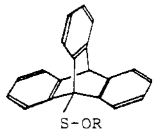
which is within the established range for C–S bonds.[14] (ii) Compound A displays its hydroxyl group absorptions at 3340 cm^{-1} (Nujol) and δ 3.2 (readily exchangable with D_2O) in IR and NMR spectra, respectively.[12b] (iii) The absence of any IR band in a range 1120–1050 cm^{-1} clearly excludes a thiosulfinate structure [RSS(O)R] from the compound A.[14] Finally, (iv) the high-resolution mass spectrum of the compound A gives correct molecular formula $C_{20}H_{14}OS$ (found *m/e* 302.0764, calcd *m/e* 302.0764).[12c,d]

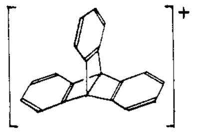
The sulfenic acid **1** (0.21 mmol), upon treatment with a twofold excess of silylating reagent such as chlorotrimethylsilane/hexamethyldisilazane and *tert*-butylchlorodimethylsilane/imidazole,[15] furnished the corresponding trimethylsilyl sulfenate **2**,[16] mp 148–149 °C, and *tert*-butyldimethylsilyl sulfenate **3**,[17] mp 139–141 °C, in 80.6% and 78.8% yield, respectively, after purification by column chromatography on silica gel. These esters are moderately stable to moisture but easily hydrolyzed back to the acid unless precautions against moisture are taken during the drying stage of the preparative TLC plates after development. The reaction shown in Scheme II and fact ii mentioned above are in good agreement with the previous result that the sulfenic acid hydrogen attaches to the oxygen atom, i.e., SOH.[2b]



2; R=$SiMe_3$

3; R=$SiMe_2Bu^t$

2, 3



4

It is of interest to note that in the mass spectrum of **1** the cleavage of the bond between the triptycene group and the sulfur atom appears to occur more easily than that of the S–O bond, as the relative intensity ratio observed for the fragments [M – SOH] (*m/e* 253) and [M – OH] (*m/e* 283) is 1:0.28, respectively. Similar trends have been also observed in the mass spectra of **2** and **3**, thus the intensity ratios of *m/e* 253 to 285 for **2** and **3** are 1:0.28 and 1:0.76, respectively. One common feature to these mass spectra of **1**, **2**, and **3** is the appearance of an intense peak at *m/e* 252, which might be equivalent to the tribenzo[2.2.2]propellatriene structure (**4**).

Addition reactions of transient sulfenic acids to carbon–carbon multiple bonds to yield sulfoxides are frequently employed to trap and characterize them.[2a,15b,18] The sulfenic acid **1** (0.25 mmol), when heated with methyl propiolate (0.5 mL) at 50 °C for 20 min, gave the expected sulfoxide, methyl *trans*-(9-triptycylsulfinyl)acrylate (**5**),[19] mp 239.5–241.5 °C, in 89.4% yield. This process can be followed by monitoring the decrease in the acetylenic proton intensity of the starting propiolate (δ 2.90) and the increase in the olefinic proton intensities of the product **5** (δ 8.48). Thus the second-order rate constant for the reaction (Scheme II) at 50 °C in chloroform-*d* has been determined as $k_2 = 1.28 \times 10^{-3}$ L·mol^{-1}·s^{-1}.

Though **1** is stable at room temperature in air for more than several weeks, it is readily converted, upon treatment with thionyl chloride at 0 °C, into a less polar compound, which is very likely to be the corresponding sulfenyl chloride on the basis of its NMR and IR spectra.[20]

The facts so far stated imply that the use of triptycene skeleton(s) may be an effective approach for stabilization of otherwise labile functional groups such as selenenic acids. Further studies are now in progress.

Acknowledgment. I acknowledge Professor Michinori Ōki, The University of Tokyo, for his generous support and Dr. Hiroshi Hirota of The University of Tokyo for taking the mass spectra.

Supplementary Material Available: IR spectra of **1** and the reaction product of 9-triptycenesulfenic acid, ^1H NMR spectra of **1**, and a mass spectral chart of **1** (7 pages). Ordering information is given on any current masthead page.

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(13) Some representative ^{13}C NMR δ values for the 9-C's of 9-triptycyl sulfides are 65.29 (2,3-Me_2-9-*t*-BuS-Trip), 65.29 (1,2,3,4-Cl_4-9-*t*-BuS-Trip), and 65.61 (9-*t*-BuS-Trip). The 9-C signal of a 9-triptycyl sulfoxide, 1,4-$(MeO)_2$-9-$PhCH_2SO$-Trip, appears at δ 72.00.

(14) Bellamy, L. J. "The Infrared Spectra of Complex Molecules"; Chapman and Hall: New York, 1980; Vol. 2, Chapter 6, p 196.

(15) (a) Chou, T. S.; Burgtorf, J. R.; Ellis, A. L.; Lammert, S. R.; Kukolja, S. P. *J. Am. Chem. Soc.* **1974**, *96*, 1609. (b) Davis, F. A.; Friedman, A. J. *J. Org. Chem.* **1976**, *41*, 897. (c) Corey, E. J.; Venkateswarlu, A. *J. Am. Chem. Soc.* **1972**, *94*, 6190.

(16) **2**: ^1H NMR ($CDCl_3$) δ 0.41 (s, 9 H, $SiMe_3$), 5.31 (s, 1 H, 10-H), 6.8–7.1 (m, 6 H, aromatic), 7.23–7.37 (m, 3 H, aromatic), 7.37–7.57 (m, 3 H, aromatic); exact mass (EI), calcd for $C_{23}H_{22}OSSi$ *m/e* 374.1151, found *m/e* 374.1150.

(17) **3**: ^1H NMR ($CDCl_3$) δ 0.40 (s, 6 H, $SiMe_2$), 0.97 (s, 9 H, *t*-Bu), 5.33 (s, 1 H, 10-H), 6.86–7.13 (m, 6 H, aromatic), 7.23–7.40 (m, 3 H, aromatic), 7.40–7.60 (m, 3 H, aromatic); exact mass (EI), calcd for $C_{26}H_{28}OSSi$ *m/e* 416.1628, found *m/e* 416.1582.

(18) (a) Jones, D. N.; Hill, D. R.; Lewton, D. A.; Sheppard, C. *J. Chem. Soc., Perkin Trans. 1* **1977**, 1574. (b) Barrett, A. G. M.; Barton, D. H. R.; Nagubandi, S. *Ibid.* **1980**, 237.

(19) **5**: ^1H NMR ($CDCl_3$) δ 3.75 (s, 3 H, OMe), 5.34 (s, 1 H, 10-H), 6.8–7.3 (m, 7 H, aromatic), 7.24 (d, 1 H, *J* = 15 Hz, olefin proton β to SO group), 7.4–7.6 (m, 3 H, aromatic), 7.90–8.06 (m, 1 H, aromatic), 8.06–8.25 (m, 1 H, aromatic), 8.48 (d, 1 H, *J* = 15 Hz, olefin proton α to SO group); ^{13}C NMR ($CDCl_3$) δ 52.33 (OMe), 54.02 (10-C), 68.37 (9-C), 122.52 (aromatic C's with H), 122.92 (olefin C β to SO), 124.01, 125.18, 126.09 (aromatic C's with H), 140.57, 146.02 (aromatic ipso-C's), 148.43 (olefin C α to SO), 166.88 (C=O); IR (Nujol) 1715 (C=O), 1065 cm^{-1} (S=O). Anal. Calcd for $C_{24}H_{18}O_3S$: C, 74.59; H, 4.69; S, 8.30. Found: C, 74.49; H, 4.65; S, 8.25.

(20) ^1H NMR ($CDCl_3$) δ 5.32 (s, 1 H, 10-H), 6.90–7.20 (m, 6 H, aromatic), 7.20–7.46 (m, 3 H, aromatic), 7.46–7.75 (m, 3 H, aromatic); ^{13}C NMR ($CDCl_3$) δ 53.82 (10-C), 65.22 (9-C), 122.62, 123.54, 125.11, 125.87 (aromatic C's with H), 142.61, 145.50 (aromatic ipso-C's); IR (CH_2Cl_2) 1155, 1090, 960, 635, 605, 530 cm^{-1}; exact mass (EI), calcd for $C_{20}H_{13}ClS$ *m/e* 320.0426, found *m/e* 320.0413.

(21) **Note Added in Proof**: In the mass spectrum of 9-(*tert*-butylthio)triptycene-*10-d*$_1$ (D content ≥95%), the intense peaks appeared at *m/e* 253 and 254. This fact indicates that the deuterium atom is retained in these peaks and argues against the formation of structure **4**. Cf.: Schwartz, L. H. *J. Org. Chem.* **1968**, *33*, 3977. Klanderman, B. H.; Criswell, T. R. *Ibid.* **1969**, *34*, 3426.

Palladium-Catalyzed Cross-Coupling of Allyl Halides with Organotins

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Received August 10, 1983

The cross-coupling of an organometallic with an organic halide catalyzed by a transition metal is an important, versatile method of stereo- and regiospecific carbon–carbon bond formation. Various organometals have been utilized in this reaction with disparate success. The copper-promoted coupling of different vinyl or aryl groups is slow and often results in extensive homocoupling.[1] Similarly, cross-coupling reactions involving Grignard reagents catalyzed by various transition metals can lead to homocoupling. The inability of many organometallic partners, such as organolithium, Grignard reagents, and organoaluminums, to tolerate more reactive functional groups, either on the organic halide or on the organometallic partner, limits the functionality that can be brought into the coupled product.[2] Other organometallic partners containing a more electropositive metal, such as boron, zinc, or zirconium, often are not conveniently synthesized, or the methods of synthesis are limited to the extent that a wide variety of structures and functional groups are not available in the organic framework of the organometallic reagent.[3] Although organo-

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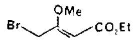
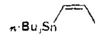
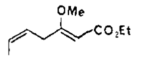
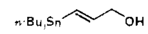
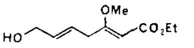
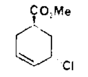
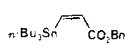
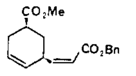
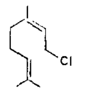
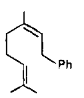
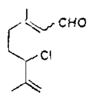
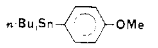
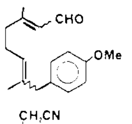
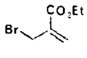
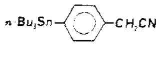
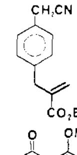
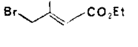
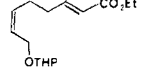
(1) Posner, G. H. "An Introduction to Synthesis Using Organocopper Reagents"; Wiley-Interscience: New York, 1982.

(2) (a) Hayashi, T.; Konishi, M.; Fukushima, M.; Mise, T.; Kagotani, M.; Tajika, M.; Kumada, M. *J. Am. Chem. Soc.* **1982**, *104*, 180 and references therein. (b) Tamao, K.; Kumada, M. *Organomet. React.*, in press.

(3) (a) Negishi, E. *Pure Appl. Chem.* **1981**, *53*, 2333. (b) Negishi, E. *Acc. Chem. Res.* **1982**, *15*, 340. (c) Negishi, E. In "Current Trends in Organic Synthesis"; Nozaki, H.; Ed.; Pergamon Press: Oxford and New York, 1983; p 269–280 and references therein.

0002-7863/83/1505-7173$01.50/0

Table I. Palladium-Catalyzed Coupling of Allyl Halides with Organotins[a]

	allyl halide	organotin	product	% yield[b,c]
1				86
2				82
3				80
4		n Bu$_3$SnPh		90
5				87
6				81
7[c]				75

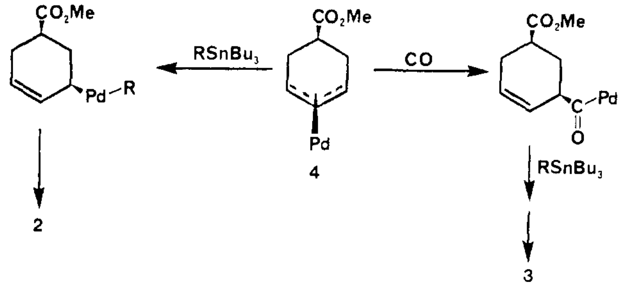
[a] The reactions were carried out in dry THF at 50 °C for 24–48 h, with equimolar amounts of the allyl halide and organotin in the presence of 3 mol % Pd(dba)$_2$ and 6 mol % PPh$_3$. [b] All compounds gave satisfactory spectral and analytical data; yields reported are isolated. [c] This reaction was run under 55 psig of carbon monoxide.

zirconium reagents undergo coupling reactions with vinyl and aryl halides containing more reactive functionality, such as ester and nitrile, the functionality on the zirconium reagent that can be carried into the coupled product is limited.[3b,c]

The palladium-catalyzed cross-coupling of acid chlorides with organotins takes place under mild conditions but, most importantly, will tolerate a wide variety of functional groups, at least on the acid chloride, including ester, nitro, nitrile, and even aldehyde.[4] Furthermore, organotins containing a variety of reactive functional groups can be prepared easily by a number of different reaction types, and these reagents are not air sensitive.[5] Thus, the cross-coupling reaction of organic halides other than acid chloride with organotins appeared to be an especially attractive, versatile method of carbon–carbon bond formation. In this paper, the palladium-catalyzed cross-coupling of allyl halides with aryl and vinyl tin reagents is reported.

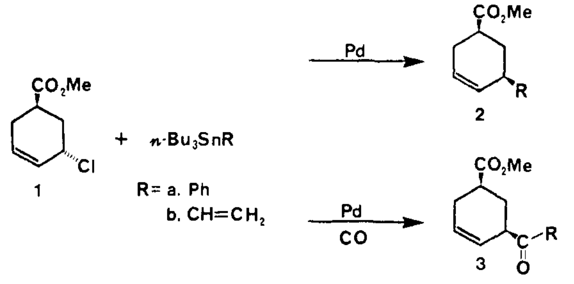
Although the cross-coupling reactions of allyl halides with allyltins gave only moderate yields of products and were not especially regiospecific,[6] the palladium-catalyzed reaction of allyl chlorides or bromides with vinyl or aryl tin reagents gave cross-coupled products in uniformly high (isolated) yields (Table I). The reaction conditions are mild and, unlike many other transition-metal-catalyzed cross-coupling reactions, is tolerant of a wide variety of reactive organic functionality, including ester (entries 1–3, 6, and 7), nitrile (entry 6), hydroxyl (entry 2), and even aldehyde (entry 5). The reaction also proceeds with retention of double-bond geometry in the vinyl partner (entries 1–3)[7] and retention of the (*Z*)-olefin geometry in neryl chloride (entry 4).[8] In the allyl partner, coupling shows regioselectivity for the primary allylic carbon rather than secondary (entry 5; compare also entries 1, 2, and 4). When the reaction is carried out under a moderate carbon monoxide pressure, product resulting from cross-coupling accompanied by carbon monoxide insertion is obtained (entry 7). Thus, this reaction exhibits stereo- and regiospecific carbon–carbon bond formation while having the ability to bring a wide variety functionality into the framework of the coupled product and the alternative of introducing ketone functionality at the coupling juncture.

In order to examine the stereochemistry of the cross-coupling reactions at the allyl halide partner, reactions with *trans*-3-chloro-5-carbomethoxycyclohexene (**1**)[9] were carried out.



The cross-coupling of **1** with either phenyltributyltin or vinyltributyltin proceeded with inversion at the allylic carbon center to yield **2**. In the presence of carbon monoxide, the carbonylated cis cross-coupled product **3** also was obtained in addition to **2**, the ratio of the two products depending upon the CO pressure. The diastereomeric composition of the allylic chloride **1**[10] and the coupled products **2**[11] and **3**[12] was determined by ^{1}H NMR.[13] When the reaction was allowed to go to partial completion at 40 °C with 5.7 mmol of **1**, 6.3 mmol of organotin, and 0.5 mol % PhCH$_2$Pd(PPh$_3$)$_2$Cl in 10 mL of dry THF either with (45 psig) or without CO, unreacted starting tin reagent and unchanged trans chloride **1** could be recovered in addition to product, **2** or **3**.

Thus, both the direct coupling reactions of the allylic chloride **1** to yield **2** and the coupling reaction in the presence of carbon monoxide to give **3** take place with inversion at the allylic carbon. The stereochemical results suggest a sequence of steps in which oxidative addition of **1** to bis(triphenylphosphine)palladium(0)[14] takes place with inversion at the carbon bearing the allylic chloride, possibly to yield an allylpalladium(II) complex, **4**.[15] The presence



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(4) (a) Milstein, D.; Stille, J. K. *J. Am. Chem. Soc.* **1978**, *100*, 3636; (b) *J. Org. Chem.* **1979**, *44*, 1613.

(5) (a) Neuman, W. P. "The Organic Chemistry of Tin"; Wiley: New York, 1970. (b) Zuckerman, J. J., Ed. *Adv. Chem. Ser.* **1976**, *No. 157*. (c) Zubieta, J. A., Zuckerman, J. J. *Progr. Inorg. Chem.* **1978**, *24*, 251.

(6) Godschalx, J.; Stille, J. K. *Tetrahedron Lett.* **1980**, *21*, 2599.

(7) The geometry in these products has been unambiguously determined through coupling constants in their ^{1}H NMR spectra.

(8) Levy, G. C.; Lichter, R. L.; Nelson, G. L. "Carbon-13 Nuclear Magnetic Resonance Spectroscopy", 2nd ed.; Wiley: New York, 1980; p 249.

(9) Available from the triphenylphosphine–carbon tetrachloride chlorination (Snyder, E. I. *J. Org. Chem.* **1972**, *37*, 1466) of the corresponding cis alcohol (Trost, B. M.: Verhoven, T. R. *J. Am. Chem. Soc.* **1980**, *102*, 4730).

(10) The cis and trans isomers of chloride **1** were differentiated on the basis of the 360-MHz ^{1}H NMR: H$_5$ (dddd) cis 2.54–2.71, trans 2.95–3.05, CO$_2$CH$_3$ (s) cis 3.71, trans 3.70 ppm.

(11) The cis and trans isomers of cross-coupled products **2** were differentiated on the basis of the 360-MHz ^{1}H NMR: **2a** (R = Ph) H$_4$ (ddd) cis 1.58–1.68, trans 2.11–2.20, H$_5$ (dddd) cis 2.72–2.82, trans 2.46–2.65, CO$_2$CH$_3$(s) cis 3.67, trans 3.64 ppm; **2b** (R = CH=CH$_2$) CO$_2$CH$_3$ (s) cis 3.69, trans 3.68 ppm.

(12) The cis and trans isomers of carbonylated cross-coupled products **3** were determined by the 360-MHz ^{1}H NMR: **3a** (R = Ph) H$_4$ (ddd) cis 1.87–1.97, trans 2.00–2.07, H$_5$ (dddd) cis 2.73–2.82, trans 2.91–2.98, CO$_2$CH$_3$ (s) cis 3.70, trans 3.72 ppm; **3b** (R =CH=CH$_2$) H$_4$(ddd) cis 1.72–1.83, trans 1.86–1.95, H$_5$ (dddd) cis 2.72–2.86, trans 2.64–2.73 ppm.

(13) Typical coupling constants for the one-proton upfield absorption corresponding to one of the H$_4$ protons: cis J = 12.6, 12.6, 12.8 Hz; trans J = 12.3, 12.4, 4.3 Hz.

(14) Milstein, D.; Stille, J. K. *J. Am. Chem. Soc.* **1979**, *101*, 4992.

of carbon monoxide in the reaction mixture does not modify the stereochemistry of this complex, since both directly cross-coupled products **2** and ketone **3** are produced at intermediate carbon monoxide pressures. Carbon monoxide diverts the allylpalladium(II) complex to ketone **3**.

Since CO insertion into a palladium-carbon bond is known to proceed with retention of configuration at carbon,[16] the overall inversion of stereochemistry observed in the formation of ketone **3** requires that oxidative addition to palladium(0) proceeds with inversion at carbon. Thus, in the formation of the cross-coupled product **2** the subsequent steps of transmetalation and reductive elimination must proceed with net retention. Since reductive elimination from palladium has been shown[14,17] to proceed with retention, the stereochemistry is most likely fixed (retention) in the transmetalation step. This stereochemistry is consistent with that observed in the palladium-catalyzed coupling of the cis allyl ester with phenyl and hexenyl allanes.[18]

Acknowledgment. This research was supported by Grants CHE-8003336 and CHE-8305468 from the National Science Foundation. The 360-MHz NMR spectra were obtained at the Colorado State University Regional NMR Center, funded by the National Science Foundation Grant CHE-78-18581.

Supplementary Material Available: Listing of spectral data and elemental analyses (1 page). Ordering information is given on any current masthead page.

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(15) Oxidative additions to Pd(0) take place with inversion in the case of allylic esters (Trost, B. M.; Weber, L. *J. Am. Chem. Soc.* **1975**, *97*, 1611) and benzyl chloride (Wong, P. K.; Lau, K. S. Y.; Stille, J. K. *Ibid.* **1974**, *96*, 5956).

(16) Hines, L. F.; Stille, J. K. *J. Am. Chem. Soc.* **1972**, *94*, 485.

(17) Milstein, D.; Stille, J. K. *J. Am. Chem. Soc.* **1979**, *101*, 4981.

(18) Matsushita, H.; Negishi, E. *J. Chem. Soc., Chem. Commun.* **1982**, 160.

Direct Conversion of Organic Halides to Aldehydes with Carbon Monoxide and Tin Hydride Catalyzed by Palladium

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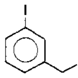
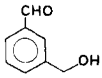
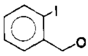
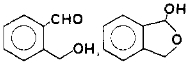
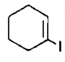
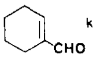
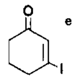
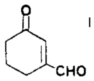
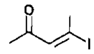
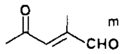
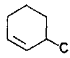
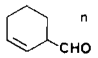
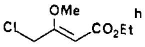
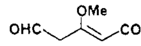
Although the uncatalyzed reduction of acid chlorides to aldehydes by tributytin hydride yields a mixture of aldehydes and esters,[1] the introduction of a palladium catalyst directs the reduction under mild conditions nearly exclusively to the aldehyde in high yield, even in the presence of other reducible groups.[2] The suggested mechanism[2] for the catalytic reaction involved sequential rapid oxidative addition of the acid chloride to the palladium(0) catalyst followed by transmetalation with tin hydride, to yield an acylpalladium hydride, and reductive elimination.

These results suggested that an organic halide could be catalytically converted directly to an aldehyde in the presence of carbon monoxide and tributyltin hydride. The transformation of a variety of organic halides to aldehydes does in fact take place with these reagents under mild reaction conditions (1-3 atm of CO, 50 °C) to give good yields of aldehydes (Table I).

$$\text{RX} + \text{CO} + n\text{-Bu}_3\text{SnH} \xrightarrow[50\,^\circ\text{C}]{\text{Pd(0)}} \text{RCHO} + n\text{-BuSnX}$$

Aryl, benzyl, vinyl, and allyl halides can be converted to aldehydes, and other functional groups in the molecule (nitro, ketone, ester, and alcohol) remain unaffected under these reaction conditions. Closely related to this reaction is the palladium-catalyzed conversion of vinyl or aryl halides to aldehydes by carbon monoxide and hydrogen (1:1); this reaction, however, requires higher temperatures ($\sim$100 °C) and pressures (1200-1500 psi).[3]

Table I. Formylation of Organic Halides[a]

halide	solvent	*P* CO, atm	product(s)	% yield[b]
C_6H_5I	toluene	1	C_6H_5CHO	95[c]
p-MeC_6H_4I	"	1	p-MeC_6H_4CHO	100
o-MeC_6H_4I	"	1	o-MeC_6H_4CHO	70
p-BrC_6H_4I	THF	3	p-BrC_6H_4CHO	88 (70)
			C_6H_5Br	9
			C_6H_6	4
p-$MeOC_6H_4I$	toluene	1	p-$MeOC_6H_4CHO$	100 (77)
p-$NO_2C_6H_4I$	"	3	p-$NO_2C_6H_4CHO$	38
			$C_6H_5NO_2$	62
	"	1	 [i]	(76)
			$C_6H_5CH_2OH$	12
	"	1	 [j]	(55)
			$C_6H_5CH_2OH$	20
$C_6H_5CH_2Br$	THF	1	$C_6H_5CH_2CHO$	75
			$C_6H_5CH_3$	12
 [d]	toluene	1	 [k]	89 (53)
 [e]	"	3	 [l]	83
 [f]	"	3	 [m]	95
 [g]	"	3	 [n]	65
 [h]	THF	3	 [n]	86

[a] Reactions were run at 50 °C with tetrakis(triphenylphosphine)palladium(0), 3.5-4 mol %. All compounds gave satisfactory spectra (IR, ^{1}H NMR, ^{13}C NMR) and GC retention times by comparison to authentic samples or known compounds. [b] Yields determined by GC; isolated yields in parentheses. [c] The same yield was obtained with either tetrakis(triphenylphosphine)palladium(0) or bis(dibenzylidene acetone)palladium (3.7 mol %) plus 8.7 mol % triphenylphosphine. [d] Pross, A.; Sternhall, S. *Aust. J. Chem.* 1970, *23*, 989. [e] Piers, E.; Grierson, J. R.; Lau, C. K.; Nagakura, I. *Can. J. Chem.* 1982, *60*, 210. [f] Piers, E.; Nagakura, I. *Synth. Commun.* 1975, 193. [g] Grob, C. A.; Knu, H.; Gagneux, A. *Helv. Chim. Acta* 1957, *40*, 130. [h] DuBois, G. E.; Crosby, G. A.; Stephenson, R. A. *J. Med. Chem.* 1981, *24*, 408. [i] Leznoff, C. C.; Wong, J. Y. *Can. J. Chem.* 1973, *51*, 3756. [j] Rieche, A.; Schultz, M. *Justus Liebigs Ann. Chem.* 1962, *653*, 32. [k] Kraus, J. L.; Sturtz, G. *Bull. Soc. Chim. Fr.* 1971, *11*, 4012. [l] Quesada, M. L.; Schlessing, R. H. *Synth. Commun.* 1976, *6*, 555. [m] This compound has the correct spectra (NMR, IR) and analysis. [n] The product of reaction by NMR. However, the double bond isomerizes in conjugation with aldehyde function on workup.

The reaction with tin hydride and carbon monoxide has the advantage over a sequence in which a main group organometallic such as an organolithium or Grignard reagent is used to convert organic halides to aldehydes in that other reactive functionality may be present. The use of disodium tetracarbonylferrate to convert aliphatic halides and sulfonates to aldehydes is limited to substrates that do not contain other groups susceptible to reaction with the strong ferrate nucleophile.[4] The tin hydride reaction has the advantage that an acid chloride is not the sub-

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