made an interpretation of the coupling impossible.

Experimental Section

For NMR spectroscopy samples were prepared as approximately 10% (w/v) solutions in CD_3CN and 3% (w/v) in Me_2SO-d_6, with Me_4Si added as an internal standard. ^1H spectra were obtained on a Bruker HX-270S spectrometer in the FT mode by using quadrature detection. For NOE measurement the 5-mm-o.d. sample tube was degassed by several freeze–pump–thaw cycles before sealing. The NOE experiment was performed by using a spectral width of 4000 Hz and 16K data points. The on-line- and off-line-irradiated spectra were sequentially obtained and stored on disk. The delay between each scan was 5 s and the saturation period 2 s. The total number of scans accumulated was 7200. The NOE effect was determined by subtraction from the two FIDs of a weight factor (1.*xx*), leading to the disappearance of the enchanced signal having *xx*% NOE effect.

The SPT (selective population transfer) ^{13}C spectra were obtained on the same instrument. Inversion of proton signals was effected by 50-ms pulses. The spectral width was 20 000 Hz, 32 K data points were used, and 2000 scans were accumulated with a delay of 5 s between each scan.

^1H NMR data of **1** (CD_3CN): δ 5.64 [br s, H(6) and H(3)], 5.49 [t, J = 3 Hz, H(8)], 5.38 [t, J = 4 Hz, H(2)], 4.19 [br s, H(1)], 2.90 [dd, J = 15, 3 Hz, H(9)], 1.77 [br s, H(15)], 1.33 and 1.31 [2 s, H(13) and H(14)]. The signals from the acyl moieties, found as expected, cover the signal due to one of the protons attached to C(9).

Periodic acid treatment was performed by leaving a methanolic solution of equal amounts of compound and periodic acid at room temperature for 18 h. After addition of water the mixture was extracted with ether. The residue obtained by concentration of the organic layer was investigated by ^1H NMR or HPLC over Lichrosorb RP 18 [7 μm; eluent, MeOH–H_2O(3:1); 4600 theoretical plates calculated for **4**].

Butanoylation of 5 was performed by leaving a solution of **5** (8 mg), butyric anhydride (20 μL), and 4-(dimethylamino)-pyridine (7 mg) in methylene chloride (2 mL) for 45 min. The organic layer obtained after addition of 2 M hydrochloric acid (10 mL) and ether (10 mL) followed by occasional shaking for 10 min was washed with 0.5 M aqueous sodium carbonate, water, and 0.5 M hydrochloric acid and concentrated to give an oil, from which **6** (5 mg) was isolated by column chromatography over silica gel [60–80 mesh; eluent; toluene–EtOAc (3:1)]: ^1H NMR data ($CDCl_3$) δ 5.88 [br s, H(6)], 5.69 [br s, H(3)], 5.41 [dd, J = 4, 5 Hz, H(2)], 5.18 [dd, J = 14, 4 Hz, H(8)], 3.72 [br s, H(1)], 2.85 [dd, J = 14, 4 Hz, H(9)], 1.90 [br s, H(15)], 1.45 [s, H(14)]. The signals from the acyl moieties, found as expected, cover the signals due to Me(11) and due to one of the protons attached to C(9).

Horeau analyses were performed by leaving a solution of **4** (5 mg) and optically inactive α-phenylbutyric anhydride (20 μL) in either pyridine (0.5 mL) or a 0.5% solution of 4-(dimethylamino)pyridine in methylene chloride (1 mL). After an adequate period of time [2 days for the pyridine-catalyzed reaction, 1 h for the 4-(dimethylamino)pyridine-catalyzed reaction at room temperature, 4 h at 0 °C, 16 h at −23 °C, and 9 days at −78 °C], the solution was mixed with water and stirred for a further 2 h. After addition of ether (5 mL) the organic layer was separated, washed with 0.5 M aqueous sodium carbonate (5 mL) and 0.25 M hydrochloric acid (5 mL), dried, and concentrated to yield a residue, which was dissolved in methylene chloride (5 mL). A sample (5 μL) of this solution was investigated by HPLC over Polygosil 60-5 [120 × 4.6 mm, eluent 2-propanol–hexane (2:98), detection 220 nm, flow rate 1 mL/min, 1600 teoretical plates as calculated for the ester of **4** and (*S*)-α-phenylbutyric acid]. Retention times: the ester of **4** and (*R*)-α-phenylbutanoic acid, 9.4 min; the ester of **4** and (*S*)-α-phenylbutanoic acid, 10.8 min. The ratio of (*R*)-α-phenylbutanoate/(*S*)-α-phenylbutanoate for the pyridine-catalyzed reaction was 3:2, and those for the 4-(dimethylamino)pyridine-catalyzed reaction were as follows: room temperature, 5:4; 0 °C, 4:3; −23 °C, 3:2; −78 °C, 3:1.

^1H NMR data of **4** esterified with (*R*)-α-phenylbutyric acid (0.75 mg in 400 μL of $CDCl_3$): δ 5.69 [br s, H(6) or H(3)], 5.55 [t, J = 3 Hz, H(8) or H(2)], 5.54 [H(6) or H(3)], 5.35 [H(8) or H(2)], 4.16 [br s, H(1)], 2.92 [dd, J = 12, 3 Hz, H(9)], 1.85 [s, H(15)], 1.49 [s, H(14)]. The signals from the acyl moieties, found as expected, cover the signals due to Me(11) and due to one of the protons attached to C(9).

^1H NMR data of **4** esterified with (*S*)-α-phenylbutyric acid (0.25 mg in 400 μL of $CDCl_3$): δ 5.69 [br s, H(3) or H(6)], 5.54 [H(8) or H(2)], 5.43 [H(8) or H(2)], 5.35 [H(3) or H(6)], 4.12 [br s, H(1)], 2.91 [dd, J = 12, 3 Hz, H(9)], 1.81 [s, H(15)], 1.39 [s, H(14)], 1.20 [s, H(13)]. The signals from the acyl moieties, found as expected, cover the signal due to one of the protons attached to C(9).

The reaction between 4 and (*S*,*S*)-α-phenylbutyric anhydride ($[\alpha]^{23}_D$ +123° (*c* 0.1, C_6H_6)) was performed as described above for the 4-(dimethylamino)pyridine-catalyzed reaction between the optically inactive anhydride and **4** at room temperature. Approximately 8% of the *R* ester could be detected in the product by HPLC.

Acknowledgment. We are grateful to the Danish Natural Science Research Council for NMR facilities and to the Danish Medical Research Council for HPLC facilities.

Registry No. **1**, 67526-95-8; **2**, 67526-94-7; **4**, 80048-99-3; **4** (*S*)-α-phenylbutanoic acid ester, 84074-11-3; **4** (*R*)-α-phenylbutanoic acid ester, 84074-12-4; **5**, 80063-01-0; **6**, 84074-13-5; α-phenylbutyric anhydride, 1519-21-7; (*S*,*S*)-α-phenylbutyric anhydride, 16906-38-0.

Palladium-Mediated Reaction of Enol Ethers with Organomercuric Acetates

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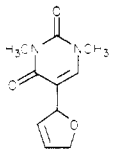
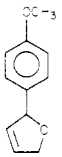
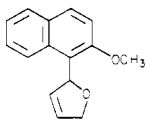
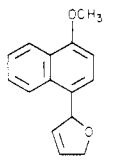
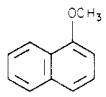
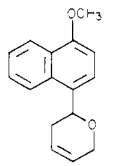
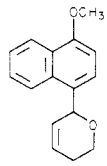
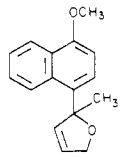
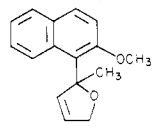
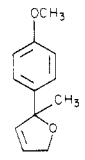
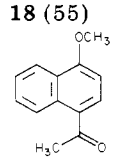
Regiospecific palladium-mediated coupling of heterocyclic or arylmercuric salts with cyclic or acylic enol ethers and acetates has been reported.[2-11] We have now extended the study of this coupling reaction utilizing selected reactant arylmercuric acetates and enol ethers to explore the scope of the reaction and to examine reaction parameters such as cyclic enol ether ring size, steric factors in both enol ether and organometallic reactants, and reaction solvent effects. For this study, the mercuric acetates used were (see Chart I) [1,3-dimethyl-2,4(1*H*,3*H*)-dioxopyrimidin-5-yl]mercuric acetate[2,3,6,7] (**1**), (4-methoxyphenyl)mercuric acetate[12] (**2**), (2-methoxynaphthyl)-mercuric acetate (**3**), and (4-methoxynaphthyl)mercuric acetate (**4**); the enol ethers used were 2,3-dihydrofuran (**5**), 3,4-dihydro-2*H*-pyran (**6**), 5-methyl-2,3-dihydrofuran (**7**), and *n*-butyl vinyl ether (**8**). All reactions utilized stoichiometric palladium acetate and were carried out at room temperature. The results of the study are summarized in Tables I and II.

(1) (a) Cetus Corp, 600 Bancroft Way, Berkeley, CA 94710. (b) Department of Chemistry, Lehigh University, Bethlehem, PA 18015.
(2) Arai, I.; Daves, G. D., Jr. *J. Am. Chem. Soc.* **1978**, *100*, 287.
(3) Arai, I.; Daves, G. D., Jr. *J. Org. Chem.* **1978**, *43*, 4110.
(4) Arai, I.; Daves, G. D., Jr. *J. Org. Chem.* **1979**, *44*, 21.
(5) Arai, I.; Daves, G. D., Jr. *J. Heterocycl. Chem.* **1978**, *15*, 351.
(6) Arai, I.; Lee, T. D.; Hanna, R.; Daves, G. D., Jr. *Organometallics* **1982**, *1*, 742.
(7) Arai, I.; Daves, G. D., Jr. *J. Am. Chem. Soc.* **1981**, *103*, 7683.
(8) Heck, R. F. *J. Am. Chem. Soc.* **1968**, *90*, 5535.
(9) Heck, R. F. *Organomet. Chem. Synth.* **1972**, *1*, 1455.
(10) Danno, S.; Moritani, I.; Fujiwara, T. *Tetrahedron* **1969**, *25*, 4819.
(11) Kasahara, A.; Izumi, T.; Fukuda, N. *Bull. Chem. Soc. Jpn.* **1977**, *50*, 551.
(12) Sipos, J. C.; Sawatsky, H.; Wright, G. F. *J. Am. Chem. Soc.* **1955**, *77*, 2759.

0022-3263/83/1948-0399$01.50/0

Table I. Coupling Reactions of Organomercuric Acetates with Enol Ethers and Stoichiometric Palladium Acetate in Acetonitrile at 24 °C for 24 h

aryl-mercuric acetate	enol ether	products (yield, %)
1	5	 9 (86)
2	5	 10 (80)
3	5	 11 (77)
4	5	 12 (90)  13 (7)
4	6	 14 (75)  15 (15)
4	7	 16 (54)
3	7	 17 (27)
2	7	 18 (55)
4	8	 19 (75)

Most noteworthy is the occurrence, in every instance, of regiospecific formation of a carbon–carbon bond between the aryl carbon bearing mercury and the enol ether olefinic carbon bonded to oxygen. This coupling reaction

Chart I

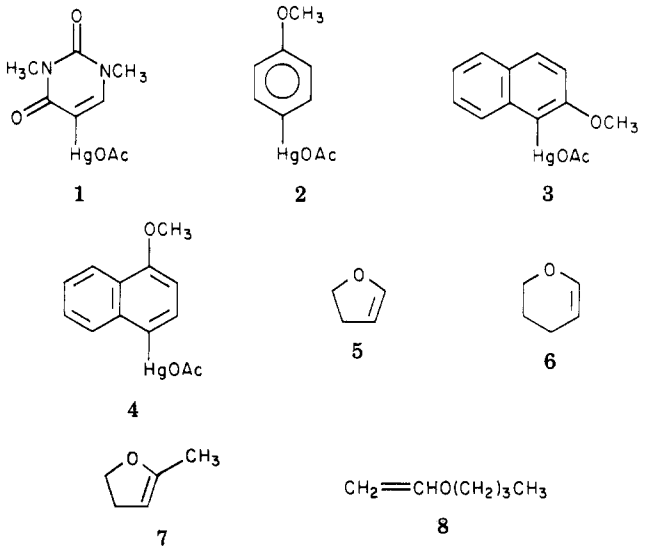


Table II. Effect of Solvent on Palladium Acetate (Stoichiometric) Mediated Coupling of (4-Methoxy-1-naphthyl)mercuric Acetate (4) with 2,3-Dihydrofuran (5)

	yields of products, %	
reaction solvent	12	13
acetonitrile	90	7
dichloromethane	55	31
tetrahydrofuran	66	27
methanol	27	14
acetic acid	15	

is under electronic control owing to the strongly polarized nature of the enol ether olefinic bond and differs from palladium-catalyzed reactions of less polarized olefins for which the regiochemistry appears to be determined by steric factors.[13] In the present study the primacy of electronic effects is most striking in reactions of 5-methyl-2,3-dihydrofuran (**7**) in which the new carbon–carbon bond is formed exclusively with the highly hindered tertiary carbon adjacent to the enol ether oxygen (i.e., formation of **16**–**18**, Table I). Although in reactions of **7** yields were lower [especially in the case of coupling with (2-methoxynaphthyl)mercuric acetate (**3**), in which both reaction centers are crowded] than for more sterically accessible enol ethers, no trace of a coupled product of opposite regiochemistry was detected.

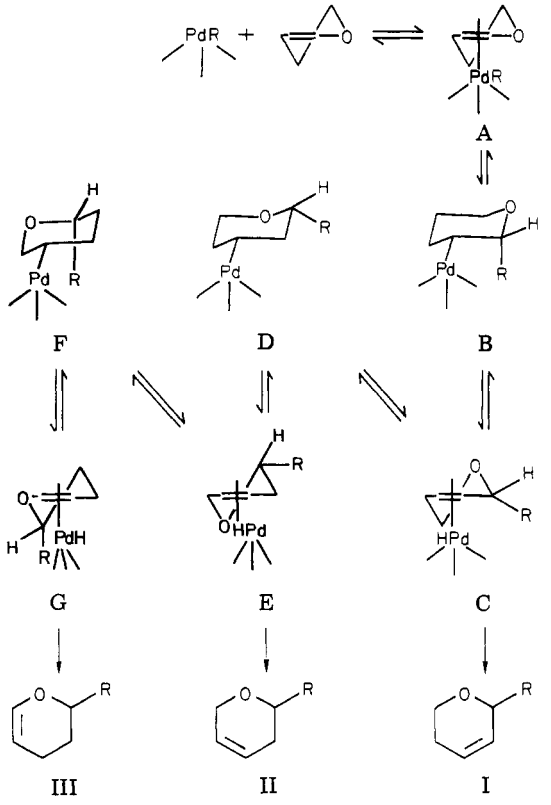
Reactions of 3,4-dihydro-2*H*-pyran (**6**) yielded two products isomeric with respect to the position of the carbon–carbon double bond of the cyclic ether (e.g., **14** and **15**). In earlier studies[3,4] using a higher reaction temperature (100 °C as opposed to 25 °C in the present study) a third double bond isomer (i.e., the cyclic enol ether with the aryl group bonded to the nonolefinic oxygen-bearing carbon) was isolated. The fourth isomer (with an enolic double bond at the substituted carbon) has not been detected. These palladium-catalyzed coupling reactions of 3,4-dihydro-2*H*-pyran which give rise to isomeric products are formulated in Scheme I[14] and are illustrative of the processes and intermediates involved in both the palladium-mediated coupling and double bond isomerization reactions.

(13) Collman, J. P.; Hegedus, L. S. "Principles and Applications of Organotransition Chemistry"; University Sciences books: Mill Valley, CA, 1980; pp 580–582.
(14) Heck, R. F. *J. Am. Chem. Soc.* **1971**, *93*, 6896.

Table III. Mass Spectrometric and ^1H Nuclear Magnetic Resonance Data for Products Formed by Palladium-Mediated Coupling of Organomercuric Acetates 1-4 with Enol Ethers 5-8

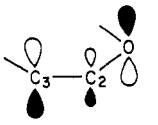
compd	MS, m/z of M^+ (calcd)	^1H NMR ($CDCl_3$), δ
9	208.0862 (208.0845)	3.31, 3.36 (NMes), 4.62-4.77 (OCH_2), 5.69 (OCH), 5.96 (olefinic), 7.15 (pyrimidinyl)
10	176.0846 (176.0838)	3.78 (OCH_3), 4.78 (OCH_2), 5.63-6.14 (OCH, olefinic), 6.75-7.33 (aryl)
11	226.0102 (226.0994)	3.91 (OCH_3), 4.86-5.08 (OCH_2), 6.04 (olefinic), 6.83 (OCH), 7.13-8.27 (aryl)
12	226.0100 (226.0994)	3.96 (OCH_3), 4.78-4.93 (OCH_2), 5.98-6.87 (OCH, olefinic), 7.20-8.42 (aryl)
14	240.1163 (240.1150)	2.22-2.38 (CH_2), 3.64-4.08 (OCH_2), 3.97 (OCH_3), 5.68-6.25 (olefinic), 6.73 (OCH), 7.32-8.38 (aryl)
15	240.1165 (240.1150)	
16	240.1162 (240.1150)	1.87 (CH_3), 3.98 (OCH_3), 4.76 (OCH_2), 5.92, 6.48 (olefinic), 6.71, 7.35-8.52 (aryl)
17	240.1165 (240.1150)	1.82 (CH_3), 3.88 (OCH_3), 4.63 (OCH_2), 5.85, 6.80 (olefinic), 7.1-8.25 (aryl)
18	190.0987 (190.0994)	1.60 (CH_3), 3.78 (OCH_3), 4.63 (OCH_2), 5.76-6.03 (olefinic), 6.67-7.45 (aryl)
19	200.0839 (200.0837)	2.62 (CH_3), 4.00 (OCH_3), 6.55-9.08 (aryl)

Scheme I. Reaction of 3,4-Dihydro-2*H*-pyran with an Arylpalladium Reagent (Formed in Situ)[a]



[a] Heck[14] formulated similarly palladium-mediated arylation reactions of cyclic olefins which also yield mixtures of double bond position isomers.

The arylpalladium reagent, formed in situ from the corresponding arylmercuric salt by transmetalation, forms a π complex (A, Scheme I) with the enol ether double bond. Collapse of the π complex by insertion of the olefin into the Pd–aryl carbon bond produces an adduct (B) in which the resulting σ-bonded palladium and aryl substituents are cis. The regiospecificity of this reaction is a result of interaction of the electrophilic palladium center with the highest occupied molecular orbital (HOMO) of the enol ether (**20**).[15] In the HOMO for the enol ether[15] the site



20

of greatest electron density is C-3 which forms a bond to palladium; C-2, which is electron deficient, forms a bond with the electron-rich aryl carbon. It is noteworthy that thio enol ethers, in which the sulfur π electrons (3p) are much less effective in interaction with the olefinic system (2p), react with opposite regiochemistry,[16] presumably owing to steric factors. Similarly, *N*-vinyl amides in which the nitrogen lone pair is cross conjugated with the olefinic carbons of the vinyl group and with the amide carbonyl give a mixture of products and exhibits little regioselectivity.[17]

The σ-palladium-bonded adduct B is unstable and undergoes facile syn β-hydride elimination to form a new π complex, C, which can (a) irreversibly decompose to coupled product (I) and an unstable hydridopalladium species or (b) experience reinsertion of the olefinic unit into the Pd–H bond to form the original (B) or an isomeric (D) σ-Pd-bonded adduct. Decomposition of this new adduct can produce an isomeric coupled product (II) or further isomerize to produce, finally, the third accessible isomer III.[3]

Several points concerning the isomerization process are noteworthy. The process involves an interconverting set of σ adducts and π complexes in which palladium is continuously bound to the same face of the cyclic ether. There is little regioselectivity associated with the syn β-hydridopalladium elimination and readditions involving nonenolic carbon–carbon double bonds. Decomposition of π complex and release of a hydridopalladium species is irreversible since this species is unstable and rapidly undergoes reductive elimination to form unreactive Pd(0). As a result, the fourth possible isomer (with the original enolic double bond restored) is not formed since this would require release of the hydridopalladium species from the olefin, its migration to the opposite face of the cyclic ether ring, and recomplexation. In acyclic systems (e.g., 8), rotation about the C-2,C-3 bond of the adduct places the C-2 hydrogen cis to palladium, permitting restoration of the enol ether bond.

The palladium-mediated reaction of (4-methoxy-1-

(15) Fleming, I. "Frontier Orbitals and Organic Chemical Reactions"; Wiley: New York, 1976; p 44.

(16) Trost, B. M.; Tanigawa, Y. *J. Am. Chem. Soc.* **1979**, *101*, 4743.
(17) Zeigler, C. B., Jr.; Heck, R. F. *J. Org. Chem.* **1978**, *43*, 2949.

naphthyl)mercuric acetate (4) with 2,3-dihydrofuran (5) was carried out in a variety of solvents (Table II). The results establish that the reaction proceeds in both protic and aprotic solvents although in protic solvents (methanol and acetic acid) the yields are quite low. A side product formed in every instance is 1-methoxynaphthalene (13). In acetonitrile solvent, in which the desired arylated dihydrofuran 12 is produced in 90% yield, 13 occurs to the extent of only 7%; in dichloromethane and tetrahydrofuran much larger quantities of 13 are produced at the expense of 12.[18]

Experimental Section

General Comments. NMR spectra were obtained with deuteriochloroform solution by using a JEOL FX90Q spectrometer. Mass spectra were recorded by using either a CEC (Du Pont) 21-110 or a Du Pont 21-491 mass spectrometer. High-resolution measurements were carried out by Dr. T. Wachs, Cornell University, using an AEI MS-902 spectrometer. Column chromatography was performed by using the method of Still.[19] High-pressure liquid chromatography was performed by using a Waters Associates instrument and octadecylsilane columns with methanol–water mixtures as eluants.

(4-Methoxyphenyl)mercuric Acetate (2). Mercuric acetate (31.8 g, 0.1 mol) was dissolved in 200 mL of methanol by heating. To this solution were added 32.4 g (0.3 mol) of anisole and 0.5 mL of perchloric acid with stirring. After 2 days, the precipitate which formed was removed by filtration. Overnight refrigeration of the filtrate resulted in an additional crop. The combined crude product was recrystallized from methanol to yield 13.4 g (37%) of (4-methoxyphenyl)mercuric acetate[12] (2).

(4-Methoxynaphthyl)mercuric Acetate (4). A mixture of 1-methoxynaphthalene (15.8 g, 0.1 mol), mercuric acetate (31.8 g, 0.1 mol), and 0.5 mL of perchloric acid in 200 mL of methanol was stirred at room temperature for 2 days. The precipitated product was collected by filtration and recrystallized from toluene to yield 36 g (87%) of (4-methoxynaphthyl)mercuric acetate (4): mp 222 °C; ^{1}H NMR ($CDCl_3$) δ 2.12 (OAc), 4.00 (OCH_3), 6.80 (d, C-3 H, enhanced upon irradiation of OCH_3 resonance), 7.33 (d, C-2 H). Anal. Calcd for $C_{13}H_{12}HgO_3$: C, 37.5; H, 2.90. Found: C, 37.6; H, 2.84.

(2-Methoxy-1-naphthyl)mercuric Acetate (3). A mixture of 2-methoxynaphthalene (5 g, 0.03 mole, mercuric acetate (9.8 g, 0.03 mol), and 0.5 mL of perchloric acid in 75 mL of methanol was heated until solution was achieved and then stirred for 7 days. The mixture was then cooled in an ice bath, and precipitated crude product was collected by filtration and recrystallized from toluene to yield 9.5 g (74%) of (2-methoxy-1-naphthyl)mercuric acetate: mp 106 °C dec; ^{1}H NMR ($CDCl_3$) δ 2.21 (OAc), 3.75 (OCH_3), 7.20 (d, C-3 H, enhanced upon irradiation of OCH_3 resonance). Anal. Calcd for $C_{13}H_{12}HgO_3$: C, 37.5; H, 2.90. Found: C, 37.2; H, 2.88.

Procedure for Palladium-Mediated Coupling of Arylmercuric Acetates with Enol Ethers. To a suspension of 0.12 mmol of an arylmercuric acetate and 0.12 mmol of palladium acetate in 5 mL of acetonitrile was added 0.24 mmol of an enol ether. The resulting mixture was stirred at room temperature for 24 hours and then filtered through Celite. The solvent was evaporated from the filtrate under reduced pressure, and the residue was separated by preparative thin-layer chromatography on silica gel with chloroform. Product yields are in Table I and characterizing spectrometric data are recorded in Table III.

In Table II are yields obtained in coupling of 4 and 5 under conditions identical except that various reaction solvents were used.

Acknowledgment. This research was supported by a grant from the National Institute of General Medical Science (GM 30310).

(18) Changes in palladium and mercuric salt counterions can also dramatically affect reaction course. See ref 6 and: Arai, I.; Hanna, R.; Daves, G. D., Jr. *J. Am. Chem. Soc.* **1981**, *103*, 7684.

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Registry No. 1, 65904-27-0; 2, 5780-90-5; 3, 84132-72-9; 4, 84132-73-0; 5, 1191-99-7; 6, 110-87-2; 7, 1487-15-6; 8, 111-34-2; 9, 84143-13-5; 10, 84132-74-1; 11, 84132-75-2; 12, 84132-76-3; 13, 2216-69-5; 14, 84132-77-4; 15, 84132-78-5; 16, 84132-79-6; 17, 84132-80-9; 18, 84132-81-0; 19, 24764-66-7; mercuric acetate, 1600-27-7; anisole, 100-66-3; 1-methoxynaphthalene, 2216-69-5; 2-methoxynaphthalene, 93-04-9; palladium acetate, 19807-27-3; acetonitrile, 75-05-8; dichloromethane, 75-09-2; tetrahydrofuran, 109-99-9; methanol, 67-56-1; acetic acid, 64-19-7.

Preparation of Oxygen-18-Labeled *m*-Chloroperoxybenzoic Acid

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During the course of our investigation[2] of oxygen transfer from oxaziridines we required a means of preparing these oxaziridines with an ^{18}O label. This was seen as an opportunity for the development of methodology suitable for the preparation of a wide variety of ^{18}O-labeled compounds.[3,4]

We report here a convenient synthesis of ^{18}O-labeled *m*-chloroperoxybenzoic acid which utilizes $^{18}O_2$ as the commercially available isotope source. Oxygen gas, 50% enriched in ^{18}O, was used in 29% efficiency to produce the peroxy acid 39% enriched in ^{18}O at the active oxygen position.[5]

The apparatus illustrated in Figure 1 was constructed for the oxidation of sodium metal by $^{18}O_2$ at elevated temperatures to yield $Na_2{}^{18}O_2$. Several runs with unlabeled O_2 consistently resulted in peroxide containing 70–80% active oxygen. The sodium peroxide was allowed to react with *m*-chlorobenzoyl chloride[5] to yield the desired peroxy acid containing 99% active oxygen. This reagent was found to be efficient in label transfer in the oxidation of diverse substrates (Table I).

Experimental Section

General Methods. ^{1}H NMR spectra were obtained on a Bruker WM-270 (270 MHz) NMR spectrometer. Chemical shifts are reported downfield from tetramethylsilane (Me_4Si, internal reference) on the δ scale. Infrared spectra were recorded on a Perkin-Elmer 283B grating infrared spectrometer. GLC/MS analyses were performed on a Varian MAT-44 mass spectrometer interfaced to a Varian Series 1400 gas–liquid chromatograph using He as carrier gas (8 ft × $^1/_8$ in., 4.1% Carbowax column on Chromosorb G). Melting points were obtained on a Thomas-Hoover capillary melting point apparatus and are uncorrected. Labeled O_2 was obtained from Cambridge Isotope Laboratories, Inc.

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(3) ^{18}O-Labeled epoxides have been efficiently prepared. See: Hanzlik, R. P.; Edelman, M.; Michaely, W. J.; Scott, G. *J. Am. Chem. Soc.* **1976**, *98*, 1952.

(4) ^{18}O-Labeled hydrogen peroxide has been prepared. See: Ball, R. E.; Edwards, J. O.; Jones, P. *J. Inorg. Nucl. Chem.* **1966**, *28*, 2458.

(5) The 39% value was determined from the intensities of the M^+ peaks at *m/e* 174, 176, and 178 adjusted for the presence of two Cl isotopes (see mass spectroscopic data in the Experimental Section).

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