H, OH), 7.33 (s, 5 H); IR (neat) 1750, 1770 cm^{-1}; mass spectrum (FD), m/e 379 (M^+), 380 (M + 1), 306 (M - CO_2Et).

When compound **11** was alkylated with diethyl bromomalonate by the general procedure, **16** was isolated as a colorless oil in 40% yield by chromatography, eluting with ethyl acetate/hexane (2:1): ^1H NMR ($CDCl_3$, 90 MHz) δ 1.28 (dt, 6 h), 1.9–2.2 (m, 2 H), 2.73 (dd, 1 H, J_{gem} = 15 Hz, J_{trans} = 3 Hz), 3.15 (dd, 1 H, J_{gem} = 15 Hz, J_{cis} = 6 Hz), 3.73 (t, 2 H), 4.1–4.5 (m, 5 H), 5.8 (br s, 1 OH); IR (neat) 1750, 1770 cm^{-1}.

When compound **19** was alkylated with diethyl bromomalonate by the general procedure, carbinolamine **20** was isolated in 55% yield by chromatography, eluting with ethyl acetate/hexane (4:1). The product was crystallized from ethyl acetate/hexane: mp 102–104 °C; ^1H NMR ($CDCl_3$, 90 MHz), δ 1.3 (dt, 6 H), 2.9 (m, 3 H), 3.5–4.0 (m, 3 H), 4.7 (m, 4 H), 5.6 (br s, 1 H, OH); IR ($CHCl_3$) 3500, 1780 cm^{-1}; mass spectrum (FD) m/e 276 (M + 1), 202 (M - CO_2Et). Anal. Calcd for $C_{11}H_{17}NO_7$: C, 47.99; H, 6.22; N, 5.09. Found: C, 47.62; H, 6.42; N, 5.14.

Condensation Reaction of Diethyl Ketomalonate with *N*-Unsubstituted β-Lactams 12 and 13. Compound **12** or **13** was dissolved in toluene (~0.1 M) and treated with diethyl ketomalonate (1.1 equiv). The solution was refluxed for 2 h with azeotropic distillation. After the solvent was evaporated, the residue was chromatographed on a silica plate. When compound **12** was condensed with diethyl ketomalonate, compound **15** was isolated in 35% yield by chromatography, eluting with ethyl acetate/hexane (2:1). Some of the starting material **12** was also recovered (30–40%).

When compound **13** was condensed with diethyl ketomalonate, carbinolamine **16** was isolated in 35% yield by chromatography.

Compounds 15 and **16**, prepared in this manner, had spectral and TLC properties identical with those of the compounds prepared by the reactions of **10** and **11** with diethyl bromomalonate.

Cyclization of the Adducts 16 and 20 by the Mitsunobu Reaction. The adduct **16** or **20**, as an approximate 0.1 M solution in dry THF, was treated with PPh_3 (1.3 equiv). To this solution was added DEAD or diisopropylazodicarboxylate (DIAD, 1.1 equiv in a small amount of dry THF) dropwise over 10 min with stirring at room temperture. The mixture was allowed to stir for 1 h at room temperature and then concentrated.

When carbinolamine **16** was cyclized by this procedure, 3-oxacepham (**17**) was isolated in 65% yield by chromatography, eluting with ethyl acetate/hexane (3:2). The product was crystallized from ethyl acetate/hexane: mp 53–54 °C; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.30 (dt, 6 H), 1.77–2.05 (7, 2 H), 2.66 (dd, 1 H, J_{gem} = 15.3 Hz, J_{trans} = 1.8 Hz), 3.33 (dd, 1 H, J_{gem} = 15.3 Hz, J_{cis} = 3.9 Hz), 3.62 (m, 1 H), 3.93 (m, 1 H), 4.16 (m, 1 H), 4.26–4.41 (m, 4 H); IR ($CDCl_3$) 1750, 1785 cm^{-1}; mass spectrum (FD), m/e 271 (M^+), 272 (M + 1), 198 (M - CO_2Et). Anal. Calcd for $C_{12}H_{17}NO_6$: C, 53.13; H, 6.32; N, 5.16. Found: C, 52.90; H, 6.49; N, 5.27.

When carbinolamine **20** was cyclized by this procedure isooxapenam (**21**) was isolated in 80% yield by chromatography eluting with ethyl acetate/hexane (3:2). The product was isolated as a colorless oil: ^1H NMR ($CDCl_3$, 90 MHz), δ 1.30 (dt, 6 H), 2.87 (dd, 1 H, J_{gem} = 15 Hz, J_{trans} = 2.5 Hz), 3.43 (dd, 1 H, J_{gem} = 15 Hz, J_{cis} = 5 Hz), 4.0 (m, 1 H), 4.15–4.45 (m, 6 H); IR (neat) 1755, 1790 cm^{-1}; mass spectrum (FD), m/e 258 (M + 1), 184 (M - CO_2Et).

Bromide 18. The alcohol **16** (60 mg, 0.3 mmol) and PPh_3 (87 mg, 0.33 mmol) were dissolved in dry acetonitrile (20 mL) and treated dropwise with CBr_4 (110 mg, 0.33 mmol) in acetonitrile (5 mL) such that the temperature did not rise above ambient. After the mixture was stirred at room temperture for 4 h, the solvent was evaporated, and the residue was chromatographed on a silica plate (1 mm, Chromatotron), eluting with ethyl acetate/hexane (1:1). Bromide **18** was isolated in 75% yield as a colorless oil: ^1H NMR ($CDCl_3$, 90 MHz) δ 1.30 (dt, 6 H), 2.0–2.7 (m, 2 H), 2.70 (dd, 1 H, J_{gem} = 15 Hz, J_{trans} = 3 Hz), 3.20 (dd, 1 H, J_{gem} = 15 Hz, J_{cis} = 6 Hz), 3.47 (dt, 2 H), 4.2–4.6 (m, 5 H), 4.97 (s, 1 H, OH); IR (neat) 1750, 1770 cm^{-1}; mass spectrum (FD), m/e 351, 353 (M^+ 1), 278, 280 (M - CO_2Et).

Cyclization of Bromide 18 to 3-Oxacepham (17). Bromide **18** (40 mg, 0.114 mmol) was dissolved in DMF/CH_2Cl_2 (2:1, 5 mL) and treated with NaH (50% oil, 5.5 mg, 0.114 mmol). The reaction mixture was allowed to stir at room temperature for 5 h and taken up in ethyl acetate (50 mL). The solution was washed several times with H_2O and brine. After the solvent was dried (Na_2SO_4) and evaporated, the residue was chromatographed on a silica plate, eluting with ethyl acetate/hexane (3:2). The desired compound **17** was isolated in 75% yield. This product had identical melting point, spectral, and TLC properties when compared with those of **17** previously prepared from **16**.

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Stereochemistry of the Dehydration of 1,2-Diphenylpropanols via Iodo Intermediates

J. Michael Robinson,* Gregory T. Daniel,[1] and Stephanie J. Hale[1]

Discipline of Chemistry, The University of Texas of the Permian Basin, Odessa, Texas 79762

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The reactivity and stereoselectivity of new dehydration reagents may be easily studied with the use of the secondary benzylic alcohol substrates, *threo*- and *erythro*-1,2-diphenylpropanol (**1** and **2**). These substrates provide the easily differentiated geometric alkene isomers (*Z*)- and (*E*)-1,2-diphenylpropene (**3** and **4**) depending upon the syn or anti mode of elimination (Scheme I). Cram and coworkers[2,3] and, more recently, Reeve and Doherty[4] have studied this system. However, there are unanswered questions important to such stereochemical studies. After a reagent effects dehydration, are the alkene products stable to reaction conditions, thus enabling direct product-mechanism analysis, or do the alkenes *rapidly* react to either return to starting material or to some adduct of different stereochemistry? Are these secondarily produced adducts unstable to reaction conditions (elimination to alkenes adds further complexity to a direct product-mechanism analysis)? Indeed, an acid-catalyzed dehydration of alcohol **1** or **2** illustrates these points, since under the same reaction conditions identical product mixtures enriched in the (*E*)-isomer **4** result. It has been proposed, however, that dehydrations of **1** and **2** induced by iodine or methyltriphenoxyphosphonium iodide (MTPI) both initially form iodo intermediates **5** and/or **6** which undergo an equilibration to predominately **5** (erythro). Subsequent E2 anti elimination affords the least stable (*Z*)-alkene **3** (Scheme II).[4] Iodine,[4] hydrobromic acid,[3a,5] and *p*-toluenesulfonic acid[2,4,6] further isomerize alkenes **3** and/or **4** to mixtures rich in the more thermodynamically stable

(1) Research performed in partial fulfillment of the B.S. degree. Presented in part at the American Chemical Society Permian Basin Section First Annual Student Affiliate Research Symposium, Alpine, TX, November, 1979.

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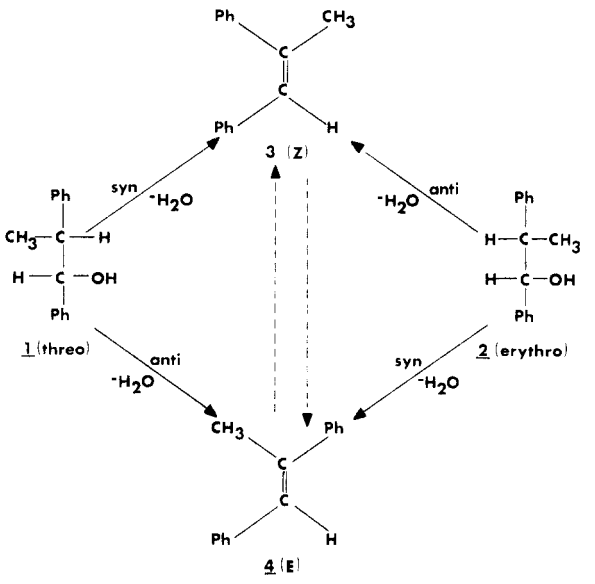
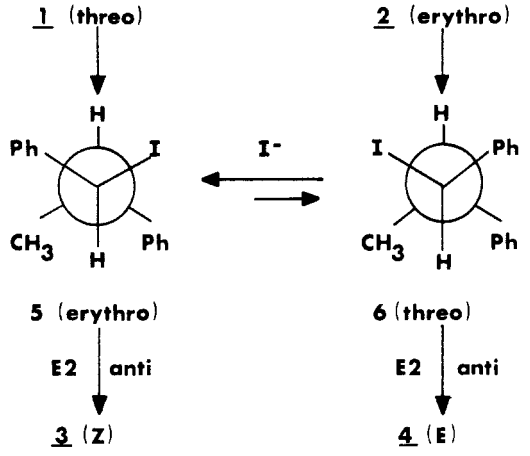
0022-3263/86/1951-0109$01.50/0

Scheme I



Scheme II



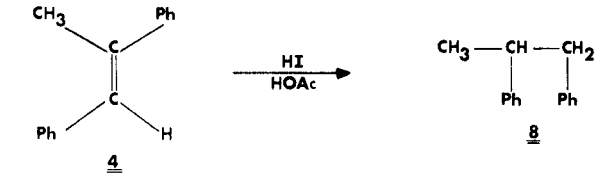
4. Therefore, we investigated the synthesis and reaction of intermediate **5**, *erythro*-1-iodo-1,2-diphenylpropane, as well as the dehydration of **1** and **2** and the isomerization of **3** and **4** with hydriodic acid.

Typically, alcohols **1** and **2** are laboriously prepared by Cram's methods.[3a] The preparation of 1,2-diphenylpropanone (**10**) from benzoin (73%) and reduction to the erythro alcohol **2** was recently made more attractive by the 90% yield of this latter step as achieved via high-pressure hydrogenation.[4] However, we have found the "one-step alternative to a Grignard"[7] expedient in preparing a mixture of alcohols. Whereas attempted Grignard reaction of α-bromoethylbenzene gave only the dimeric 2,3-diphenylbutane (**7**),[5] the corresponding lithium organometallic, conveniently generated *in the cold* from 1-chloroethylbenzene and reacted with benzaldehyde in situ, afforded 59% of a 1:1 mixture of **1** and **2** and only 16% of **7**.

On treatment of the mixture of alcohols **1** and **2** with excess hydriodic acid at room temperature *only* the erythro iodo intermediate **5** was isolated.[8a] Formation of the more thermodynamically stable iodo compound supports Scheme II. That **5** is the erythro isomer[8b] is supported by (1) this mode of formation, (2) large NMR benzylic coupling, J = 10.99 Hz, and expecially (3) E2 anti elimination to the least stable (Z)-alkene **3** (see Experimental Section). Compound **5** reacts with iodide ion from either MTPI (2 mol excess) or sodium iodide (0.2 mol ratio) in warm HMPA to afford essentially a quantitative conversion to olefins **3** and **4** in a 7:1 or 60:1 ratio, respectively. These results, as compared to the 3:1 ratio of alkenes obtained directly from pure threo alcohol **1** on treatment with a threefold excess of MTPI,[4] furnish strong support for the proposed mechanism. The smaller the iodide ion concentration the smaller the amount of inversion of erythro iodide **5** to threo iodide **6** occurs prior to elimination. Control reactions (see Table I) with MTPI indicate no subsequent isomerization of alkene formed under these dehydration conditions by any species present or generated during the dehydration.

Although we found HI not to furnish dehydration of alcohols **1** and **2** in HMPA, the potential isomerizations of **3** and **4** still needed investigation. In warm HMPA solvent the (E)-alkene **4** showed no reaction with HI. Alternatively, the (Z)-alkene **3** may be completely converted to **4** under these conditions.

In acetic acid solvent, catalytic amounts of HI show isomerization of **4** to a dynamic equilibrium mixture of **3** and **4** similar to that obtained with HBr[3a,5] and *p*-TsOH.[4,6] However, as the ratio of HI to **4** in acetic acid is increased from catalytic to stoichiometric amounts (see Table I), and eventually to a several-fold excess, **4** undergoes an entirely different reaction: reduction to 1,2-diphenylpropane (**8**).



This reduction is probably the result of initial Markovnikov addition of HI to the alkene and subsequent HI reduction of this tertiary benzylic iodide. Although evidence of benzylic iodide reductions with HI is found in the literature,[11] this is the first example of reduction of an alkene.

These detailed analyses of acid-catalyzed isomerization (Table I) show the need for a truly catalytic amount of reagent and that careful selection of solvent, temperature, and catalyst may afford a significant shift in the position of equilibrium. Cram's[2] conditions give almost exclusively the more stable isomer **4**. Our synthesis of the least stable (Z)-1,2-diphenylpropene (**3**) from (1-chloroethyl)benzene (**9**) via an alcohol mixture (**1** and **2**) and iodide **5** represents a 45% yield in three steps. Isomerization affords complete conversion to **4**.

Experimental Section

Melting points were measured in capillary tubes with a

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(8) (a) The crude reaction product was shown by NMR to be a 94:6 mixture of **5** and **6**. (b) Incorrectly assigned earlier[3c] as *threo*, therefore, this higher melting point isomer is not the only exception in the relationship of melting points in a series of halogen derivatives. Kingsbury and Best[13] correctly interpreted NMR spectra for the erythro isomer but did not correct the melting point error. Private communications in these matters with Kingsbury revealed their great difficulty in attempts to synthesize the threo isomer by Cram's method, evidence in itself for an unstable isomer. We have found this erythro iodide to be stable as a dry solid at room temperature for more than 1 year in the absence of light. However, iodine color is liberated within 1 h in a $CDCl_3$ solution.

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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.

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

Julie Lynn Holcombe[9] and Tom Livinghouse*

Department of Chemistry, University of Minnesota, Minneapolis, Minnesota 55455

Received August 1, 1985

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

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