_3$), 42.8 ($\underline{C}_q(CH_3)_2$), 43.9 ($\underline{C}H_2$), 51.8 (O$\underline{C}H_3$), 126.0 (PhCH$\underline{C}$H), 126.1 (Ar$\underline{C}$H), 127.1 (Ph$\underline{C}$H), 128.5 (Ar$\underline{C}$H), 133.0 (Ar$\underline{C}$H), 137.4 (Ar$\underline{C}_q$), 178.0 ($\underline{C}O_2$Me); HRMS (HR EI, 70 eV): calc: 218.130680, found: 218.131484.

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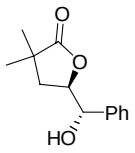
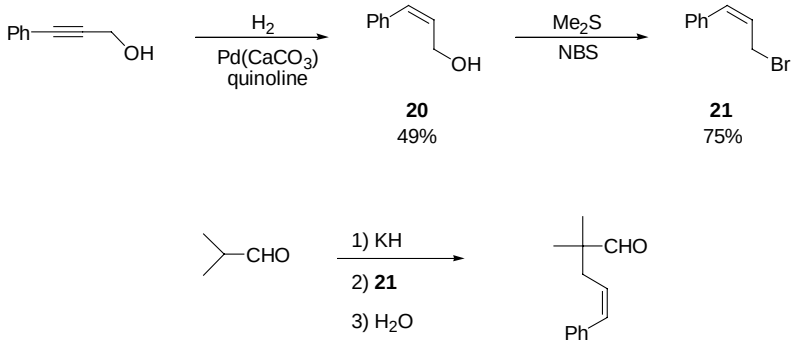
[3] Kulenovic, S. T.; Arnold, R. T. *J. Org. Chem.* **1980**, *45*, 891.



17

α,α-Dimethyl-γ-(hydroxyphenylmethyl)valerolactone (**17**) Following the normal O_2 transfer reaction conditions, trimethylacetaldehyde (33 mg, 0.38 mmol, 1.65 eq) was reacted with **16** (49.5 mg, 0.23 mmol) for 18 h. As determined by ^{1}H NMR and GC, **17** was provided in 94% conversion, based on remaining **16**. Following an aqueous $NaHSO_3$ wash, normal workup, and recrystallization by vapor fusion method from Et_2O / pentane, **17** (6 mg, 12 %) was isolated as white crystals suitable for X-ray crystallography. Mp = 133-134 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.20 (s, 3H, C$\underline{H}_3$), 1.27 (s, 3H, C$\underline{H}_3$), 1.69 (dd, 1H, J = 12.8, 6.4, $\underline{H}$CH), 2.22 (dd, 1H, J = 12.8, 9.9, HC$\underline{H}$), 2.34 (d, 1H, J = 3.1, O$\underline{H}$), 4.61 (ddd, 1H, J = 9.9, 6.4, 3.7, γ-$\underline{H}$), 5.14 (t, 1H, J = 3.5, PhC$\underline{H}$), 7.28-7.35 (m, 5H, Ar$\underline{H}$). ^{13}C NMR ($CDCl_3$, 125MHz): δ 24.5 ($\underline{C}H_3$), 24.8 ($\underline{C}H_3$), 35.4 ($\underline{C}H_2$), 40.1 ($\underline{C}_q(CH_3)_2$), 72.2 (γ-$\underline{C}$H), 79.7 (Ph$\underline{C}$H), 125.9 (Ar$\underline{C}$H), 127.9 (Ar$\underline{C}$H), 128.4 (Ar$\underline{C}$H), 138.2 (Ar$\underline{C}_q$), 181.9 ($\underline{C}$=O); HRMS (HR EI, 70 eV): calc: 220.109945, found: 220.109900.

Details of crystallographic investigation may be obtained from the Cambridge Crystallographic Database (**deposit@ccdc.cam.ac.uk**): Registry Number 137494.



[4] Kulenovic, S. T.; Arnold, R. T. *Croatica Chemica Acta* **1979**, *52*, 299.

3-Phenyl-2(*Z*)-propen-1-ol (**20**)[5,6] Following the procedure by Weisenburger,[5] Pd($CaCO_3$) (237 mg) was pre-hydrogenated under 1 atm of H_2 in 2 mL of benzene for 30 min. Distilled quinoline (0.18 mL, 1.5 mmol) was then added, followed by 3-phenyl-2-propyn-1-ol (0.40 mL, 3.04 mmol). The heterogeneous mixture was stirred under 1 atm of H_2 for 5 h. The crude reaction mixture was filtered through a plug of silica gel, followed by 3 X 10 mL extractions by 1 N HCl and standard workup, to provide the crude product as a yellow oil. The compound was purified by silica gel flash column chromatography (3:1 Pet Eth : EtOAc, R_f = 0.26) to afford 0.204 g (49 %) of **20**. GC: R_f = 4.83. This compound possessed identical spectroscopic characteristics as reported values.[5,6] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.98 (br s, 1H, O<u>H</u>), 4.43 (d, 2H, J = 6.4, C<u>H</u>$_2$), 5.87 (dt, 1H, J = 12.1, 6.3, PhCHC<u>H</u>), 6.54 (d, 1H, J = 11.9, PhC<u>H</u>), 7.2-7.5 (m, 5H, Ar<u>H</u>); HRMS (HR EI, 70 eV): calc: 134.073165, found: 134.073634.

1-Bromo-3-phenyl-2(*Z*)-propene (**21**)[5,7] Following the Corey-Kim bromination procedure of Weisenburger,[5] Me_2S (200 μL, 2.7 mmol) was added dropwise to a mixture of NBS (404 mg, 2.3 mmol) in 5 ml of CH_2Cl_2 at 0 ºC. The mixture was cooled to –20 ºC and the alcohol **20** (203 mg, 1.5 mmol) was added in 2 mL of CH_2Cl_2. The mixture was warmed to 0 ºC and stirred for 2 h, and then warmed to room temperature and stirred for another 1 h. Standard workup provided the crude product as a yellow oil. The compound was purified by silica gel flash column chromatography (15:1 Pet Eth : EtOAc, R_f = 0.65) to afford **21** (223 mg, 75 %). GC: R_f = 5.41. This compound possessed identical spectroscopic characteristics as reported values. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.17 (d, 2H, J = 8.6, C<u>H</u>$_2$), 6.00 (dt, 1H, J = 11.3, 8.6, PhCHC<u>H</u>), 6.61 (d, 1H, J = 11.2, PhC<u>H</u>), 7.25-7.42 (m, 5H, Ar<u>H</u>); HRMS (HR EI, 70 eV): (M^+ -1) calc: 194.980936, found: 194.981045.

2,2-Dimethyl-5-phenyl-4(*Z*)-pentenal (**19**) A procedure similar to that for the synthesis of **4** was used. Isobutyraldehyde (250 μL, 2.8 mmol), KH (410 mg, 35% by weight, 3.6 mmol), and **21** (550 mg, 2.8 mmol) were used. The reaction was stopped one hour after the addition of **21** to prevent olefin isomerization. The aldehyde was purified by silica gel flash chromatography (10:1 Pet Eth : EtOAc, R_f = 0.65) as needed and used immediately thereafter. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.08 (s, 6H, C<u>H</u>$_3$), 2.52 (dd, 2H, J = 7.3, 1.5, C<u>H</u>$_2$), 5.59 (dt, 1H, J = 11.7, 7.3, PhCHC<u>H</u>), 6.57 (d, 1H, J = 11.7, PhC<u>H</u>), 7.25-7.36 (m, 5H, Ar<u>H</u>), 9.46 (s, 1H, C(O)<u>H</u>). ^{13}C NMR ($CDCl_3$, 125MHz): δ 21.2 (<u>C</u>H$_3$), 35.4 (<u>C</u>H$_2$), 46.2 (<u>C</u>(CH$_3$)$_2$), 126.8 (PhCH<u>C</u>H), 128.2 (Ph<u>C</u>H), 128.3 (Ar<u>C</u>H),128.9 (Ar<u>C</u>H), 131.7 (Ar<u>C</u>H), 137.2 (Ar<u>C</u>$_q$), 205.9 (<u>C</u>(O)H).

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[5] Weisenburger, G.; University of Illinois: Urbana, IL, **1998**.
[6] Takeuchi, R.; Kashio, M. *J. Am. Chem. Soc.* **1998**, *120*, 8647.
[7] Corey, E. J.; Kim, C. U.; Takeda, M. *Tetrahedron Lett.* **1972**, *42*, 4339.