# **Supporting Information**

## A General Gold-Catalyzed Direct Oxidative Coupling of Non-activated Arenes

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Table S1.	Catalyst	screening	ofox	idative	homo-c	oupling	of <i>p</i> -x	ylene <sup>[a]</sup>

		ме		ŗ	ve Me	
			2 mol% cat	talyst 📈		
		() + Ox	Solvent 55 °	$2C_{17h}$		
		Me		0, 11 II	Mo	
entry	catalyst	solvent	ovidant	vield [%] <sup>[b]</sup>		TOF $[h^{-1}]^{[d]}$
1				74	27	<u>101 [ll ]</u>
1	Au(OAa)	HOAC	$PIII(OAC)_2$ PhI(OAc)	20	20	2.2
2	$Au(OAC)_3$	ПОАС	$PIII(OAC)_2$	39	20	1.1
5	AUCN	HOAC	$Phi(OAc)_2$	0	0	0
4	AuCl(PPh <sub>3</sub> )	HOAC	$PhI(OAc)_2$	76	38	2.2
5	nıl	HOAc	$Phl(OAc)_2$	0	0	0
6 <sup>[e]</sup>	FeCl <sub>3</sub>	HOAc	$PhI(OAc)_2$	0	0	0
7	NH <sub>4</sub> FeCl <sub>4</sub>	HOAc	$PhI(OAc)_2$	0	0	0
8	AgNO <sub>3</sub>	HOAc	$PhI(OAc)_2$	0	0	0
9	CuCl <sub>2</sub>	HOAc	$PhI(OAc)_2$	0	0	0
10	BF <sub>3</sub> •OEt <sub>2</sub>	HOAc	PhI(OAc) <sub>2</sub>	0	0	0
11 <sup>[e]</sup>	HAuCl <sub>4</sub>	<i>p</i> -xylene	PhI(OAc) <sub>2</sub>	58	29	1.7
$12^{[e]}$	HAuCl <sub>4</sub>	CH <sub>3</sub> CN	PhI(OAc) <sub>2</sub>	0	0	0
13 <sup>[e]</sup>	HAuCl <sub>4</sub>	CH <sub>3</sub> NO <sub>2</sub>	PhI(OAc) <sub>2</sub>	12	6	0.4
$14^{[e]}$	HAuCl <sub>4</sub>	ClCH <sub>2</sub> CH <sub>2</sub> Cl	PhI(OAc) <sub>2</sub>	42	21	1.2
15	HAuCl <sub>4</sub>	TFA	PhI(OAc) <sub>2</sub>	>1	>1	>0.1
16 <sup>[</sup>	HAuCl <sub>4</sub>	$Ac_2O$	PhI(OAc) <sub>2</sub>	71	36	2.1
17	HAuCl <sub>4</sub>	HOAc	PhI(OCOCF <sub>3</sub> ) <sub>2</sub>	69	35	2.1
18	HAuCl <sub>4</sub>	HOAc	$K_2S_2O_8$	0	0	0
19	HAuCl <sub>4</sub>	HOAc	Oxone®	0	0	0
20	HAuCl <sub>4</sub>	HOAc	35% CH <sub>3</sub> CO <sub>3</sub> H	0	0	0
21	HAuCl <sub>4</sub>	HOAc	$Cu(OAc)_2$	0	0	0
22	HAuCl <sub>4</sub>	HOAc	45% IBX	1	>1	>0.1

[a] Reaction conditions: Oxidant (1.0 mmol), *p*-xylene (10.0 mmol), dodecane (55  $\mu$ L, internal standard) and catalyst (0.02 mmol, 2.0 mol%) were heated in the appropriate solvent (1.0 mL) at 55 °C in air for 17 h. [b] Calibrated GC yields were reported; % Yield = (no. of moles of biaryl)/(no. of moles of oxidant) x 100%. [c] Turnover number (TON) = (no. of moles of biaryl produced)/(no. of moles of catalyst). [d] Turnover frequency (TOF) = (no. of moles of biaryl produced)/[(no. of moles of catalyst)]. [e] 2 mL of solvent was used.

**General:** NMR spectra were recorded on a Bruker ARX 300 spectrometer, operating at 300 MHz for <sup>1</sup>H NMR, 75 MHz for <sup>13</sup>C NMR were reported downfield from CDCl<sub>3</sub> ( $\delta$ : 7.27 ppm) for <sup>1</sup>H NMR. For <sup>13</sup>C NMR, chemical shifts were reported in the scale relative to the solvent of CDCl<sub>3</sub> ( $\delta$ : 77.0 ppm) used as an internal reference. Mass spectra were in general recorded on an AMD 402/3 or a HP 5989A mass selective detector. Column chromatographies were performed with silica gel Fluka 60 (70-230 mesh ASTM).

General Procedure for HAuCl<sub>4</sub> Catalyzed Homo-coupling of Arenes: To a 10 mL vial arene (10 mmol),  $PhI(OAc)_2$  (1 mmol),  $HAuCl_4$  (0.02 mmol) and acetic acid (1 mL) were added. The mixture was stirred for 17 h at 55 °C and then quenched with water (10 mL). The reaction mixture was extracted with EtOAc (3 x 10 mL) and the combined organic layer was washed

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with saturated NaHCO<sub>3</sub> (2 x 20 mL), brine (10 ml), dried over  $Na_2SO_4$ , filtered and concentrated. The residue was purified by column chromatography to afford the desired product.



**Table 1; Entry 1**<sup>1</sup>

 $R_f = 0.63$  (Hexane); <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 2.04$  (s, 6 H), 2.35 (s, 6 H), 6.90-7.00 (m, 2 H), 7.05-7.2 (m, 4 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 19.30$ , 20.93, 127.70, 129.59, 129.94, 132.59, 134.82, 141.58; ATR-IR (cm<sup>-1</sup>): 3017m, 2917m, 2859s, 1610m, 1501s, 1446w, 1376s, 1145s, 1037w, 890s, 882s, 812s, 754s, 7646s, 475m; MS (EI): *m/z* (rel. int.) 211 (11), 210 (66), 196 (15), 195 (100), 180 (33), 179 (23), 178 (21), 165 (31); HRMS calcd. for C<sub>16</sub>H<sub>18</sub> *m/z* 210.14030, found m/z 210.13994.



Table 1; Entry 4<sup>2</sup>

 $R_f = 0.60$  (Hexane); colorless oil; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 2.05$  (s, 6 H), 2.39 (s, 6 H), 6.90-7.20 (m, 6 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 19.79$ , 21.11, 126.19, 129.43, 130.52, 135.80, 136.51, 138.63; ATR-IR (cm<sup>-1</sup>): 3013w, 2919w, 2858w, 1612s, 1487s, 1443w, 1377s, 1233s, 1035w, 1007s, 874s, 815s, 768s, 724s; MS (EI): m/z (rel. int.) 211 (15), 210 (90), 209 (7), 195 (100), 180 (39), 179 (27), 178 (22), 165 (35), 89 (11); HRMS calcd. for C<sub>16</sub>H<sub>18</sub> m/z 210.14030, found m/z 210.14022.



Table 1; Entry 5<sup>3</sup>

 $R_f = 0.64$  (Hexane); colorless oil; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 1.34$  (s, 18 H), 2.05 (s, 3 H), 7.18-7.25 (m, 4 H), 7.28-7.33 (m, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 19.37$ , 31.45, 34.41, 123.83, 126.64, 129.44, 132.78, 141.50, 148.30; ATR-IR (cm<sup>-1</sup>): 2960w, 2867s, 1607s, 1491s, 1463m, 1391w, 1260m, 1202s, 1151m, 1113s, 894s, 847s, 818s, 738m, 682m; MS (EI): *m/z* (rel. int.) 295 (5), 294 (22), 280 (23), 279 (100), 57 (11); HRMS calcd. for C<sub>22</sub>H<sub>30</sub> *m/z* 294.23420, found m/z 294.23429.



 $R_f = 0.14$  (Hexane:ethyl acetate = 100:3); white solid; mp = 123-124 °C (hexane); <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.73$  (s, 6 H), 6.88 (dd, J = 8.9, 4.6 Hz, 2 H), 6.96 (dd, J = 8.9, 2.8 Hz, 2 H), 7.00 (ddd, J = 8.8, 8.0, 3.2 Hz, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 56.29, 112.08$  (d,  ${}^{3}J_{CF} = 8.4$  Hz), 114.81 (d,  ${}^{2}J_{CF} = 22.6$  Hz), 118.11 (d,  ${}^{2}J_{CF} = 23.2$  Hz), 127.77 (dd,  $J_{CF} = 8.0, 1.6$ 

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Hz), 153.034 (d,  ${}^{4}J_{CF} = 1.9$  Hz), 156.64 (d,  ${}^{1}J_{CF} = 238.2$  Hz); ATR-IR (cm<sup>-1</sup>): 3126w, 3072w, 3020w, 2958w, 2940w, 2910w, 2837w, 1869w, 1592w, 1506m, 1486s, 1464m, 1441m, 1425m, 1406w, 1293m, 1271m, 1256m, 1245s, 1219m, 1180s, 1158s, 1136m, 1035s, 1023s, 947m, 937w, 868s, 844m, 818s, 749s, 722s, 696m; MS (EI): m/z (rel. int.) 251 (14), 250 (100), 235 (24), 220 (40), 204 (32), 164 (10); HRMS calcd. for  $C_{14}H_{12}F_2O_2$  m/z 250.07999, found m/z 250.079793; elemental analysis calcd. for C<sub>14</sub>H<sub>12</sub>F<sub>2</sub>O<sub>2</sub> C 67.20 H 4.83, found C 67.02 H 4.68.



#### Table 1; Entry 7 (Minor isomer)

 $R_f = 0.21$  (Hexane); colorless oil; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.72$  (d, <sup>5</sup> $J_{\rm HF} = 1.5$  Hz, 3 H), 3.92 (s, 3 H), 6.97-67.10 (m, 4 H), 7.24 (ddd, J = 8.4, 2.1, 1.1 Hz, 1 H), 7.30 (dd, J = 12.5, 2.2 Hz, 1 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 56.22, 61.17$  (d,  ${}^{4}J_{CF} = 4.4$  Hz), 113.01 (d,  ${}^{4}J_{CF} = 1.9$  Hz), 115.86 (d,  ${}^{2}J_{CF} = 19.4$  Hz), 116.99 (d,  ${}^{2}J_{CF} = 19.4$  Hz), 123.76 (d,  ${}^{3}J_{CF} = 8.4$  Hz), 124.96 (d,  ${}^{3}J_{CF}$  = 3.7 Hz), 125.48 (d,  ${}^{4}J_{CF}$  = 2.6 Hz), 130.22 (dd,  $J_{CF}$  = 6.8, 3.0 Hz), 135.11 (unresolved dd), 145.04 (d,  ${}^{2}J_{CF}$  = 10.8 Hz), 147.06 (d,  ${}^{2}J_{CF} = 10.8$  Hz), 151.93 (d,  ${}^{1}J_{CF} = 245.2$  Hz), 156.07 (d,  ${}^{1}J_{CF} = 246.3$  Hz); MS (EI): m/z (rel. int.) 251 (17), 250 (100), 235 (33), 220 (34), 204 (39), 175 (12), 164 (24); HRMS calcd. for C14H12F2O2 m/z 250.07999, found m/z 250.79764.



Table 1; Entry 7 (Major isomer)<sup>5</sup>

 $R_f = 0.11$  (Hexane); white solid; mp = 155-156 °C (hexane); <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.85$  (s, 6 H), 6.90-6.95 (m, 2 H), 7.13-7.21 (m, 4 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta$  = 56.33, 113.67 (d, <sup>4</sup>J<sub>CF</sub> = 2.6 Hz), 114.37 (d, <sup>2</sup>J<sub>CF</sub> = 19.3 Hz), 122.17 (d,  ${}^{3}J_{CF} = 3.3 \text{ Hz}$ ), 132.94 (dd,  $J_{CF} = 6.5$ , 2.0 Hz), 146.94 (d,  ${}^{2}J_{CF} = 10.3 \text{ Hz}$ ), 152.54 (d,  ${}^{1}J_{CF} = 245.9 \text{ Hz}$ ); ATR-IR (cm<sup>-1</sup>): 3051w, 3030w, 2977w, 2950w, 2922w, 2845w, 2582w, 2031w,1618m, 1575m, 1499s, 1463s, 1440s, 1404m, 1303s, 1260s, 1209s, 1179s, 1133s, 1043s, 1013s, 863s, 839s, 800s, 760s; MS (EI): m/z (rel. int.) 251 (13), 250 (73), 236 (15), 235 (100), 207 (14), 192 (17), 164 (17); HRMS calcd. for C<sub>14</sub>H<sub>12</sub>F<sub>2</sub>O<sub>2</sub> m/z 250.07999, found m/z 250.080037; elemental analysis calcd. for C<sub>14</sub>H<sub>12</sub>F<sub>2</sub>O<sub>2</sub> C 67.20 H 4.83, found C 67.64, H 4.67.



Table 1; Entry 8<sup>4,6</sup>

 $R_f = 0.29$  (Ethyl Acetate: Hexane = 1:19); white solid; ; mp = 109-110 °C; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.77$  (s, 6 H), 6.90 (d, J = 8.6 Hz, 2 H), 7.21 (d, J = 2.8 Hz, 2 H), 7.30 (dd, J = 8.6, 2.8, Hz, 2 H); m/z (rel. int.) = 286 (11), 285 (9), 284 (6), 283 (15), 282 (100), 232 (49), 217 (19), 189 (11); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 55.97$ , 112.23, 125.18, 127.94, 128.62, 131.01, 155.56; ATR-IR (cm<sup>-1</sup>): 3004m, 2934m, 2835s, 1766w, 1729w, 1590,1499w, 1487w, 1469m, 1443m, 1288w, 1271m, 1242b, 1230m, 1181s, 1147s, 1134s, 1096s, 1021s, 889s, 878, 865s, 810, 753s, 727m, 654s; HRMS calcd. for C<sub>14</sub>H<sub>12</sub>Cl<sub>2</sub>O<sub>2</sub> m/z 282.02089, found m/z 282.02140.

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 $R_f = 0.28$  (Ethyl acetate: hexane = 1:19); white solid; ; mp = 105-107 °C; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.78$  (s, 6 H), 6.85 (d, J = 8.8 Hz, 2 H), 7.34 (d, J = 2.6, Hz, 2 H), 7.44 (dd, J = 8.8, 2.6, Hz, 2 H); m/z (rel. int.) = 374 (49), 373 (16), 372 (100), 371 (9), 370 (51), 278 (41), 263 (25), 261 (27), 235 (16), 233 (15), 139 (20), 138 (12), 126 (27), 63 (12); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 55.91$ , 112.48, 112.71, 128.35, 131.62, 133.77, 156.09; ATR-IR (cm<sup>-1</sup>): 3077s, 3017m, 2936w, 2835w, 1599w, 1584w, 1496w, 1479w, 1458m, 1435s, 1410w, 1377s, 1289s, 1257w, 1242w, 1221w, 1180w, 1148s, 1138s, 1082s, 1029w, 1018w, 883s, 868w, 842w, 815w, 806w, 742s, 724w, 670w; HRMS calcd. for C<sub>14</sub>H<sub>12</sub>Br<sub>2</sub>O<sub>2</sub> m/z 369.91986, found m/z 369.91955.



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MeO

Table 1; Entry 10 (Major isomer)<sup>4,8</sup>

 $R_f = 0.20$  (Ethyl acetate: hexane = 1:19); white solid; mp = 150-152 °C; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.92$  (s, 6 H), 6.87 (d, J = 8.6 Hz, 2 H), 7.46 (d, J = 8.6, 2.3, Hz, 2 H), 7.98 (d, J = 2.3, Hz, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 55.47$ , 87.41, 110.96, 127.73, 133.90, 137.57, 157.45; ATR-IR (cm<sup>-1</sup>): 2963s, 2933m, 2833m, 1594s, 1565s, 1549s, 1474w, 1434m, 1404s, 1375w, 1269w, 1244s, 1183s, 1144w, 1049s, 1027w, 941s, 872w, 842s, 805w, 792w, 719s, 706s, 693s, 661s; MS (EI): m/z (rel. int.) 468 (3), 467 (16), 466 (100), 451 (42), 309 (149, 167 (12), 126 (18); HRMS calcd. for C<sub>16</sub>H<sub>16</sub>I<sub>2</sub>O<sub>2</sub> m/z 465.89212, found m/z 465.891914.



#### Table 1; Entry 10 (Minor isomer)

 $R_f = 0.31$  (Ethyl acetate: hexane = 1:19); viscous liquid; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.44$  (s, 3 H), 3.94 (s, 3 H), 6.84-6.94 (m, 2 H), 7.24-7.31 (m, 1 H), 7.56 (dd, J = 8.5, 2.2 Hz, 1 H), 7.75 (dd, J = 7.9, 1.6 Hz, 1 H), 7.56 (d, J = 2.2 Hz, 1 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 56.40, 60.29, 85.67, 93.15, 110.50, 126.19, 130.09, 131.11, 132.09, 133.83, 138.49, 139.61, 156.74, 157.62; ATR-IR (cm<sup>-1</sup>): 3001w, 2929m, 2837s, 1724s, 1594s, 1546m, 1492s, 1455m, 1413s, 1373s, 1281m, 1249m, 1233m, 1180s, 1146m, 1074s, 1053s, 1016m, 999w, 888s, 814s, 781m, 755s, 731s, 664s; MS (EI):$ *m/z*(rel. int.) 468 (2), 467 (16), 466 (100), 324 (35), 309 (17), 139 (14), 126 (12); HRMS calcd. for C<sub>16</sub>H<sub>16</sub>I<sub>2</sub>O<sub>2</sub>*m/z*465.89212, found m/z 465.892060. Supplementary Material (ESI) for Chemical Communications This journal is (c) The Royal Society of Chemistry 2007



Table 1; Entry 11<sup>9</sup>

 $R_f = 0.16$  (Hexane: ethyl acetate = 8:2); Yellow solid; mp = 266-268 °C (hexane); <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta$  = 3.90 (s, 6 H), 7.07 (d, J = 9.2 Hz, 2 H), 8.18 (d, J = 2.8 Hz, 2 H), 8.32 (d, J = 9.2, 2.8 Hz, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta$  = 56.39, 110.63, 125.85, 125.89, 127.19, 141.15, 161.85; ATR-IR (cm-1): 3129w, 2921w, 2847s, 1896s, 1611s, 1580w, 1510s, 1470m, 1453w, 1422w, 1335s, 1259s, 1182s, 1146, 1112w, 1097w, 1034s, 1013s, 900s, 863s, 823s, 786s, 747m, 713s; MS (EI): m/z (rel. int.) 305 (21), 304 (100), 245 (14), 228 (16), 73 (13), 44 (46), 32 (72).



Table 1; Entry 12 (Major isomer)

 $R_f = 0.19$  (Hexane: ethyl acetate = 7:3); white solid; mp = 115-117 °C (hexane); <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.93$  (s, 6 H), 3.95 (s, 6 H), 7.05 (d, J = 8.8 Hz, 2 H), 7.66 (dd, J = 8.8, 2.4 Hz, 2 H), 8.00 (d, J = 2.4, Hz, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 52.13$ , 56.17, 112.49, 120.29, 129.79, 131.48, 131.86, 158.37, 166.60; ATR-IR (cm<sup>-1</sup>): 3033s, 2925w, 2840s, 1704s, 1611s, 1567s, 1489s, 1433m, 1380s, 1313s, 1258s, 1236w, 1181s, 1154s, 1094s, 1059s, 1013s, 960s, 900s, 841s, 801s, 778s, 705s, 678s; MS (EI): m/z (rel. int.) 331 (20), 330 (100), 315 (12) 300 (7), 299 (36), 297 (15), 285 (10), 139 (9); HRMS calcd. for C<sub>18</sub>H<sub>18</sub>O<sub>6</sub> m/z 330.1098, found m/z 330.1103.



Table 1; Entry 12 (Minor isomer)

 $R_f = 0.27$  (Hexane: ethyl acetate = 7:3); colorless oil; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 3.50$  (s, 3 H), 3.91 (s, 3 H), 3.95 (s, 3 H), 3.98 (s, 3 H), 7.05 (d, J = 8.7 Hz, 1 H), 7.22 (t, J = 7.1 Hz, 1 H), 7.50 (dd, J = 5.7, 2.0 Hz, 1 H), 7.71-7.78 (m, 2 H), 8.01 (d, J = 2.0 Hz, 1 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 52.08$ , 52.31, 56.11, 61.54, 111.92, 119.92, 124.04, 125.71, 129.43, 130.38, 132.27, 134.24, 134.60, 135.07, 157.14, 158.59, 166.45, 166.81; MS (EI): m/z (rel. int.) 331 (22), 330 (100), 299 (42), 297 (22), 256 (12), 239 (19), 238 (13), 209 (10), 139 (13), 126 (129, 44 (11), 32 (11);



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 $R_f = 0.36$  (Hexane: ethyl acetate = 9:1); white solid; mp = 134-136 °C (hexane); <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 2.11$  (s, 6H), 3.90 (s, 6 H), 7.36 (d, J = 8.0 Hz, 2 H), 7.79 (d, J = 1.8, Hz, 2 H), 7.96 (dd, J = 8.0, 1.8 Hz, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 20.04$ , 52.01, 127.84, 128.78, 130.14, 130.42, 140.65, 141.41, 167.02; ATR-IR (cm<sup>-1</sup>): 3429s, 3024s, 2954w, 1721s, 1606s, 1574s, 1433s, 1295m, 1254w, 1231w, 1189m, 1110w, 1045s, 997m, 966m, 912s, 853s, 834s, 790w, 761w; MS (EI): m/z (rel. int.) 299 (18), 298 (92), 283 (11), 268 (17), 267 (100), 239 (13), 180 (31), 179 (25), 178 (209, 165 (40), 118 (26), 103 (13), 89 (13); HRMS calcd. for C<sub>18</sub>H<sub>18</sub>O<sub>4</sub> m/z 298.11996, found m/z 298.119669.



 $R_f = 0.63$  (Hexane); white solid; mp = 62-65 °C; <sup>1</sup>H NMR (300.1 MHz, CDCl<sub>3</sub>):  $\delta = 2.48$  (d, J = 1.1 Hz, 6 H), 6.65 (qd, J = 3.8, 1.1 Hz, 2 H), 6.89 (d, J = 3.8 Hz, 2 H); <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>):  $\delta = 15.30$ , 122.84, 125.72, 135.49, 138.45; ATR-IR (cm<sup>-1</sup>): 3066s, 2972w, 2914m, 2850m, 2736s, 1717s, 1570s, 1531s, 1450w, 1381m, 1202s, 1155m, 1051s, 1036w, 871s, 858s, 778w, 737s, 667s; MS (EI): m/z (rel. int.) 195 (18), 194 (100), 193 (73), 179 (10), 161 (33), 96 (8), 32 (30); HRMS calcd. for  $C_{10}H_{10}S_2 m/z$  194.02184, found m/z 194.022189.

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