# Nickel-Catalyzed, Sodium Iodide-Promoted Reductive Dimerization of Alkyl Halides, Alkyl Pseudohalides, and Allylic Acetates

Michael R. Prinsell, Daniel A. Everson, Daniel J. Weix\* Department of Chemistry, University of Rochester, Rochester, NY 14627

**Supporting Information** 

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## I. General Information and Methods

### Chemicals

NiCl<sub>2</sub> glyme was purchased from Strem or prepared according to literature procedure.<sup>2</sup> The stoichiometry of the NiCl<sub>2</sub> glyme was found to be variable (the NiCl<sub>2</sub> glyme used in this study was found to contain 0.1 gylme per nickel) and the glyme is easily lost upon exposure to vacuum. If the glyme content of NiCl<sub>2</sub> glyme is unknown, calculate the weight based on a 1:1 adduct of nickel:glyme. A slight excess of nickel does not diminish yields, but a nickel deficit can result in long reaction times. The ligand, 4,4',4"-tri-*tert*-butyl-2,2':6',2"-terpyridine, was purchased form Aldrich and used as received or synthesized according to the literature procedures.<sup>3,4</sup> Manganese powder (-325 mesh, 99%+) was purchased from Aldrich and used as received. All other compounds were purchased from Aldrich, Alfa, Acros, or TCI America and used as received with the exception of iodooctane, benzyl bromide, and benzyl chloride, which were filtered through basic alumina prior to use.

Anhydrous DMF was prepared from ACS grade, inhibitor free solvent by passage though activated alumina and molecular sieves in a Glass Contour solvent purification system. The DMF could be stored over 4 Å MS in a 20 mL vial scintillation vial with a polyseal cap on the bench top, and used as is for up to one week without being deleterious to the homocoupling reactions.

All other compounds were synthesized according to the literature procedures: N-(tert-butoxycarbonyl)-3-bromopropylamine,<sup>5</sup> N-Benzyloxycarbonyl-3-bromopropylamine,<sup>6</sup> 7-bromo-2-heptanone,<sup>7</sup> octyl methanesulfonate,<sup>8</sup> benzyl trifluoroacetate,<sup>9</sup> octyl trifluoroacetate,<sup>10</sup> cinnamyl acetate,<sup>11</sup> cyclohex-2-ene-1-ol,<sup>12, 13</sup> 1-cyclohex-2-enyl acetate.<sup>13</sup>

#### Methods.

NMR chemical shifts are reported in ppm and referenced to the residual solvent peak CDCl<sub>3</sub> ( $\delta$  = 7.26 ppm <sup>1</sup>H or  $\delta$  = 77.16 ppm <sup>13</sup>C) as an internal standard, and *J* values given in Hz. NMR spectra were recorded on Bruker model Avance NMR spectrometer operating at 400.13 MHz or 500.13 MHz proton NMR frequency, and data analysis was performed using the iNMR software package (version 3.1.5).

GC analyses were performed on an Agilent 7890A GC equipped with dual DB-5 columns (20 m x 180  $\mu$ m x 0.18  $\mu$ m), dual FID detectors, and with hydrogen carrier gas. The analysis method used in all cases was 1  $\mu$ L inj. of sample, inj. temp of 300 °C, 100:1 split ratio, initial inlet pressure was 20.3 psi but varied as the column flow was held constant at 1.8 mL/min for the duration of the run. Initial oven temperature of 50 °C was held for 0.46 min followed by a temperature ramp up to 300 °C at 65 °C/min and finally the temperature was held at 300 °C for 0.69 min. Total run time was ~ 5 min. FID temperature was 325 °C.

GC/MS analyses were performed on a Shimadzu GCMS-QP2010 equipped with an RTX-XLB column (30 m x 0.25 mm x 0.28  $\mu m$ ) with a quadrupole mass analyzer and

helium carrier gas. The analysis method used in all cases was  $5 \ \mu L$  inj. of sample, inj. temp of 225 °C, 25:1 split ratio, initial inlet pressure was 7.8 psi, but varied as the column flow was held constant at 1.0 mL/min for the duration of the run, the interface temperature was held at 250 °C, and the ion source (EI, 30 eV or 70 eV) was held at 250 °C. Initial oven temperature was held at 50 °C for 3 min with the detector off followed by a temperature ramp, with the detector on, to 280 °C at 40 °C/min, and finally the temperature was held at 280 °C for 3 min. Total run time was 11.75 min. The parent ion and the base peak are reported. APCI MS in positive ion mode was carried out on a Shimadzu LCMS-2010 equipped with a single quadrupole mass analyzer using direct injection.

Chromatography was performed on silica gel (EMD, silica gel 60, particle size 0.040-0.063 mm) using standard flash techniques. Products were visualized by one of the following methods: UV light, ninhydrin stain, DNPH stain, KMnO<sub>4</sub> stain or by GC.

Melting points were measured with a Mel-Temp melting point apparatus (50/60 cycles, 110-120 V, 200 W) and are reported uncalibrated.

IR spectra were recorded on a Schimadzu FTIR-8400S spectrometer.

### **II. General Procedures**

### (A) Procedure for reactions set up on bench and run under air.

This procedure was used for Table 1, entries 3-24.

No precautions were taken to exclude air or moisture from the reaction vessel, but anhydrous DMF was used as the solvent. Reactions were set up on the bench in 1 dram vials that were stored in air. NiCl<sub>2</sub>glyme (1: 0.1 Ni: glyme, MW = 138.61) was used at the following loadings; 0.5 mol % (1.4 mg, 0.0101 mmol), 2 mol % (5.5 mg, 0.0397 mmol) or 5 mol % (13.9 mg, 0.1003 mmol), and 4,4',4''-tri-*tert*-butyl-2,2':6',2''-terpyridine (MW = 401.59) was used at the following corresponding loadings 0.5 mol % (16.1 mg, 0.0401 mmol), or 5 mol % (40.2 mg, 0.1001 mmol). The NiCl<sub>2</sub>glyme and 4,4',4''-tri-*tert*-butyl-2,2':6',2''-terpyridine were added to the vials, followed by sodium iodide (if used, 150 mg, 1.00 mmol or 300 mg, 2.00 mmol), a teflon coated stir bar, DMF (2.0 mL), aliphatic substrate (2.00 mmol) and Mn (110 mg, 2.00 mmol). The vial was then capped with a PTFE-faced silicone septum and placed on a heated reaction block set to 40 °C.

In all cases the reaction progress was followed by GC analysis. At appropriate times  $10 \ \mu\text{L}$  aliquots of reaction mixture were removed with a  $50 \ \mu\text{L}$  gas-tight syringe and quenched with  $15 \ \mu\text{L}$  of 1 M aqueous NaHSO<sub>4</sub>, diluted with ethyl ether or ethyl acetate (1 mL), and filtered through a short silica pad (0.5-1.5 cm) in a pipette packed with glass wool. The filtrate was analyzed by gas chromatography.

### (B) Procedure for reactions set up in a glove box and run under nitrogen.

This procedure was used for Table 1, entries 1, 4, 6, 10 and 17 (each of these was only run once by this method. All entries except 1 were repeated using Procedure A).

As in (A), but reactions were set up in a nitrogen filled glove box. NiCl<sub>2</sub> glyme and 4,4',4"-tri-*tert*-butyl-2,2':6',2"-terpyridine were massed and added to an oven dried 1-dram vial. A Teflon-coated stir-bar was then added, followed by the aliphatic substrate and DMF. Finally, Mn was added, the vials were capped with a PTFE-faced silicone septum, removed from the box, and placed in a reaction heating block where the temperature was set to the desired level.

### (C) Procedure for reactions set up on the bench and run under nitrogen.

This procedure was used for Table 1, entries 2 and 12 (both of these was run only once by this method. Entry 12 was repeated using Procedure A).

As in (A), but reactions were performed in 25mL Schlenk tubes. The tubes were equipped with a teflon-coated stir bar. flame dried, and cooled under a stream of dry nitrogen. NiCl<sub>2</sub> glyme and 4,4',4"-tri-*tert*-butyl-2,2':6',2"-terpyridine were massed and added to the tube, and the tube was then purged with nitrogen. The aliphatic substrate and DMF were injected through a rubber septum and then the reaction liquid was sparged with nitrogen for 30 s to 1 min. Mn was then added, the tube sealed, purged with nitrogen, and stirred in a heated oil bath.

## **III. Optimization and Control Reactions**

Table S1 Optimization of the Dimerization of Alkyl Halides

	C <sub>8</sub> H <sub>17</sub> –X	y mol % NiCl <sub>2</sub> (g y mol % ligand 2 equiv Mn DMF	glyme)	$\frac{1}{2}$	C <sub>8</sub> F	H <sub>17</sub> –C	<sub>8</sub> H <sub>17</sub>	
Entry	L	igand		Х	у	T (°C)	t (h)	Yield (%)
1	R		$\frac{1}{R = t-Bu}$	I	20	60	16	28
2	R	N N	R = OMe	Ι	20	60	16	31
3	<b>D</b> 1	R <sup>2</sup>	${\bf 3}$ R <sup>1</sup> ,R <sup>2</sup> ,R <sup>3</sup> = H	Ι	20	60	16	38
4		N	$4$ $R^{1}, R^{2} = H$ $R^{3} = Me$	Ι	20	60	16	1
5		N N		I	20	60	16	22
6	R <sup>1</sup>	R <sup>2</sup>	$6$ $R^{1} = Ph$ $R^{2}, R^{3} = H$	Ι	20	60	16	39
7	ſ	R N	7 R = H	I	20	60	16	25
8	Ĺ	N I R	<b>8</b> R = Me	Ι	20	60	16	23
9 10	(-)-s (2-py) <sub>2</sub> PC	sparteine H <sub>2</sub> CH <sub>2</sub> P(2-py) <sub>2</sub>	9 10	I I	20 20	60 60	16 16	2 22
11 12 13 14 15 16 17 <sup>b</sup>	f-Bu	N N t-Bu	11	I Br Br Br Br Br	20 20 10 5 1 2 0.5	60 60 60 60 100 40	16	55 69 67 73 53 89 96

<sup>*a*</sup> Reactions were run with 0.5 mmol of alkyl halide in 2 mL DMF (0.25 M) and yields were determined by GC analysis, corrected vs. internal standard (dodecane). <sup>*b*</sup> Reaction run on 2 mmol scale in 2 mL of DMF (1 M in alkyl halide).

### Table S2 Control Reactions



### **IV. Reductive Dimerization Reactions**

Table 1 Scope of the Reductive Dimerization of Alkyl Bromides<sup>a</sup>

	y mol % NiCl <sub>2</sub> y mol % terpy	(glyme) ridine <b>1</b>	_ 1		
	R–X 1 equiv Mn DMF, 40-80 °	- ۹ equiv Mn ۸F, 40-80 °C, 13-40 h			
Entry	Substrate	у	Additive	t (h)	Yield $(\%)^b$
	C <sub>7</sub> H <sub>15</sub> Br		-		
1	in glove box	0.5	-	17	98°
2	under $N_2$ on benchtop	0.5	-	17	98°
3	under air on benchtop	0.5	-	19	96°
4	Ph	0.5	-	17	97
5	Ph Br	0.5	-	20	81
6	EtO <sub>2</sub> C Br	0.5	-	27	95
7	BocHN	0.5	-	24	81 <sup>c</sup>
8	CbzHN Br	0.5	-	24	93
9	O ()4 Br	0.5	-	14	90 <sup>c</sup>
10	Ph Br	0.5	-	18	$80^d$
11	Br	2	-	18	78
12	C <sub>7</sub> H <sub>15</sub>	2	-	17	94
13	Ph	2	-	20	81
14	Ph	5	-	20	84 <sup>e</sup>
15	OAc	5	-	40	46 <sup><i>d</i></sup>
16		2	-	100	(20)
$17^{g}$	C <sub>7</sub> H <sub>15</sub> Cl	2	Nal	18	86
18		2	-	13	(NR)
19	C <sub>7</sub> H <sub>15</sub> OWs	2	Nal	21	82
20		2	-	13	(NR)
21		2	Nal	15	57
22	~	2	-	13	(NR)
23 <sup>g</sup>	C <sub>7</sub> H <sub>15</sub> OC(O)CF <sub>3</sub>	2	Naľ	21	35
$24^{g}$		2	NaI″	25	$45^{\circ}$

<sup>*a*</sup> Reaction conditions: On the benchtop, 2 mmol alkyl halide or pseudohalide, 2 mmol Mn powder, NiCl<sub>2</sub>(glyme), and terpyridine 1 were combined and stirred in 2 mL of anhydrous DMF. <sup>*b*</sup> Isolated yields, average of two runs. Yields in parenthesis are GC yields, corrected vs. internal standard. NR = only sm and/or hydrolysis products observed. <sup>*c*</sup> Single run. <sup>*d*</sup> Product is a 1:1 mixture of diastereomers. <sup>*e*</sup> Crude product has a 94:6 [linear]:[other isomers] ratio. <sup>*f*</sup> 50 mol % NaI was added to the reaction. <sup>*g*</sup> At 80 °C. <sup>*h*</sup> 100 mol % NaI was added to the reaction.

## Hexadecane (Table 1, Entry 1) [544-76-3].<sup>14</sup>

Procedure **B** was followed with a catalyst loading of 0.5 mol % and 1-bromooctane (346  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (~250 mL). Concentration of the filtrate yielded

pure hexadecane (222.7 mg, 98%) as a straw-colored oil; analytical data matched those reported in the literature.  $\delta_{H}(500 \text{ MHz}; \text{CDCl}_{3})$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{C}(125 \text{ MHz}; \text{CDCl}_{3})$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 39%), 57 (100).

### Hexadecane (Table 1, Entry 2) [544-76-3].<sup>14</sup>

Procedure C was followed with a catalyst loading of 0.5 mol % and 1-bromooctane (346  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (~250 mL). Concentration of the filtrate yielded pure hexadecane (222.3 mg, 98%) as a faintly yellow oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{CDCl}_3)$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 2%), 57 (100).

### Hexadecane (Table 1, Entry 3) [544-76-3].<sup>14</sup>

Procedure A was followed with a catalyst loading of 0.5 mol % and 1-bromooctane (346  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 19 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (~250 mL). Concentration of the filtrate yielded pure hexadecane (217.2 mg, 96%) as a faintly yellow oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{CDCl}_3)$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 2%), 57 (100).



### 1,4-diphenylbutane (Table 1, Entry 4) [1083-56-3].<sup>15</sup>

Procedure **B** was followed with a catalyst loading of 0.5 mol % and (2bromoethyl)benzene (273  $\mu$ L, 1.99 mmol). The reaction mixture was stirred at 40 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with ether) and the silica gel pad was washed with additional ether (~250 mL). Concentration of the filtrate yielded pure 1,4-diphenylbutane (397.3 mg, 95%) as a colorless oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{ CDCl}_3)$  7.15-7.4 (10 H, m), 2.67 (4 H, m), 1.70 (4 H, m);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  142.7, 128.5, 128.4, 125.8, 36.0, 31.2; m/z (EI) 210 (M<sup>+</sup>, 93%), 91 (100); mp 53-55 °C (lit., <sup>16</sup> 52 °C). The reaction was repeated outside the box using procedure **A** with identical reagent amounts, yielding pure 1,4-diphenylbutane (205.6 mg, 98%) after 15.5 hours.



### 1,2-diphenylethane (Table 1, Entry 5) [103-29-7].<sup>15</sup>

Procedure **A** was followed with a catalyst loading of 0.5 mol % and benzyl bromide (238  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 20 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (~250 mL). Concentration of the filtrate yielded pure 1,2-diphenylethane (149.1 mg, 82%) as a white solid; analytical data matched those reported in the literature.  $\delta_{\rm H}$ (500 MHz; CDCl<sub>3</sub>) 7.18-7.32 (5 H, m), 2.94 (2 H, s);  $\delta_{\rm C}$ (125 MHz; CDCl<sub>3</sub>) 141.9, 128.6, 128.5, 126.0, 38.1; m/z (EI) 182 (M<sup>+</sup>, 27%), 91 (100); mp 45-49 °C (lit.,<sup>17</sup> 52 °C). The reaction was repeated under identical conditions yielding pure 1,2-diphenylethane (143.8 mg, 79%) after 17 hours.

**Octanedinoic acid diethyl ester (diethyl suberate) (Table 1, Entry 6) [2050-23-9].** Procedure **B** was followed with a catalyst loading of 0.5 mol % and Ethyl-4bromobutyrate (286  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with 10% ether in pentane) and the silica gel pad was washed with additional ether (~250 mL). Concentration of the filtrate yielded pure octanedinoic acid diethyl ester (216.1 mg, 94%) as a faintly yellow oil; analytical data matched those found in the Aldrich <sup>1</sup>H/<sup>13</sup>C NMR Spectral Database.  $\nu_{max}$ (neat liquid)/cm<sup>-1</sup> 1732 (CO);  $\delta_{H}$ (500 MHz; CDCl<sub>3</sub>) 4.11 (2 H, q, *J* 7.2), 2.28 (2 H, t, *J* 7.5), 1.62 (2 H, m), 1.33 (2 H, m), 1.25 (3H, t, *J* 7.1);  $\delta_{C}$ (125 MHz; CDCl<sub>3</sub>) 173.9, 603, 34.4, 28.9, 24.9, 14.4; m/z (EI) 231 (M + H, 5%), 185 (100). The reaction was repeated outside the box using procedure **A** with identical reagent amounts, yielding pure octanedinoic acid diethyl ester (219.9 mg, 95%) after 15.5 hours.

BocHN

*N*,*N*<sup>'</sup>-bis(*tert*-butoxycarbonyl)-1,6-hexanediamine (Table 1, Entry 7) [16644-54-5].<sup>18</sup> Procedure A was followed with a catalyst loading of 0.5 mol % and *N*-(*tert*-butoxycarbonyl)-3-bromopropylamine (476.2 mg, 2.00 mmol). The reaction mixture was stirred at 40 °C for 24 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was diluted with ether (50 mL) and transferred to a separatory funnel resulting in a cloudy brown suspension. Upon the addition of water (50 mL), the organic layer appeared to separate into two phases. The aqueous layer was discarded and the two organic layers were washed with water (4 × 50 mL). Dichloromethane was added until the two organic layers became one and the resulting solution was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated on a rotary evaporator to give pure *N*,*N*-bis(*tert*-butoxycarbonyl)-1,6-hexanediamine (254.8 mg,

81%) as a white solid; analytical data matched those reported in the literature.  $v_{max}(solid)/cm^{-1}$  3371 (NH), 1686 (CO);  $\delta_{H}(500 \text{ MHz}; \text{CDCl}_{3})$  4.56 (2 H, br s), 3.08 (4 H, q, *J* 6.3), 1.42 (18 H, s), 3.02 (4 H, m);  $\delta_{C}(125 \text{ MHz}; \text{CDCl}_{3})$  156.1, 79.1, 40.5, 30.1, 30.1, 28.5, 26.4; m/z (APCI) 317 (M + H, 100%); mp 99-100 °C (lit., <sup>18</sup> 101-102 °C).

CbzHN NHCbz

*N*,*N*'-Bis(benzyloxycarbonyl)hexane-1,6-diamine (Table 1, Entry 8) [16644-57-8].<sup>19</sup> Procedure A was followed with a catalyst loading of 0.5 mol % and N-Benzyloxycarbonyl-3-bromopropylamine (544.3 mg, 2.00 mmol). The reaction mixture was stirred at 40 °C for 24 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was diluted with ether (50 mL) and transferred to a separatory funnel resulting in a cloudy brown suspension. Upon the addition of water (50 mL), the organic layer appeared to separate into two phases, with a white solid forming at the organic/water interface. The aqueous layer was discarded and the two organic layers were washed with water ( $4 \times 50$  mL). Dichloromethane was added until the two organic layers became one (and the white solid dissolved) and the resulting solution was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated on a rotary evaporator to give pure  $N_N$ -Bis(benzyloxycarbonyl)hexane-1,6-diamine (354.5 mg, 92%) as white solid; analytical data matched those reported in the literature.  $v_{max}(solid)/cm^{-1}$  3325 (NH), 1682 (CO); δ<sub>H</sub>(500 MHz; CDCl<sub>3</sub>) 7.27-7.45 (10 H, m), 5.09 (4 H, s), 4.76 (2 H, br s), 3.18 (4 H, q, J 6.3), 1.49 (4 H, m), 1.33 (4 H, m); δ<sub>C</sub>(125 MHz; CDCl<sub>3</sub>) 156.5, 136.8, 128.6, 128.3, 128.2, 66.7, 41.0, 30.0, 26.3; m/z (APCI) 385 (M + H, 100%); mp 118-122 °C (lit.,<sup>19</sup> 126-132 °C). The reaction was repeated under identical conditions yielding pure *N*,*N*'-Bis(benzyloxycarbonyl)hexane-1,6-diamine (361.5 mg, 94%) after 17 hours.



### 2,13-tetradecanedione (Table 1, Entry 9) [7029-11-0].<sup>20</sup>

Procedure **A** was followed with a catalyst loading of 0.5 mol % and 5-bromopentyl methyl ketone (386  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 14 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was diluted with ether (50 mL) and transferred to a separatory funnel resulting in a cloudy brown suspension. Upon the addition of water (50 mL), the organic layer appeared to separate into two phases. The aqueous layer was discarded and the two organic layers were washed with water (4 × 50 mL). Dichloromethane was added until the two organic layers became one and the resulting solution was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated on a rotary evaporator to give pure 2,13-tetradecanedione (203.6 mg, 90%) as white solid; analytical data matched those reported in the literature.  $v_{max}$ (solid)/cm<sup>-1</sup> 1701 (CO);  $\delta_{H}$ (500 MHz; CDCl<sub>3</sub>) 2.40 (4 H, t, *J* 7.5), 2.12 (6 H, s), 1.55 (4 H, m), 1.26 (12 H, s);  $\delta_{C}$ (125 MHz; CDCl<sub>3</sub>) 209.5, 43.9, 30.0, 29.5, 22.5, 22.5, 29.3, 24.0; m/z (EI) 227 (M + H, 24%), 58 (100); mp 70-72 °C (lit.<sup>20</sup> 71.5-72 °C).



# 2,3-diphenylbutane (Table 1, Entry 10) [1857-74-5, 2726-21-8, 4539-34-8, 4613-11-0, 5789-35-5, 71606-46-7].<sup>21</sup>

Procedure B was followed with a catalyst loading of 0.5 mol % and (1bromoethyl)benzene (273 µL, 2.00 mmol). The reaction mixture was stirred at 40 °C for 18 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately 1/2 full with silica gel that was pre-wetted with ether) and the silica gel pad was washed with additional ether (~250 mL). The filtrate was concentrated and purified by chromatography (1% ethyl acetate in hexanes) to give 2,3diphenylbutane (157.8 mg, 75%) as a white solid; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{CDCl}_3)$  7.33 (6 H, m), 7.0-7.25 (16 H, m), 2.95 (1.8 H, m), 2.80 (2 H, m), 1.28 (4.01 H, dd, J 4.9, 1.9) 1.02 (5.22 H, dd, J 4.5, 2.0); δ<sub>C</sub>(125 MHz; CDCl<sub>3</sub>); 146.6, 146.0, 128.4, 128.0, 127.8, 126.2, 125.8, 47.4, 46.6, 21.2, 18.1; m/z (EI) isomer #1: 210 (M<sup>+</sup>, 2%), 105 (100), isomer #2 210 (M<sup>+</sup>, 2%), 105 (100). The reaction was repeated outside the box using procedure A with identical reagent amounts, after 17 h the reaction mixture was filtered through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately  $\frac{1}{2}$  full with silica gel that was pre-wetted with pentane) and the silica gel pad was washed with additional pentane (~250 mL). The filtrate was concentrated yielding pure 2,3-diphenylbutane (177.1 mg, 84%).

# $\bigcirc \frown \bigcirc$

### Bicyclopentyl (Table 1, Entry 11) [CAS: 1636-39-1].<sup>22</sup>

Procedure A was followed with a catalyst loading of 2.0 mol % and cyclopentyl bromide (214  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 18 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (3 × 30 mL). Concentration of the filtrate yielded pure bicyclopentyl (113.2 mg, 82%) as a colorless oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{CDCl}_3)$  1.73 (4 H, m), 1.42-1.64 (10 H, m) and 1.13 (4 H, m);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  46.5, 32.0, 25.5; m/z (EI) 138 (M<sup>+</sup>, 10%), 96 (71), 82 (86), 67 (100). The reaction was repeated under identical conditions yielding pure bicyclopentyl (101.6 mg, 73%) after 17 hours.

### Hexadecane (Table 1, Entry 12) [544-76-3].<sup>14</sup>

Procedure C was followed with a catalyst loading of 2.0 mol % and 1-iodooctane (361  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (~250mL). Concentration of the filtrate yielded

pure hexadecane (217.3 mg, 96%) as a colorless oil; analytical data matched those reported in the literature.  $\delta_{H}(500 \text{ MHz}; \text{CDCl}_3)$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{C}(125 \text{ MHz}; \text{CDCl}_3)$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 2%), 57 (100). The reaction was repeated outside the box using procedure **A** with identical reagent amounts, yielding pure hexadecane (208.2 mg, 92%) after 17 hours.



### 1,2-diphenylethane (Table 1, Entry 13) [103-29-7].<sup>15</sup>

Procedure A was followed with a catalyst loading of 2.0 mol % and benzyl chloride (230  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 20 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (3 × 30 mL). Concentration of the filtrate yielded pure 1,2-diphenylethane (153.9 mg, 84%) as a white solid; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{CDCl}_3)$  7.18-7.32 (5 H, m), 2.94 (2 H, s);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  141.9, 128.6, 128.5, 126.0, 38.1; m/z (EI) 182 (M<sup>+</sup>, 23%), 91 (100); mp 46-50 °C (lit.,<sup>17</sup> 52 °C). The reaction was repeated under identical conditions yielding pure 1,2-diphenylethane (133.6 mg, 78%) after 17 hours.



# (E,E)-1,6-Diphenylhexa-1,5-diene (Table 1, Entry 14) [58463-02-8, 33788-20-4, 87368-02-3, 4439-45-6].<sup>24</sup>

Procedure A was followed with a catalyst loading of 5.0 mol % and cinnamyl acetate (334  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 20 h and progress was monitored by GC analysis. When the reaction was judged complete (gc analysis of the crude reaction mixture showed a 94:6, linear:branched ratio), the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (~250 mL). Concentration of the filtrate yielded pure (*E*,*E*)-1,6-Diphenylhexa-1,5-diene (185.8 mg, 79%) as a white solid; analytical data matched those reported in the literature.  $\delta_{\rm H}(400 \text{ MHz}; \text{ CDCl}_3)$  7.35 (4 H, m), 7.30 (4 H, m), 7.20 (2 H, m), 6.44 (2 H, d, *J* 15.8), 6.28 (2 H, d, *J* 15.8), 2.40 (4 H, m);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  137.9, 130.5, 130.1, 128.6, 127.1, 126.1, 33.1; m/z (EI) 234 (M<sup>+</sup>, 4%), 117 (100); mp 68-73 °C (lit.,<sup>24</sup> 78-80 °C). The reaction was repeated under identical conditions yielding pure (*E*,*E*)-1,6-Diphenylhexa-1,5-diene (207.8 mg, 89%) after 24.5 hours (gc analysis of the crude reaction mixture showed a 93:7, linear:branched ratio).

3,3'-bicyclohexenyl (Table 1, Entry 15) [1541-20-4, 42347-45-5, 42347-46-6, 57705-09-6].<sup>25</sup>

Procedure **A** was followed with a catalyst loading of 5.0 mol % and cyclohex-2-en-1-yl acetate (280  $\mu$ L, 2.00 mmol). The reaction mixture was stirred at 40 °C for 40 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with n-pentane) and the silica gel pad was washed with additional pentane (~250 mL). Concentration of the filtrate yielded pure 3,3'-bicyclohexenyl (70.6 mg, 43%, 1:1 mixture of *meso* and *dl*) as a yellow oil; analytical data matched those reported in the literature.  $\delta_{H}(500 \text{ MHz}; \text{CDCl}_3)$  5.73 (2 H, m), 5.61 (1 H, m), 5.56 (1 H, m), 1.26-2.23 (14 H, m);  $\delta_{C}(125 \text{ MHz}; \text{CDCl}_3)$  130.8, 130.6, 128.1, 127.8, 40.3, 40.2, 26.2, 26.1, 25.6, 25.5, 22.4, 22.4; m/z (EI) 161 (M - H, 2%), 81 (100). The reaction was repeated under identical conditions yielding pure 3,3'-bicyclohexenyl (78.8 mg, 49%, 1:1 mixture of *meso* and *dl*) after 65 hours.

### Hexadecane (Table 1, Entry 17) [544-76-3].<sup>14</sup>

Procedure **B** was followed with a catalyst loading of 2.0 mol %, 1-chlorooctane (340  $\mu$ L, 2.00 mmol) and sodium iodide (150 mg, 1.00 mmol). The reaction mixture was stirred at 80 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was prewetted with hexanes) and the silica gel pad was washed with additional hexanes (~250 mL). Concentration of the filtrate yielded pure hexadecane (199.9 mg, 88%) as a colorless oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{CDCl}_3)$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{\rm C}(125 \text{ MHz}; \text{CDCl}_3)$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 2%), 57 (100). The reaction was repeated outside the box using procedure **A** with identical reagent amounts, yielding pure hexadecane (190.6 mg, 84%) after 18 hours.

### Hexadecane (Table 1, Entry 19) [544-76-3].<sup>14</sup>

Procedure **A** was followed with a catalyst loading of 2.0 mol %, octyl methanesulfonate (416.7 mg, 2.00 mmol) and sodium iodide (150 mg, 1.00 mmol). The reaction mixture was stirred at 40 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (3 × 30). Concentration of the filtrate yielded pure hexadecane (186.2 mg, 82%) as a colorless oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{ CDCl}_3)$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{\rm C}(125 \text{ MHz}; \text{ CDCl}_3)$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 3%), 57 (100). The reaction was repeated under identical conditions yielding pure hexadecane (184.1 mg, 81%) after 24 hours.



### 1,2-diphenylethane (Table 1, Entry 21) [103-29-7].<sup>23</sup>

Procedure A was followed with a catalyst loading of 2.0 mol %, benzyl trifluoroacetate (408.4 mg, 2.00 mmol) and sodium iodide (150 mg, 1.00 mmol). The reaction mixture was stirred at 40 °C for 13 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (3 × 30). Concentration of the filtrate yielded pure 1,2-diphenylethane (99.2 mg, 54%) as a white solid; analytical data matched those reported in the literature.  $\delta_{H}(500 \text{ MHz}; \text{CDCl}_3)$  7.18-7.32 (5 H, m), 2.95 (2 H, s);  $\delta_{C}(125 \text{ MHz}; \text{CDCl}_3)$  141.9, 128.6, 128.5, 126.0, 38.1; m/z (EI) 182 (M<sup>+</sup>, 26%), 91 (100); mp 48-51 °C (lit.,<sup>17</sup> 52 °C). The reaction was repeated under identical conditions yielding pure 1,2-diphenylethane (107.4 mg, 59%) after 17 hours.

### Hexadecane (Table 1, Entry 23) [544-76-3].<sup>14</sup>

Procedure A was followed with a catalyst loading of 2.0 mol %, octyl trifluoroacetate (452.6 mg, 2.00 mmol) with sodium iodide (150 mg, 1.00 mmol). The reaction mixture was stirred at 80 °C for 17 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (3 × 30). Concentration of the filtrate yielded pure hexadecane (107.5 mg, 47%) as a colorless oil; analytical data matched those reported in the literature.  $\delta_{\rm H}(500 \text{ MHz}; \text{ CDCl}_3)$  1.2-1.35 (28 H, m), 0.88 (3H, t, *J* 6.8);  $\delta_{\rm C}(125 \text{ MHz}; \text{ CDCl}_3)$  32.1, 29.9, 29.8, 29.5, 22.9, 14.3; m/z (EI) 226 (M<sup>+</sup>, 2%), 57 (100). The reaction was repeated under identical conditions yielding pure hexadecane (49.9 mg, 22%) after 25 hours.

### Hexadecane (Table 1, Entry 24) [544-76-3].<sup>14</sup>

Procedure A was followed with a catalyst loading of 2.0 mol %, octyl trifluoroacetate (452.6 mg, 2.00 mmol) with sodium iodide (300 mg, 2.00 mmol). The reaction mixture was stirred at 80 °C for 25 h and progress was monitored by GC analysis. When the reaction was judged complete, the reaction mixture was passed through a pad of silica gel (60-mL fritted funnel, porosity "C," filled approximately ½ full with silica gel that was pre-wetted with hexanes) and the silica gel pad was washed with additional hexanes (3 × 30). Concentration of the filtrate yielded pure hexadecane (102.3 mg, 45%) as a colorless oil; analytical data matched those reported in the literature. Data matched those from Table 1, Entry 23.

### **Large Scale Reaction**



# Hexadecane (40 mmol Reaction) [544-76-3].<sup>14</sup>

This procedure is based on procedure **A**, but scaled 20 fold. A 100-mL round-bottom flask, equipped with a large teflon stirbar, was charged with NiCl<sub>2</sub>(glyme)<sub>0.1</sub> (27.7 mg, 0.200 mmol), 4,4',4''-tri-*tert*-butyl-2,2':6',2''-terpyridine (80.3 mg, 0.200 mmol), 1-bromooctane (6.91 mL, 40.0 mmol), DMF (40 mL) and Mn<sup>0</sup> powder (2.20 g, 40.0 mmol). The reaction vessel was capped with a rubber septa and stirred at 40 °C in an oil bath. When the reaction was judged complete (determined by GC analysis to be 24 h) the reaction mixture was filtered through a pad of silica gel (~2" pad in a ~2" diameter column) and the silica gel pad was washed with 100% hexanes. After concentration of the filtrate on a rotary evaporator, the residue was purified by chromatography on silica gel (~4" of silica gel in a 2" diameter column, eluted with 100% hexanes) to yield pure hexadecane (4.53g, 96%). Spectra match those reported above.



Figure 1. Large-scale reaction.

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# VI. <sup>1</sup>H and <sup>13</sup>C NMR Spectra

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Table 1, Entry 1



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