palladium on charcoal (50 mg) was stirred in MeOH (5 mL) at room temperature under argon. Following the addition of $NaBH_4$ (400 mg, 10 mmol) in MeOH (5 mL), the mixture was stirred 30 min. The yellow solution was then filtered into H_2O (50 mL) and extracted with Et_2O. The combined Et_2O extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4, and the solvent removed in vacuo to yield 910 mg (97%) of compound 4 as an orange oil: TLC (C_6H_6) R_f 0.20; IR (neat, NaCl) 3450 and 3380 (NH_2), 2930 and 2860 (CH_2), 820 (1,4-substituted Ph) cm^{-1}; MS, m/z (relative intensity) 253 ($M^+[^{35}Cl]$, 6), 106 (100); ^1H NMR ($CDCl_3$) 1.38 (s, 10 H, $(CH_2)_5$), 1,63 (m, 4 H, $PhCH_2CH_2$ and $ClCH_2CH_2$), 2.45 (t, 2 H, J = 6 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 6.8 (AA'BB', 4 H, J = 7 Hz, aromatic). Anal. Calcd for $C_{15}H_{24}NCl$: C, 71.01; H, 9.47; N, 5.5. Found: C, 70.94; H, 9.56; N, 5.70.

1-[4-(9-Chlorononyl)phenyl]-3,3-(1,5-pentanediyl)triazene (5). The amine intermediate 4 (253 mg, 1 mmol) was stirred at 0–5 °C with 0.5 N HCl (4 mL). Sodium nitrite (69 mg, 1 mmol) in H_2O (1 mL) was added dropwise, and the mixture was stirred at 0–5 °C for 5 min. Piperidine (403 mg, 4.5 mmol) in H_2O (3 mL) was then added dropwise while the temperature of the reaction mixture was maintained at 0–5 °C. The solution was stirred at 0–5 °C for 30 min, poured into H_2O (50 mL), and extracted several times with CH_2Cl_2. The combined organic extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4, and the solvent was removed in vacuo. The crude product was dissolved in C_6H_6 (2 mL) and chromatographed on silicic acid (basic, 25 g) slurried in petroleum ether. Elution with 2% ether–petroleum ether gave 5 in fractions 8–13 (143 mg, 41%) as an orange oil. A single component was detected by TLC (2% ether–petroleum ether, R_f 0.20): IR (neat, NaCl) 2910 and 2840 (CH_2), 835 (1,4-substituted Ph) cm^{-1}; MS, m/z (relative intensity) 351 ($M^+[^{35}Cl]$, 100); ^1H NMR (CDCl3) 1.38 (s, 10 H, $(CH_2)_5$), 1.63 (m, 10 H, $PhCH_2CH_2$, $ClCH_2CH_2$ and piperidinyl $CH_2CH_2CH_2$), 2.60 (t, 2 H, J = 6.6 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 3.70 (m, 4 H, piperidinyl CH_2NCH_2), 7.20 (AA'BB', 4 H, J = 8 Hz, aromatic). Anal. Calcd for $C_{20}H_{32}N_3Cl$: C, 68.67; H, 9.16. Found: C, 69.18; H, 9.00.

1-Chloro-9-(*p*-iodophenyl)nonane (6). Method A. Sodium iodide (15 mg, 0.1 mmol) and 2 mL of acetone were stirred at 0–5 °C. Trifluoroacetic acid (114 mg, 1 mmol) was added in 3 mL of acetone. The triazene 5 (35 mg, 0.1 mmol) in 3 mL of acetone was added, and the resulting mixture was stirred at 0–5 °C for 5 min, poured into 50 mL of H_2O, and extracted several times with ether. The combined ether extracts were washed twice with 25 mL of 10% sodium metabisulfite and several times with H_2O and dried over anhydrous Na_2SO_4, and the solvent was removed in vacuo. The crude product was chromatographed on silicic acid (acidic, 25 g). Elution with petroleum ether gave 6 in fractions 7–10 (21 mg, 60%) as a colorless oil.

Method B. To a solution of 1-chloro-9-[*p*-[bis(trifluoroacetyl)thallium]phenyl]nonane (2; 2.04 g, 3 mmol) in water (30 mL) was added potassium iodide (3.00 g, 17 mmol), and the resulting cloudy solution was stirred at room temperature for 15 min. Sodium metabisulfite (1 g) was then added, and the mixture was stirred until the solution turned yellow. After being stirred 30 min, the solution was made basic with 1 N NaOH, filtered, and extracted thoroughly with ether. The combined ether extracts were washed with H_2O, dried over anhydrous Na_2SO_4, and evaporated in vacuo to afford a yellow residue. The residue was taken up in benzene (2 mL) and chromatographed as described in method A to yield 0.64 g (64%) of 1-chloro-9-(*p*-iodophenyl)nonane (6).

The 1-chloro-9-(*p*-iodophenyl)nonane (6) exhibited one component (R_f 0.65) upon TLC analysis (petroleum ether): IR (neat, NaCl) 2930 and 2860 (CH_2) cm^{-1}; MS, m/z (relative intensity) 364 ($M^+[^{35}Cl]$, 40), 217 (100), 91 (tropylium, 85); ^1H NMR ($CDCl_3$) 1.38 (s, 10 H, $(CH_2)_5$), 1.63 (m, 4 H, $PhCH_2CH_2$ and $ClCH_2CH_2$), 2.52 (t, 2 H, J = 5 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 7.20 (AA'BB', 4 H, J = 7 Hz, aromatic protons).

Dimethyl 6,7-Ditelluradodecanedioate (7). Tellurium metal (1.27 g, 10 mmol), sodium hydride (0.44 g, 11 mmol), and dry dimethylformamide (DMF, 50 mL) were stirred at 70 °C under an argon atmosphere for 3 h. The purple Na_2Te_2 solution was cooled to room temperature, and a mixture of methyl 5-bromovalerate (2.15 g, 11 mmol) in 10 mL of argon-purged dry DMF was added. The resulting mixture was stirred at room temperature for 60 min, cooled, poured into water (100 mL), and extracted several times with Et_2O. The combined orange extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4, and the Et_2O was removed in vacuo to give a dark orange oil. The crude ditelluride was chromatographed on silicic acid (basic, 125 g). Elution with $CHCl_3$ gave 1.38 g of 7 as a dark orange oil (53%) which exhibited a single component (R_f 0.56) on TLC (solvent, $CHCl_3$): IR (neat, NaCl) 2920 (CH_2), 1740 (C=O) cm^{-1}; MS, m/z (relative intensity) 490 ($M^+[^{130}Te]$, 12), 360 ($M^+[^{130}Te]$ − Te, 50), 260 (Te_2^+, 10); ^1H NMR ($CDCl_3$) 1.63 (m, 4 H, $(CH_2)_2$), 2.32 (t, 2 H, J = 7 Hz, CH_2COO), 3.06 (t, 4 H, J = 7 Hz, CH_2TeCH_2), 3.65 (s, 3 H, OCH_3).

Methyl 15-(*p*-Iodophenyl)-6-tellurapentadecanoate (9). The ditelluride 7 (800 mg, 1.65 mmol) was stirred at room temperature in MeOH under argon and reduced to a colorless solution of sodium (methylvaleryl)telluride (8) by cautious stepwise addition of $NaBH_4$. The substrate 6 (1.0 g, 3 mmol) was added in MeOH (2 mL). The mixture was refluxed 1 h, cooled, poured into H_2O, and extracted twice with Et_2O, and the combined organic extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4. Following evaporation of the solvent in vacuo, the oily product was chromatographed on silicic acid (basic, 25 g). Elution with C_6H_6 gave 9 in fractions 5–7 (1.60 g, 90%) as a light yellow oil: TLC (C_6H_6) R_f 0.47; IR (neat, NaCl) 1060 (*p*-iodophenyl), 1740 (C=O) cm^{-1}; MS, m/z (relative intensity) 574 (M^+, 14), 543 ($M^+ - OCH_3$, 4), 447 (M^+ − I, 20), 330 (100), 279 (40); ^1H NMR ($CDCl_3$, 200 MHz) 2.27 (t, J = 7 Hz, 2 H, CH_2COO), 2.56 (t, 6 H, J = 7 Hz, CH_2TeCH_2 and CH_2Ph), 3.61 (s, 3 H, $COOCH_3$), 7.19 (AA'BB', 4 H, J = 7 Hz, aromatic).

15-(*p*-Iodophenyl)-6-tellurapentadecanoic Acid (10). The methyl ester 9 (97 mg, 0.168 mmol) was dissolved in EtOH (20 mL) and refluxed with 1 N NaOH (1 mL) under argon for 30 min. The mixture was cooled, poured into H_2O, acidified to pH 2–3 with 10% H_2SO_4, and extracted twice with Et_2O. Following thorough washing with H_2O, the organic layer was dried over anhydrous Na_2SO_4 and the solvent evaporated to give 66 mg (70%) of 15-(*p*-iodophenyl)-6-tellurapentadecanoic acid (10). Crystallization from petroleum ether gave plates: mp 72–73 °C; TLC (8% MeOH–$CHCl_3$) R_f 0.55; ^1H NMR ($CDCl_3$) 2.39 (t, $J \simeq 7$ Hz, 2 H, CH_2COO), 2.62 (t, $J \simeq 7$ Hz, 6 H, CH_2TeCH_2 and CH_2Ph) and 7.23 (AA'BB', 4 H, J = 7 Hz, aromatic); MS, m/z (relative intensity) 560 (M^+, 11), 433 (M^+ − I, 25), 330 (31), 230 (22). Anal. Calcd for $C_{20}H_{31}O_2TeI$: C, 43.03; H, 5.60. Found: C, 43.19; H, 5.69.

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Registry No. 1, 61440-38-8; 2, 81815-22-7; 3, 81815-23-8; 4, 81830-39-9; 5, 81815-24-9; 6, 81815-25-0; 7, 81815-26-1; 8, 81815-27-2; 9, 81815-28-3; 10, 81815-29-4; thallium(III) trifluoroacetate, 23586-53-0; piperidine, 110-89-4; tellurium, 13494-80-9; methyl 5-bromovalerate, 5454-83-1.

Oxidative Decarboxylation of α-Hydroxy Carboxylic Acids with *N*-Iodosuccinimide

T. R. Beebe,* R. L. Adkins, A. I. Belcher, T. Choy,
A. E. Fuller, V. L. Morgan, B. B. Sencherey, L. J. Russell,
and S. W. Yates

Department of Chemistry, Berea College,
Berea, Kentucky 40404

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Recently,[1] we found that 1,2-diols were easily cleaved with *N*-iodosuccinimide (NIS). At this time, we report that

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Table I
Reactions with Irradiations

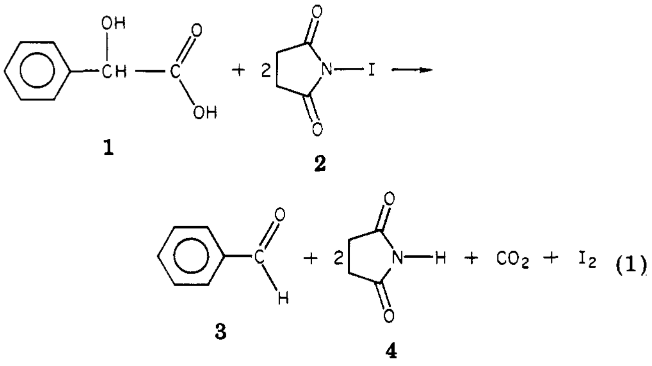
α-hydroxy carboxylic acid	carbonyl products	% yield	time, min	solvents	no. of runs
mandelic acid	benzaldehyde	93-99	10-15	benzene, THF	8
α-hydroxyisobutyric acid	acetone	91-98	5-10	benzene, THF	3
α-ethyl-α-hydroxybutyric acid	3-pentanone	94-96	5-10	chlorobenzene	3
α-hydroxy-α-methylbutyric acid	butanone	82-98	7-10	chlorobenzene	3
α-isopropylmandelic acid	isobutyrophenone	87-103	5-8	benzene	3
benzilic acid	benzophenone	80-97	5-10	benzene, THF	4
phenyllactic acid	phenylacetaldehyde	97-101	5-10	benzene	2

Dark Reactions at Ambient Conditions

α-hydroxy carboxylic acid	carbonyl product followed by % yield and time (min)	solvent
α-ethyl-α-hydroxybutyric acid	3-pentanone: 7 (23), 35 (115), 51 (159), 84 (315)	chlorobenzene
α-hydroxy-α-methylbutyric acid	butanone: trace (5), 34 (60), 71 (180), 88 (360), 99 (420)	chlorobenzene
mandelic acid	benzaldehyde: 85 (5), 93 (15), 96 (30), 98 (60)	benzene

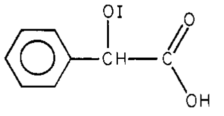
α-hydroxy carboxylic acids also give excellent yields of bond-cleavage products when treated with NIS. Thus, NIS is another reagent to consider in addition to lead tetraacetate[2] and sodium periodate[3] to prepare aldehydes and ketones via decarboxylation of α-hydroxy carboxylic acids.

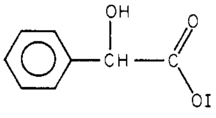
The reaction with NIS can be illustrated by the example of mandelic acid (1). When the acid 1 in benzene was treated with NIS (2), high yields of benzaldehyde (3) (93–99%) and carbon dioxide (99%) could be obtained at ambient temperatures after 5–10 min with irradiation. The stoichiometry shown in eq 1 was supported by good yields of iodine and succinimide (4).



Also, good yields of benzaldehyde (93–98%) were found when mandelic acid was treated with NIS in the dark at ambient temperatures.

Two pathways are possible for the formation of the carbonyl products and carbon dioxide when α-hydroxy carboxylic acids react with *N*-iodosuccinimide. With mandelic acid as an example, either the alkyl hypoiodite 5 or the acyl hypoiodite 6 could be intermediates. There


5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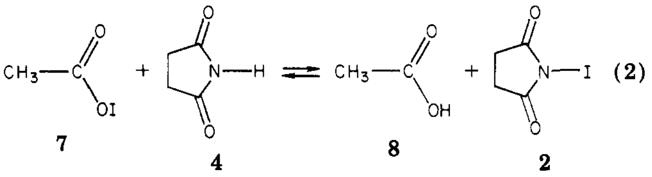
is good evidence[4,5] for the formation of alkyl hypoiodites from the reaction of alcohols and *N*-iodosuccinimide and the subsequent decomposition of the alkyl hypoiodites to produce alkoxy radicals. The chemistry of the reaction of simple carboxylic acids with NIS to form acyl hypoiodites has not been established. Quantitative yields[6] of *N*-iodosuccinimide (2) and acetic acid (8) are produced when acetyl hypoiodite (7) and succinimide (4) are mixed (eq 2), which indicates that little acyl hypoiodite is present



when succinimide is available to be iodinated. Therefore, it is felt that alkyl hypoiodites are more likely to be involved as the main intermediates than are acyl hypoiodites in the reaction of NIS with α-hydroxy carboxylic acids.

A series of seven α-hydroxy carboxylic acids was subjected to *N*-iodosuccinimide degradation with irradiation, and all gave excellent yields in 5–15 min. We found that both *sec*-hydroxy carboxylic acids (mandelic and phenyllactic acid) and *tert*-hydroxy carboxylic acids (benzilic acid, α-hydroxyisobutyric acid, α-ethyl-α-hydroxybutyric acid, α-isopropylmandelic acid, and α-hydroxy-α-methylbutyric acid) gave high yields of products with NIS. Also good yields were obtained with either phenyl groups or alkyl groups attached to the carbon atom holding the hydroxyl group. Oxidations of α-hydroxy carboxylic acids with NIS occurred in the dark at ambient temperatures but at a much slower rate. Irradiation of the reactions increased the speed at which bond cleavage occurred.

Table I outlines the oxidative cleavage of seven structurally different α-hydroxy carboxylic acids with NIS. Reactions were performed in several solvents: benzene, chlorobenzene, and tetrahydrofuran. When carbon dioxide was collected, diphenyl ether, a low-vapor-pressure solvent, was used. Succinimide and iodine were recovered in good yields in all reactions. Carbon dioxide analysis was done with mandelic acid and benzilic acid, yielding 99% and 96%, respectively. Most reactions were run with a slight excess of α-hydroxy carboxylic acid.

Experimental Section

Analyses were performed on a Perkin-Elmer 810 VPC and a Varian Aerograph Model 700 VPC. Liquid chemicals used in reaction mixture and standard VPC mixtures all had greater than

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99.5% purity as determined on a gas chromatograph. The benzene and tetrahydrofuran solvents were spectroscopically pure and were used without further purification. The chlorobenzene solvent was purified by double distillation. VPC analyses were done on 6 ft × 0.25 in. copper columns of 10% SE-30, 7% SE-30 and 3% Carbowax 20M, and 10% Carbowax 20M. The *N*-iodosuccinimide was determined to have 98.5–99.5% active iodine and was used as purchased. Irradiation of reaction mixtures was effected with a GE Projector Spot 150-W, 130-V tungsten lamp.

Oxidation of α-Hydroxy-α-methylbutyric Acid with NIS and Irradiation. A mixture of 0.0726 g (0.615 mmol) of α-hydroxy-α-methylbutyric acid, 0.2448 g (1.088 mmol) of NIS, and 5 mL of chlorobenzene was irradiated and stirred. Reaction times and percentage yields were as follows: 10 min (96%), 30 min (94%), 40 min (96%).

Oxidation of α-Hydroxy-α-methylbutyric Acid with NIS in the Dark at Ambient Temperatures. A mixture of 0.0752 g (0.637 mmol) of α-hydroxy-α-methylbutyric acid, 0.2532 g (1.125 mmol) of NIS, and 5 mL of chlorobenzene was stirred in the dark for 25 h. Reaction times and percentage yields were as follows: 1 h (34%), 3 h (71%), 6 h (88%), 7 h (99%), 24 h (104%), 25 h (103%).

Oxidation of Mandelic Acid with NIS and Irradiation. Determination of I_2 and CO_2 Percentages. Mandelic acid (6.506 g, 0.0428 mol) and 20 mL of diphenyl ether were placed in a 50-mL round-bottomed flask. NIS (6.2308 g, 0.0277 mol) was weighed in another flask. Both flasks were placed on a vacuum rack, and the system was evacuated. The chemicals were mixed, stirred, and irradiated for 20 min. Analyses gave a 99% yield of iodine and a 99% yield of carbon dioxide with a molecular weight of 43.8 g/mol. Mass spectrometer analysis of the carbon dioxide indicated a purity of 99.9%.

Iodine Determination. The iodine produced in the oxidation of α-hydroxy carboxylic acids with NIS was determined by adding reaction mixtures to 25 mL of a 1:1 mixture of acetic acid and water. Several drops of concentrated hydrochloric acid were added, and the iodine was titrated with a standardized solution of thiosulfate. The iodine was found in 85–99% yield, assuming that 1 mol of iodine is produced from 2 mol of NIS.

Succinimide Determination. Succinimide was recovered from the completed reactions by pouring the reaction mixtures into diethyl ether and extracting the ether solution with water. The combined water extracts were washed with fresh ether, and the water solution was evaporated. Succinimide was recovered in 80–98% yields.

Acknowledgment. We are grateful to the Research Corp. for financial support of this research. We also thank the Department of Chemistry of the University of Kentucky for mass spectrometry data.

Registry No. 1, 90-64-2; 2, 516-12-1; α-hydroxy-α-methylbutyric acid, 3739-30-8; α-hydroxyisobutyric acid, 594-61-6; α-ethyl-α-hydroxybutyric acid, 3639-21-2; α-isopropylmandelic acid, 15879-60-4; benzilic acid, 76-93-7; phenyllactic acid, 156-05-8.

Synthesis of Benzhydryl Ethers by a C–C-Forming Reaction Using Benzhydryl 2-Chloroethyl Ether. A Method for Attaching a Protected 2-Hydroxyethyl Group to a Benzylic Carbon

Constantinos G. Screttas* and Maria Micha-Screttas

Institute of Organic Chemistry, The National Hellenic Research Foundation, Athens 501/1, Greece

Received March 16, 1981

The established preparative route to an alcohol like $Ph_3CCH_2CH_2OH$, for example, involves a rather lengthy synthesis[1,2] (eq 1). The obvious organometallic route to triphenylpropanol, i.e., reaction of Ph_3CLi and ethylene oxide, fails to give the expected product. The reason is that the charge-delocalized organolithium reagents may exhibit reactivities toward ethylene oxide comparable to those of lithium alkoxides. Therefore, on addition of ethylene oxide to a reagent like Ph_3CLi, two competing reactions take place, namely, addition of the carbanion to the oxirane linkage and poly- or oligomerization of ethylene oxide by the catalytic action[3] of the alkoxide $Ph_3CCH_2CH_2OLi$. Perhaps, this is the reason that reactions of charge-delocalized organometallics with ethylene oxide have attracted the interest of polymer scientists.[4] One method[5] claims an ca. 50% yield of 3-(2-pyridyl)propanol from 2-picolyllithium and ethylene oxide, which is not impressive.

$$Ph_3COH \xrightarrow{CH_2(CO_2H)_2} Ph_3CCH_2CO_2H \rightarrow Ph_3CCH_2CH_2OH \quad (1)$$

Our need for a number of propanols with aryl substituents in the 3-position prompted us to search for a more convenient method of preparation. We chose to study the coupling between charged-delocalized organolithium reagents with benzhydryl 2-chloroethyl ether, 1. The reasons for choosing 1 are its ready availability[6] and the ease with which benzhydryl ethers can be cleaved or solvolyzed.[7] An additional reason was that benzhydryl ethers themselves constitute an interesting class of organic compounds from both synthetic[8] and mechanistic[9] standpoints.

Charge-delocalized organolithiums like trityllithium, for example, react very readily with 1 and give the expected coupling product (eq 2). The yields, which ranged from fair to excellent, are summarized, along with other relevant data, in Table I. Butyllithium does not couple with 1 in the presence of THF. This probably indicates that the charge-delocalized organolithiums behave as strong nucleophiles,[10] a contrasting feature to the simple alkyllithiums which, most probably, react by a SET mechanism.[11] Despite the simple structure of the benzhydryl ethers in Table I, all were previously unknown. This, perhaps, indicates the synthetic value of the present method. Noteworthy could be the ability of the method to

$$Ph_3CLi + ClCH_2CH_2OCHPh_2 \xrightarrow[90\%]{THF} Ph_3CCH_2CH_2OCHPh_2 \quad (2)$$

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