Table I. Isomerizations of (*E*)-1,2-Diphenylpropene (4)

reagent	ratio[a]	solvent	temp, °C	time, h	yield, % 3	4
MTPI	2.5	HMPA	75	1.0	0	100
MTPI[c]	5.0	HMPA	80	3.0	29[c]	71[c]
HI	1.0	HMPA	80	3.0	0	100
HI	1.0	HOAc	113	1.0	12[d]	41[d]
HI	4.0	HOAc	113	2.0	0[e]	0[e]
HI	0.1	HOAc	113	3.0		75
HBr[3a]	1.22	HOAc	100	2.0	(21)	(77)
HBr[5]	0.06	HOAc	100	1.5	(14)	(81)
TsOH[2]	0.05	HOAc	75	9.5	(3)[f]	(96)[f]
TsOH[4]	0.04	xylene	138	20.0		73
TsOH[6]	0.03	PhH	80	24.0		79

[a] Mole ratio of reagent to 4. [b] Results obtained by GC and NMR. Values in parentheses were not supplied in references but obtained in our labs after repetition of literature procedure. Totals of 3 + 4 differ from 100% by amount of impurity which is assumed in most cases to be α-benzylstyrene.[6] [c] Plus 1 equiv of alcohol mixture. Results are not significantly different from those expected. Calculations with data from slightly different reaction conditions[4,10] and assuming that no isomerization of the initial 4 gives 34% and 66% of 3 and 4 respectively. [d] 47% Reduction to 1,2-diphenylpropane (8). [e] Complete reduction to 8, see Experimental Section. [f] Reported[2] 97% of 3 by UV analysis, but the α-benzylstyrene was not considered.

Thomas–Hoover melting point apparatus and are uncorrected. ^{1}H NMR (60-MHz) spectra were measured in $CDCl_3$ and recorded on a Perkin-Elmer R-12-B spectrometer. All chemical shifts are reported in parts per million (δ) downfield from tetramethylsilane (1%) as the internal standard. The ^{13}C NMR spectra were obtained with a Bruker WH-90FT instrument. MS (70 eV) were taken on a DuPont (CEC) 21-110 instrument. High-resolution ^{1}H and ^{13}C NMR spectra (67.9 MHz) were more recently recorded with internal deuterium lock on a JEOL FX-270 superconducting NMR spectrometer. IR spectra were recorded on a Perkin-Elmer 710-A grating spectrophotometer and are reported in micrometers (μm). GC analyses were conducted on a Varian Aerograph 2700 with a 1.5%, OV-101, 100/120 HP-Chromosorb G, 5 ft × $^1/_4$ in. stainless steel column, with an oven temperature of 80 °C and a He gas flow of 50 mL/min. Preparative column chromatography was performed with a 2 ft × 1 in. glass column and MCB 60–200-mesh silica gel with ligroin and ethyl acetate eluents.

(1-Chloroethyl)benzene (9) was prepared by shaking 110 mL (111.5 g, 0.9 mol) of α-methylbenzyl alcohol with 250 mL of concentrated HCl in a separatory funnel for 22 min. The aqueous layer was removed, and an additional 50 mL of concentrated HCl was combined with the organic layer and shaken for 22 min. The aqueous layer was removed and washed 4 successive times with 50 mL of H_2O followed by a wash of 60 mL of 5% $NaHCO_3$. The organic layer was dried with $CaCl_2$ and stored in a brown bottle in the refrigerator. This reaction afforded **9** (115.0 mL, 96%): bp 88–9 °C (34 mm), lit.[9] bp 67 °C (10 mm); ^{1}H NMR δ 1.77 (CH_3, 3 H, d, J = 7), 5.04 (CH_3CH, 1 H, q, J = 7), 7.17–7.52 (Ar H, 5 H, m). The dry material is suitable for reaction and does not require distillation.

***threo*- and *erythro*-1,2-diphenylpropanol (1 and 2)** were prepared by combining 57 mL (60.53 g, 0.43 mol) of **9** with 45.75 mL (47.76 g, 0.45 mol) of benzaldehyde in an addition funnel fitted to a three-necked round-bottomed flask containing 200 mL of THF and lithium wire (5.99 g, 0.86 mol). The flask was immersed in an ice/water bath at 0–5 °C, under an Ar atmosphere, and the mixture in the addition funnel was slowly added (~3 h) to the magnetically stirred flask. After the addition was completed, the temperature was allowed to slowly equilibrate to room temperature while stirring over 18 h. To the resulting mixture was added 250 mL of 5% HCl which provided separation of an organic layer. The organic layer was washed with 100 mL of 10% $NaHSO_3$, diluted with 150 mL of CH_2Cl_2, washed several times with 120 mL of H_2O, and dried with Na_2SO_4. Removal of solvent by vacuum afforded a yellow oil which on recrystallization with 100% EtOH resulted in crystals of *meso*-2,3-diphenylbutane (**7**): mp 121–2 °C (lit.[5] mp 124–5 °C); ^{1}H NMR δ 1.02 (CH_3, 6 H, d, J = 6), 2.66–2.97 ($CHCH_3$, 2 H, m), 7.05–7.40 (Ar H, 10 H); IR 3.40–3.55 (CH), 6.65, 6.85, 7.10, 13.5, and 14.5 (Ar); MS, 210 (12.0), 178 (21.5), 115 (31.6), 105 (100), 91 (51.5), 77 (64.3); ^{13}C NMR δ 21.01 (CH_3), 47.34 (CH), 126.16, 127.73, 128.38, 146.65. The resulting oil was preparatively chromatographed, in three portions, to remove the remaining dimer, 7.4 g (total, 16.4%).

Further elution afforded the alcohols **1** and **2** as a viscous oil: 54.0 g (59.3%); ^{1}H NMR analysis indicated a 1:1 mixture.

***erythro*-1-Iodo-1,2-diphenylpropane (5)** was prepared by reacting 2.11 g (10 mmol) of the alcohol mixture (**1** and **2**) and 11.22 g (50 mmol) of HI (57%) while stirring for 46 h at 25 °C under an Ar atmosphere. Ice was added to the resulting mixture which was then treated with $NaHSO_3$ to destroy free iodine, mixed with 100 mL of 5% NaOH, and extracted with 100 mL of ligroin. The organic layer was washed 3 times with 100-mL portions of H_2O, dried over Na_2SO_4, and evaporated to afford **5**: (2.455 g, 76%), mp 130–2 °C (lit.[3c] mp 130–1 °C, see note 8); ^{1}H NMR δ 1.133 (CH_3, 3 H, d, J = 7.32), 3.3–4.8 (PhC*H*) 1 H, m) 5.170 (PhIC*H*, 1 H, d, J = 10.99), 6.72–7.58 (Ar H, 10 H, m); ^{13}C NMR δ 19.9 (CH_3), 41.9 (PhCHI), 49.5 ($PhCHCH_3$).

1,2-Diphenylpropane (8). α-Methylstilbene (**4**) (199 mg, 1 mmol) and excess HI (898 mg, 4 mmol) (57%) were refluxed in 10 mL of glacial acetic acid for 2 h under an Ar atmosphere. The resulting mixture was added to ice, treated with $NaHSO_3$, and mixed with 100 mL of 5% NaOH and 100 mL of ligroin. The organic layer was washed 3 times with 100 mL of H_2O, dried over Na_2SO_4, and evaporated to afford 172.3 mg (54%) of **8**: bp 55–78 °C (0.25 mm) (lit.[12] bp 88 °C (0.5 mm)); ^{1}H NMR δ 1.23 (CH_3, 6 H, d, J = 7), 2.65–3.14 (Ar C*H*, 2 H, m), 6.83–7.46 (Ar H, 10 H, m); ^{13}C NMR δ 21.14 (CH_3, q), 41.88 (CH, d), 45.13 (CH_2, t); MS, *m/e* 196 (4.8), 194 (17.9), 179 (24.9), 115 (44.9), 105 (100), 91 (95.6), 77 (82).

(*Z*)-1,2-Diphenylpropene (3) was prepared by reacting **5** (0.896 g, 2.78 mmol), MTPI (2.27 g, 5 mmol), and 6.6 mL of dry HMPA placed in an 80 °C oil bath for 1 h. Workup as above with ice/5% NaOH/10% $NaHSO_3$ was followed by extraction with ligroin. The organic layer was washed 3 times with 100 mL of H_2O and dried with Na_2SO_4. Removal of solvent in vacuum afforded a viscous oil (528 mg, 98%). GC and ^{1}H NMR showed only (*Z*)- and (*E*)-olefins, **3** and **4**, in an 88:12 ratio.

3 was also prepared by (1) reacting **5** (322 mg, 1 mmol) with excess NaOEt in EtOH at reflux for 24 h and (2) reacting **5** (324 mg, 1 mmol) with NaI (28 mg, 0.2 mmol) in 30 mL of HMPA at 80 °C for 3 h to give essentially complete conversion to **3** in both reactions.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the Americal Chemical Society, for the partial support of this research. We also appreciate the help of Dr. Ben Shoulders at The University of Texas at Austin for assistance in obtaining ^{13}C NMR and mass spectra as well as Dr. William D. Vernon of El Paso Products Company, Odessa, TX, who kindly ran the high-resolution ^{1}H and ^{13}C NMR spectra.

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A New and Specific Method for the Protection of Phenols as the Corresponding *tert*-Butyl Ethers

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The protection of alcohols and carboxylic acids via their conversion into the corresponding *tert*-butyl derivatives

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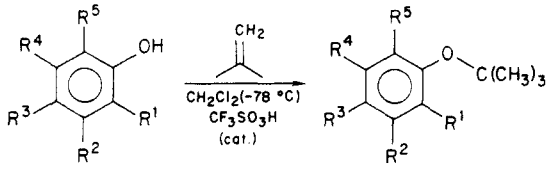
Table I. *tert*-Butylation of Representative Phenols 1

1	R^1	R^2	R^3	R^4	R^5	product	yield, %
1a	OCH_3	H	H	H	H	**2a**	76
1b	H	H	OCH_3	H	H	**2b**	82
1c	H	OCH_2O		H	H	**2c**	71
1d	$CH_2CH{=}CH_2$	H	H	H	H	**2d**	90
1e	H	Br	H	H	H	**2e**	99
1f	H	$C(=O)CH_3$	H	H	H	**2f**	56
1g	OCH_3	H	$CH_2CH{=}CH_2$	H	H	**2g**	70

Scheme I



1 2

has become a widely utilized procedure in organic synthesis.[1] Recently, we required an efficient procedure for the protection of a variety of phenols in the form of their *tert*-butyl ethers. The reaction conditions that are commonly employed to effect the *tert*-butylation of typical hydroxylic compounds involves the exposure of these species to excess isobutylene in the presence of boron trifluoride–phosphoric acid complex[2] or sulfuric acid[3] at 0 to 25 °C. Unfortunately, repeated efforts to achieve the protection of typical phenols by way of the foregoing experimental protocol were complicated by the rapid Friedel–Crafts-type rearrangement of the initially formed ether. In all instances intractable mixtures of nuclear substitution products were produced. In this note we describe a mild and exceedingly efficient method for the protection of phenols as their *tert*-butyl ethers.

In principle, the restoration of the desired selectivity for the *tert*-butylation of phenols could be attained by operating at low reaction temperatures. Efforts to achieve this objective utilizing the standard modes of catalysis (e.g., $BF_3{\cdot}H_3PO_4$ or H_2SO_4)[2,3] were routinely stifled by exceedingly low rates of substrate conversion at temperatures below −35 °C.[4] It was subsequently discovered that the substitution of trifluoromethanesulfonic acid as the catalytic species facilitated the formation of *tert*-butyl ethers from a variety of representative phenols (e.g., 1 → 2, Scheme I) at temperatures as low as −78 °C. No detectable quantities of products arising from nuclear alkylation were observed when these reaction conditions were employed. The results of Table I indicate the generality associated with the aforementioned *tert*-butylation procedure.

It is noteworthy that typical alcohols are derivatized only with great sluggishness under the above set of reaction conditions. Accordingly, 1-dodecanol (**3**) was converted to the corresponding *tert*-butyl ether **4** to the extent of only 2% upon treatment with isobutylene in the presence of trifluoromethanesulfonic acid at −78 °C for 3 h. By way of contrast, the protection of 1-dodecanol (**3**) could be accomplished in 76% isolated yield at higher reaction temperatures (−5 °C, 3 h). It is significant that treatment of a 1:1 mixture of guaiacol (**1a**) and 1-dodecanol (**3**) with isobutylene and a catalytic quantity of trifluoromethanesulfonic acid in CH_2Cl_2 at −50 °C resulted in the *selective* protection of the phenol.

$$\underset{\mathbf{3}}{CH_3(CH_2)_{10}CH_2OH} \xrightarrow[CH_2Cl_2\ (-5\ °C),\ CF_3SO_3H]{CH_2{=}C(CH_3)_2} \underset{\mathbf{4}}{CH_3(CH_2)_{10}CH_2O\text{-}t\text{-}Bu}$$

Initial efforts to achieve the deprotection of the ethers **2a–g** employing trifluoroacetic acid[6] (25 °C, 16 h) afforded complex mixtures containing rearrangement products in addition to the desired phenols **1a–g**. Similar results were obtained when the deprotection was attempted in the presence of $TiCl_4$.[7] After considerable experimentation, the following procedure for the deprotection of the ethers **2a–g** was determined to be optimum. Treatment of a solution of a representative phenyl *tert*-butyl ether in anhydrous trifluoroethanol with a catalytic quantity of trifluoromethanesulfonic acid (−5 °C, 60 s) was found to provide the corresponding phenols in quantitative yield.

Scheme II[a]

$$2 \xrightarrow{a} 1$$

[a](a) CF_3SO_3H (catalyst), CF_3CH_2OH (−5 °C, 60 s)

The method for the protection of phenols as the corresponding *tert*-butyl ethers reported herein avoids the usual problem of nuclear substitution encountered with this reaction. Prominent among the advantages of this new procedure are its economy and convenience.

Experimental Section

***4-tert*-Butoxyanisole (2b).** An oven-dried 100-mL three-necked flask equipped with a magnetic stirring bar, thermometer, and nitrogen inlet was charged with 25 mL of CH_2Cl_2 and cooled to −30 °C through the aid of a dry ice–CH_3OH/H_2O slush bath. To the flask were added 20 mL of liquified isobutylene and 2.48 g (25 mmol) of *p*-methoxyphenol (**1b**). To the vigorously stirred reaction mixture was added 177 μL (2 mmol) of trifluoromethanesulfonic acid. After completion of the addition, the resultant mixture was stirred for an additional 4.0 h at −78 °C, during which time it gradually became homogeneous.[8] Triethylamine, 0.279 g, (2 mmol) was then added, and the reaction mixture was then allowed to warm to room temperature.

The solution was transferred to a 50-mL flask, and the solvents were evaporated. The oily residue was triturated with 100 mL of petroleum ether, the solids were filtered, and the solvent was evaporated. Analysis of the crude product by gas chromatography on a 5% SE 30 column revealed the product to be 92% pure.

This material was purified by vacuum distillation, 50.5–52.5 °C (0.5 torr), to afford 3.15 g (82%) of pure 4-*tert*-butoxyanisole (**2b**) as a colorless liquid: NMR ($CDCl_3$, Me_4Si) δ 1.31 (s, 9 H, $C(CH_3)_3$), 3.73 (s, 3 H, CH_3), 6.80 (m, 4 H, Ar); IR (cm^{-1}) (film) 2770–3100, 1390, 1365. Anal. Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.19; H, 8.98.

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(4) At temperatures above −30 °C under these conditions, rearrangement of the product *tert*-butyl ethers became an insurmountable obstacle.
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(9) Undergraduate Research Assistant (1984–1985).

2-*tert*-Butoxyallylbenzene (2d). An oven-dried 100-mL three-necked flask equipped with a magnetic stirring bar, thermometer, and nitrogen inlet was charged with 25 mL of CH_2Cl_2 and cooled to -78 °C through the aid of a dry ice-acetone bath. To the flask were added 20 mL of liquified isobutylene and 2.68 g (25 mmol) of *o*-allylphenol (**1d**). To the vigorously stirred reaction mixture was added 177 μL (2 mmol) of trifluoromethanesulfonic acid. After completion of the addition, the resultant homogeneous mixture was stirred for an additional 4.0 at -78 °C. Triethylamine, 0.279 g, (2 mmol) was then added, and the reaction mixture was then allowed to warm to room temperature.

The solution was transferred to a 50-mL flask, and the solvents were evaporated. The oily residue was triturated with 100 mL of petroleum ether, the solids were filtered, and the solvent was evaporated. Analysis of the crude product by gas chromatography on a 5% SE 30 column revealed the product to be 98% pure.

The material obtained in this manner was purified by vacuum distillation, 43-45 °C (0.85 torr), to afford 3.46 g (90%) of pure 2-*tert*-butoxyallylbenzene (**2d**) as a colorless liquid: NMR ($CDCl_3$, Me_4Si) δ 1.45 (s, 9, CH_3), 3.38 (d, J = 7, 2 H, CH_2), 5.05 (m, 2 H, vinyl), 5.95 (m, 1 H, vinyl), 7.1 (m, 3 H, Ar); IR (cm^{-1}) (film) 3130-2780, 1390, 1367. Anal. Calcd for $C_9H_{10}O$: C, 82.06; H, 9.00. Found: C, 82.30; H, 9.27).

1-*tert*-Butoxydodecane (4). An oven-dried 100-mL three-necked flask equipped with a magnetic stirring bar, thermometer, and nitrogen inlet was charged with 12.5 mL of CH_2Cl_2 and cooled to -78 °C with a dry ice-acetone bath. To the flask was added 10 mL of liquified isobutylene and 2.34 g (16.6 mmol) of 1-dodecanol (**3**). To the vigorously stirred reaction mixture was added 235 μL (2.66 mmol) of trifluoromethanesulfonic acid. After the addition, the resultant mixture was stirred for an additional 3.0 at -5 °C, in an ice-salt bath. Triethylamine, 0.557 g (4 mmol), was then added to the reaction mixture.

The solution was transferred to a 50-mL flask, and the solvents were evaporated. The oily residue was triturated with 100 mL of petroleum ether, the solids were filtered, and the solvent was evaporated.

The crude product obtained in this manner was purified by vacuum distillation, 58-61 °C (1.5 mm), to afford 2.24 g (76%) of pure 1-*tert*-butoxydodecane (**4**) as a colorless liquid. Analysis of the purified product by gas chromatography on a 5% SE 30 column revealed the product to be 99% pure: NMR ($CDCl_3$, Me_4Si) δ 0.7-1.9 (aliphatic envelope), 1.3 (s, 9 H, CH_3), 3.3 (t, J = 7.1, CH_2); IR (cm^{-1}, film) 3080-2730, 1390, 1365; Anal. Calcd for $C_{16}H_{34}O$: C, 79.27; H, 14.13. Found: C, 79.33; H, 14.08.

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Registry No. **1a**, 90-05-1; **1b**, 150-76-5; **1c**, 533-31-3; **1d**, 1745-81-9; **1e**, 591-20-8; **1f**, 121-71-1; **1g**, 97-53-0; **2a**, 16222-38-1; **2b**, 15360-00-6; **2c**, 73673-86-6; **2d**, 99376-82-6; **2e**, 99376-83-7; **2f**, 99376-84-8; **2g**, 99376-85-9; **3**, 112-53-8; **4**, 61548-83-2; $H_2C{=}C(CH_3)_2$, 115-11-7; F_3CSO_3H, 1493-13-6.

A Convenient Synthesis of 2,3-Dihydrothiophene[1]

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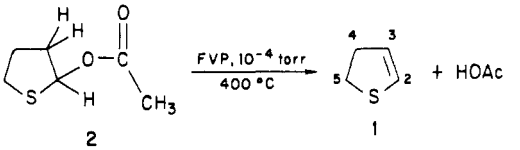
For the purpose of continuing studies of the mechanism of hydrodesulfurization of thiophene,[2] it became necessary to prepare 2,3-dihydrothiophene (**1**). This reactive vinyl thioether, which is proposed as a key intermediate in the hydrodesulfurization of thiophene,[3] is also of interest as a synthetic intermediate. The utility of vinyl thioethers in synthesis has been well-documented.[4] In addition, the chemistry of this reactive olefin is relatively unexplored.[5] Herein we report the preparation of 2,3-dihydrothiophene (**1**) in high yield by flash vacuum pyrolysis (FVP) of 2-(acetoxy)tetrahydrothiophene (**2**) at 400 °C.



2 1

Mixtures of 2,3- and 2,5-dihydrothiophene have been previously obtained by other methods,[6] including the Birch reduction of thiophene.[6a] The separation of these two isomers is complicated by the reactivity of the 2,3-dihydrothiophene which polymerizes upon heating and in the presence of acids.[6a] Small amounts of the 2,3-isomer were isolated from these mixtures by preparative gas chromatography.[7] Only 2,3-dihydrothiophene was obtained from preparative methods described by Sosnovsky.[8] The desired isomer was isolated in 20% yield by heating neat 2-(acetoxy)tetrahydrothiophene (**2**) at 130-150 °C for 1 h and in 60% yield by heating neat 2-(benzoyloxy)-tetrahydrothiophene at 100 °C for 2 h.[8] Extensive dimerization and polymerization of the dihydrothiophene were also reported under these conditions. This synthesis is made somewhat arduous by the purification of the rather unstable benzoyloxy derivative, which cannot be distilled under reduced pressure but must be purified chromatographically.[9]

FVP is a method which is ideally suited for the preparation of reactive molecules like 2,3-dihydrothiophene (**1**). Generally, thermal eliminations of molecules from compounds like **2** proceed readily under FVP conditions with the resulting unsaturated product being directly condensed at liquid nitrogen temperatures.[10]

Results and Discussion

2-(Acetoxy)tetrahydrothiophene (**2**) was pyrolyzed at 400 °C and 10^{-4} torr (higher pressures can be used[11]). Products were collected in a cold trap at -196 °C. A ^1H NMR spectrum of the product mixture showed complete conversion of the starting acetate to 2,3-dihydrothiophene (**1**) and acetic acid. The acetic acid was readily removed by passing the product mixture slowly through a frit covered with solid Na_2CO_3. The 2,3-dihydrothiophene (**1**) may be

(1) Based on the work of N.N.S. in partial fulfillment of the requirements for the Ph.D. Degree at Iowa State University.

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0022-3263/86/1951-0113$01.50/0