6 H), 4.87 (m, 6 H), 7.34–7.46 (m, 15 H); IR (KBr) 3320, 3030, 2940, 2860, 2600, 1728, 1655, 1550, 1455, 1410 cm^{-1}; FABMS calcd for $C_{46}H_{63}N_5O_{10}$ 846.04, found 846.5 ((M + H)$^+$).

***N'*-[5-[[4-[[5-[Acetyl(phenylmethoxy)amino]pentyl]amino]-1,4-dioxobutyl](phenylmethoxy)amino]pentyl]-*N*-(4-((phenylthio)carbonyl)butyl)-*N*-(phenylmethoxy)butanediamide (10).** The acid **9** (2.09 g, 2.47 mmol) and thiophenol (0.299 g, 2.72 mmol) were dissolved with stirring in methylene chloride (50 mL). To the ice-cooled solution was added dropwise dicyclohexylcarbodiimide (0.637 g, 3.09 mmol) in 15 mL of methylene chloride followed by (dimethylamino)pyridine (0.059 g, 0.48 mmol) in 10 mL of methylene chloride. After the addition was complete, the ice bath was removed and the reaction mixture stirred overnight. The cloudy solution was filtered, and the white solid washed with additional solvent. The organic solution was washed with an aqueous 0.5 M HCl solution and a saturated $NaHCO_3$ solution, then dried (Na_2SO_4), and evaporated to dryness under reduced pressure. The crude oil **10** (1.89 g) was used directly without further purification (HPLC = ~87%): ^{1}H NMR (CH_3OD) δ 1.27–1.36 (m, 4 H), 1.42–1.56 (m, 4 H), 1.57–1.70 (m, 8 H), 2.05 (s, 3 H), 2.37–2.46 (m, 4 H), 2.65–2.77 (m, 6 H), 3.10–3.17 (t, J = 8, 4 H), 3.60–3.71 (m, 6 H), 4.86–4.93 (m, 6 H), 7.34–7.46 (m, 20 H); IR (CH_2Cl_2) 3680, 3440, 3340, 3040, 2940, 2860, 1700, 1660, 1520, 1450, 1410 cm^{-1}; FABMS calcd for $C_{52}H_{67}N_5O_9S$ 938.21, found 938 ((M + H)$^+$).

***N'*-[5-[[4-[[5-[Acetyl(phenylmethoxy)amino]pentyl]amino]-1,4-dioxobutyl](phenylmethoxy)amino]pentyl]-*N*-(5-hydroxypentyl)-*N*-(phenylmethoxy)butanediamide (11).** Under a blanket of argon sodium borohydride (0.20 g, 5.28 mmol) was added to a solution of the thioester **10** (1.10 g, 1.17 mmol) in absolute ethanol (20 mL) at 0 °C. The reaction mixture was stirred at room temperature for 2 h. The solution was cooled, and then saturated aqueous ammonium chloride (30 mL) was added. The resulting mixture was poured into ice-cold aqueous 1 N HCl (50 mL) and extracted with methylene chloride (3 × 75 mL). The combined organic layers were washed with brine, dried (Na_2SO_4) and concentrated in vacuo. Purification was effected by chromatography on silica eluting with 4% CH_3OH/CH_2Cl_2 to afford 11 (0.83 g, 85%): ^{1}H NMR ($CDCl_3$) δ 1.23–1.37 (m, 6 H), 1.45–1.55 (m, 6 H), 1.58–1.66 (m, 6 H), 2.09 (s, 3 H), 2.45–2.51 (m, 4 H), 2.75–2.84 (m, 4 H), 3.15–3.21 (m, 4 H), 3.57 (t, J = 7, 2 H), 3.58–3.67 (m, 6 H), 4.80 (s, 2 H), 4.85 (d, J = 4, 4 H), 6.38–6.50 (m, 2 H), 7.37 (s, 15 H); IR (CH_2Cl_2) 3680, 3610, 3440, 3340, 3040, 2940, 2860, 1660, 1520, 1450, 1410 cm^{-1}; FABMS calcd for $C_{46}H_{65}N_5O_9$ 832.06, found 832 (M + H)$^+$.

***N'*-[5-[[4-[[5-(Acetylhydroxyamino)pentyl]amino]-1,4-dioxobutyl]hydroxyamino]pentyl]-*N*-(4-carboxybutyl)-*N*-hydroxybutanediamide (2).** The acid **9** (0.52 g, 0.61 mmol) was dissolved in 50 mL of absolute ethanol. To the cloudy solution was added palladium on carbon (0.26 g) under a blanket of argon. The argon atmosphere was replaced with hydrogen, and the mixture stirred vigorously overnight under ambient conditions. Hydrogen was removed at reduced pressure and replaced with argon. The suspension was heated to 80 °C and then filtered over Celite, followed by washing with hot ethanol. Solvent removal followed by recrystallization from water afforded **2** (0.28 g, 80%) as a white solid: mp 140–141 °C ^{1}H NMR (CD_3OD) δ 1.28–1.39 (m, 4 H), 1.47–1.56 (m, 4 H), 1.57–1.69 (m, 8 H), 2.09 (s, 3 H), 2.29–2.35 (t, J = 7, 2 H), 2.42–2.49 (t, J = 7, 4 H), 2.72–2.80 (t, J = 7, 4 H), 3.12–3.20 (t, J = 7, 4 H), 3.55–3.65 (m, 6 H); IR (KBr) 3420, 3300, 3140, 2930, 2860, 1720, 1620, 1565, 1460, 1395 cm^{-1}; FABMS ((M + H)$^+$) = 576. Anal. Calcd for $C_{25}H_{45}N_5O_{10}$: C, 52.16; H, 7.88; N, 12.17. Found: C, 51.92; H, 7.83; N, 12.10.

General Procedure for Preparation of Complexes. *N'*-[5-[[4-[[5-(Acetylhydroxyamino)pentyl]amino]-1,4-dioxobutyl]hydroxyamino]pentyl]-*N*-(4-carboxybutyl)-*N*-hydroxybutanediamide Ferric Complex (2a). To a suspension of the acid **2** (23 mg, 0.04 mmol) in water (5 mL) was added ferric acetylacetonate (13 mg) dissolved in ethyl acetate (5 mL). The two-phase system was stirred vigorously for 2 h at room temperature, and then the layers were separated. The aqueous solution was washed with ethyl acetate and then lyophilized to afford the product **2a** (24 mg, 96%) as a red solid: FABMS calcd for $C_{25}H_{42}FeN_5O_{10}$ 628.49, found 629 ((M + H)$^+$).

***N'*-[5-[[4-[[5-(Acetylhydroxyamino)pentyl]amino]-1,4-dioxobutyl]hydroxyamino]pentyl]-*N*-(5-hydroxypentyl)-*N*-hydroxybutanediamide (3).** To a solution of the alcohol **11** (0.472 g, 0.567 mmol) in absolute ethanol (50 mL) was introduced 10% palladium on carbon (0.23 g). The suspension was stirred under a hydrogen atmosphere overnight. After being heated to 80 °C, the mixture was filtered over Celite. Removal of solvent in vacuo followed by recrystallization from water furnished **3** (0.25 g, 79%) as a white solid (mp 135–136 °C): ^{1}H NMR (CH_3OD) δ 1.28–1.42 (m, 6 H), 1.47–1.58 (m, 6 H), 1.58–1.68 (m, 6 H), 2.09 (s, 3 H), 2.45 (t, J = 7, 4 H), 2.76 (t, J = 7, 4 H), 3.15 (t, J = 7, 4 H), 3.54 (t, J = 7, 2 H), 3.60 (t, J = 7, 6 H); IR (KBr) 3420, 3300, 3140, 2930, 2860, 1620, 1565, 1460, 1395 cm^{-1}; FABMS ((M + H)$^+$) = 562. Anal. Calcd for $C_{25}H_{47}N_5O_9 \cdot H_2O$: C, 51.80; H, 8.52; N, 12.08. Found: C, 51.87; H, 8.36; N, 12.17.

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Registry No. 1 (free base), 70-51-9; 1 (free base aluminum complex), 97633-18-6; 1 (free base iron complex), 14836-73-8; **2**, 123152-37-4; 2 (aluminum complex), 123169-11-9; **2a**, 123169-09-5; 3, 123152-38-5; 3 (aluminum complex), 123169-12-0; 3 (iron complex), 123169-10-8; 4 benzylated dioxolane derivative, 123152-45-4; 4 dioxolane derivative, 123152-46-5; **5**, 84211-46-1; **6**, 112139-65-8; 7, 123152-39-6; 8, 123152-40-9; **9**, 123152-41-0; **9** (X = H), 123152-44-3; **10**, 123152-42-1; 11, 123152-43-2.

Supplementary Material Available: ^{1}H NMR spectra for compounds 2, 3, and 7–11 (7 pages). Ordering information is given on any current masthead page.

Simple Titration Method Using Diphenyl Ditelluride as a Colored Indicator for the Determination of Organolithium and Organomagnesium Reagents[1]

Yoshio Aso, Hironori Yamashita, Tetsuo Otsubo, and
Fumio Ogura*

Department of Applied Chemistry, Faculty of Engineering, Hiroshima University, Saijo, Higashi-Hiroshima 724, Japan

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Rapid and accurate determination of the concentration of organolithium and organomagnesium reagents has become increasingly important with the wide use of these reagents in organic synthesis.[2] Recently developed colorimetric single-titration methods[3-11] are significantly

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[1] Organotelluriums, part 18.

[2] Stowell, J. C. *Carbanions in Organic Synthesis*; Wiley: New York, 1979.

[3] Watson, S. C.; Eastham, J. F. *J. Organomet. Chem.* **1967**, *9*, 165–168.

[4] Eppley, R. L.; Dixon, J. A. *J. Organomet. Chem.* **1967**, *8*, 176–178.

[5] Duhamel, L.; Plaquevent, J. C. *J. Org. Chem.* **1979**, *44*, 3404–3405.

[6] Bergbreiter, D. E.; Pendergrass, E. *J. Org. Chem.* **1981**, *46*, 219–220.

[7] Kofron, W. G.; Baclawski, L. M. *J. Org. Chem.* **1976**, *41*, 1879–1880.

[8] Winkle, M. R.; Lansinger, J. M.; Ronald, R. C. *J. Chem. Soc., Chem. Commun.* **1980**, 87–88.

[9] Lipton, M. F.; Sorensen, C. M.; Sadler, A. C.; Shapiro, R. H. *J. Organomet. Chem.* **1980**, *186*, 155–158.

[10] Juaristi, E.; Martinez-Richa, A.; Garcia-Rivera, A.; Cruz-Sanchez, J. S. *J. Org. Chem.* **1983**, *48*, 2603–2606.

[11] Suffert, J. *J. Org. Chem.* **1989**, *54*, 509–510.

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Table I. Titrations of Organometallic Reagents[a]

organometallic reagent	present method		other method concn, M		
	solvent	concn, M			
n-BuLi (lot 1)	THF	1.41 ± 0.01	1.41 ± 0.01[b]	1.39 ± 0.01[c]	1.42 ± 0.01[d]
n-BuLi (lot 1) + *n*-BuOLi[e]	THF	1.41 ± 0.02			
n-BuLi (lot 2)[f]	THF	0.47 ± 0.02	0.50 ± 0.01[b]	0.47 ± 0.02[c]	0.48 ± 0.01[d]
n-BuLi (lot 3)[f]	THF	0.050 ± 0.005	0.048 ± 0.002[c]	0.045 ± 0.002[d]	
t-BuLi	THF	1.40 ± 0.02	1.40 ± 0.03[b]	1.39 ± 0.02[c]	1.40 ± 0.01[d]
t-BuLi + *t*-BuOLi[e]	THF	1.40 ± 0.04			
MeLi	THF	0.97 ± 0.02	0.96 ± 0.01[b]	0.98 ± 0.02[c]	0.99 ± 0.04[d]
PhLi (lot 1)	THF	1.46 ± 0.01	1.50 ± 0.01[b]	1.49 ± 0.01[c]	1.49 ± 0.01[d]
PhLi (lot 2)	ether	2.17 ± 0.02	2.14 ± 0.02[d]		
	benzene	2.15 ± 0.01			
$(i\text{-Pr})_2NLi$	THF	0.78 ± 0.01	0.78 ± 0.01[b]	0.79 ± 0.01[c]	0.80 ± 0.01[d]
n-BuMgBr	THF	0.75 ± 0.01	0.72 ± 0.02[b]		
MeMgI	THF	0.92 ± 0.01	0.93 ± 0.01[b]		
PhMgBr (lot 1)	THF	0.79 ± 0.02	0.82 ± 0.01[g]		
PhMgBr (lot 2)	ether	0.89 ± 0.01	0.84 ± 0.01[g]	0.88 ± 0.01[h]	
	benzene	0.86 ± 0.01			
$CH_2{=}CHMgBr$	THF	0.99 ± 0.01	0.99 ± 0.01[b]	1.00 ± 0.02[h]	
$n\text{-}C_6H_{13}C{\equiv}CLi$	THF	0.49 ± 0.01			

[a] Each value is an average of three titrations. [b] 2,2′-Bipyridine/*sec*-BuOH method in THF or ether.[3] [c] Diphenylacetic acid method in THF.[7] [d] 1,3-Diphenyl-2-propanone tosylhydrazone method in THF.[9] [e] One equivalent of lithium alkoxide was added to the THF solution containing the reagent 1. [f] The commercial reagent was diluted with hexane. [g] 1,10-Phenanthroline/*sec*-BuOH method in ether.[3] [h] *N*-Phenyl-1-naphthylamine/*sec*-BuOH method in ether.[6]

useful and eliminate the need for a conventional double titration,[12] because these methods are simple and rapid to practice in laboratories and enable the direct analysis of a reactive organometallic reagent without interference from any alkoxide or hydroxide base arising from its unavoidable oxidation or hydrolysis with time. These methods can be classified into three methodological categories: (1) a coordination of organometallic reagents to polycyclic aromatic bases such as 1,10-phenanthroline, 2,2′-biquinoline, and 2,2′-bipyridine to form a colored charge-transfer complex, which is titrated with a standard solution of *sec*-butyl alcohol;[3] (2) single deprotonation of triphenylmethane,[4] *N*-benzylidenebenzylamine,[5] or *N*-phenyl-1-naphthylamine[6] with an organometallic reagent to afford a colored anion, which is titrated with a standard solution of benzoic acid or *sec*-butyl alcohol; (3) double deprotonation of diphenylacetic acid,[7] 2,5-dimethoxybenzyl alcohol,[8] 1,3-diphenyl-2-propanone tosylhydrazone,[9] 4-phenylbenzyl alcohol,[10] and *N*-pivaloyl-*o*-alkylaniline[11] with a strongly basic organometallic reagent to form the corresponding colored dianion after the generation of a colorless monoanion. The last class of colored dianion methods is most convenient for the analysis of strongly basic organolithium reagents, because no standard acid solution is required, and the reagent to be titrated is added via a syringe to a solution of the indicator as in the way usually used in most reactions. However, these methods are inapplicable to the estimation of organomagnesium reagents owing to their weaker basicity. Even in the other classes, only two single-titration methods can be employed for this purpose.[3,6] We present here an alternative general single-titration method classified into a new category, that is, the principle depending on tellurophilic cleavage of diphenyl ditelluride (1) by organometallic reagents and applying to the determination of both organolithiums and organomagnesiums.

Diaryl ditellurides have been known to be deeply colored and react with excess aryllithium and arylmagnesium bromide to afford unsymmetrical diaryl telluride and arenetellurolate.[13] As a part of our interest on organotellurium chemistry, we have examined the utilization of this property for the analysis of organometallic reagents by using diphenyl ditelluride (1) as an indicator (eq 1).

$$\underset{\mathbf{1}}{\mathrm{PhTeTePh}} + \mathrm{RM} \rightarrow \mathrm{RTePh} + \mathrm{PhTeM} \tag{1}$$

$$\mathrm{M = Li\ or\ MgBr}$$

Diphenyl ditelluride (1) imparts an intense red color to various organic solvents and reacts rapidly even at low temperature with organolithiums or organomagnesium halides. A remarkable change to pale yellow is observed at the end point, indicating the complete consumption of diphenyl ditelluride (1). The concentration of the organometallic reagent is determined from the amount consumed. The results obtained by this titration procedure are shown in Table I along with those obtained by the other established single-titration methods. Not only alkyl- and phenyllithium reagents but also less basic organomagnesium halides could be successfully titrated. The values obtained by our procedure were reproducible within ±3% on three titrations and were in fair agreement with those obtained by the other methods. Addition of 1 equiv of *n*-BuOLi or *t*-BuOLi did not change the result, indicating that there is no interaction between 1 and lithium alkoxides. Even at concentrations as low as 0.05 M, satisfactory results were obtained. These data show that the present procedure is both general and reliable. In addition, the organometallic reagents can be conveniently analyzed in most solvents usually used in carbanion chemistry due to the good solubility of 1. No convenient method for the determination of alkynyllithium reagents is hitherto known because of their insufficiency of basicity required for the colored anion methods and of nucleophilicity required for Gilman's double-titration method. On the other hand, the titration of 1-octynyllithium generated from 1-octyne with butyllithium was successfully accomplished by our method and gave a reasonable concentration (0.49 M), that is, good correspondence with that (0.50 M) estimated from the quantity of used butyllithium.

To clarify the discoloration process, products at the end point of the titration of BuLi and BuMgBr were analyzed by vapor pressure chromatography (VPC). When the pale yellow end-point mixture was quenched with water and exposed to air, the organic layer gradually became deep red by reproduction of diphenyl ditelluride (1) from benzenetellurolate. The VPC analysis of the organic layer

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(12) Gilman, H.; Cartledge, F. K. *J. Organomet. Chem.* **1964**, *2*, 447–454.

(13) Petragnani, N. *Chem. Ber.* **1963**, *96*, 247–248.

from the titration of BuLi with 1.04 mmol of 1 indicated a mixture of BuTePh (0.06 mmol), BuTeBu (0.49 mmol), and PhTePh (0.45 mmol) together with 1 (0.54 mmol) derived from benzenetellurolate. The pale yellow end-point color is attributable to these telluride species. The main formation of the symmetrical tellurides is against our prediction based on eq 1. These results indicate the reaction pathways as shown in Scheme I. Highly nucleophilic BuLi reacts rapidly not only with 1 but also with BuTePh formed initially. The latter transmetalation liberates BuTeBu and PhLi, and then PhLi again reacts with 1 to afford PhTePh and PhTeLi. The accumulation of the symmetrical tellurides indicates that the second reaction predominates the first one. On the other hand, the titration of BuMgBr with 0.92 mmol of 1 formed 1 (0.46 mmol) and BuTePh (0.89 mmol) together with trace amounts of BuTeBu (0.03 mmol) and PhTePh (0.03 mmol). The less nucleophilicity of BuMgBr seems to depress transmetalation reaction following eq 1.

Scheme I

$$\mathrm{PhTeTePh + BuLi \rightarrow BuTePh + PhTeLi}$$

$$\mathrm{BuTePh + BuLi \rightarrow BuTeBu + PhLi}$$

$$\mathrm{PhTeTePh + PhLi \rightarrow PhTePh + PhTeLi}$$

$$\mathrm{2PhTeTePh + 2BuLi \rightarrow BuTeBu + PhTePh + 2PhTeLi}$$

The present single-titration method using diphenyl ditelluride (1) as an indicator is simple to practice, and it is easy to observe the end point, like the colored dianion methods. In particular, this procedure is significantly useful in titrations of organomagnesium halides and weakly basic alkynyllithium reagents.

Experimental Section

Tetrahydrofuran (THF) was freshly distilled from sodium benzophenone ketyl. Diethyl ether was distilled over calcium hydride and stored over sodium. Benzene was distilled over calcium hydride and stored in a Schlenck tube under argon. Organolithium and organomagnesium reagents were purchased from Aldrich Chemical Co. or Kanto Chemical Co. or prepared by using unexceptional procedures. Tellurium was purchased as a 300-mesh powder from Nacalai Tesque Inc.

Diphenyl Ditelluride (1). The synthesis of 1 was carried out by modification of the previously reported method[14] as follows. To a suspension of tellurium powder (63.8 g, 0.50 mol) in THF (400 mL) was added a solution of phenylmagnesium bromide, which was prepared from bromobenzene (70.7 g, 0.45 mol) and magnesium (11.2 g, 0.45 mol) in THF (600 mL), with a double-tipped transfer needle. The mixture was stirred under reflux for 4 h and then poured into ice–water. The mixture was acidified with HCl (10% aqueous solution) and extracted with benzene. The organic phase was washed with brine and dried over MgSO$_4$. Evaporation of the solvent and recrystallization from benzene–hexane afforded 1 as orange-red crystals (80.6 g, 88%), mp 65–66 °C (lit.[14] mp 66 °C).

General Procedure for the Titration Using Diphenyl Ditelluride (1) in THF. A 50-mL round-bottom flask containing a magnetic stirring bar was baked out with a heat gun under reduced pressure and cooled to room temperature under argon pressure. The dry flask was charged with accurately weighed diphenyl ditelluride (1, $\sim$1.0 mmol), fitted with a rubber septum cap, and flushed with argon. Anhydrous THF (10 mL) was then added, and stirring was started. After 1 was completely dissolved, the flask was cooled to 0 °C in an ice bath, and the organometallic reagent was added dropwise via a 1.00-mL syringe graduated by 0.01 mL until the red solution faded to pale yellow. The amount consumed contains 1 equiv of organometallic reagent relative to 1.

Diphenyl ditelluride is a low volatile stable compound. Waste solutions from titrations should be, however, handled in a hood, because some alkylphenyl tellurides have a peculiarly bad odor and toxicity data on organotellurium compounds are sparse.

Registry No. 1, 32294-60-3.

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[14] Haller, W. S.; Irgolic, K. J. *J. Organomet. Chem.* 1972, 38, 97–103.

Regioselective 1,2-Reduction of Conjugated Enones and Enals with Sodium Monoacetoxyborohydride: Preparation of Allylic Alcohols

Charles F. Nutaitis* and Joseph E. Bernardo

Department of Chemistry, Lafayette College, Easton, Pennsylvania 18042

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The need to effect a regioselective reduction of an α,β-unsaturated aldehyde or ketone is frequently encountered in organic synthesis. Reduction of these systems with sodium borohydride, one of the most widely utilized reducing agents, is highly solvent dependent and generally does not result in useful regioselectivity (Table I).

It has been previously demonstrated that treatment of borohydride with a controlled amount of acetic acid affords a reducing agent which is weaker and thereby more selective than the traditional sodium borohydride/ethanol reduction system. For example, Nutaitis and Gribble[2] have shown that tetra-*n*-butylammonium triacetoxyborohydride effectively reduces aldehydes in the presence of ketones, even with excess hydride present in the reaction medium. This selectivity is not possible with unmodified borohydride. Thus, we felt that utilization of an (acyloxy)borohydride species to reduce α,β-unsaturated carbonyl compounds would result in enhanced regioselectivity.

Our initial studies utilized 2-cyclohexen-1-one and 3-methyl-2-cyclohexen-1-one. These substrates were subjected to reduction with sodium mono-, di-, and triacetoxyborohydride, which were generated by addition of the requisite amount of glacial acetic acid to a suspension of sodium borohydride in dry THF. A fourth reaction, which consisted of addition of sodium borohydride pellets to a solution of the substrate in neat acetic acid, was also performed. The results of these experiments are shown in Table II.

As can be seen, the greatest regioselectivity was realized with either sodium monoacetoxyborohydride or sodium triacetoxyborohydride. Lower selectivity was exhibited by sodium diacetoxyborohydride as well as by the sodium borohydride/acetic acid system. The latter result is not unreasonable; as the pellet slowly dissolves, there will be a finite concentration of unmodified (more highly reactive) borohydride coexisting with the substrate in the reaction system for a period of time. As discussed above, unmodified borohydride does not exhibit high regioselectivity with regard to conjugated enone and enal reduction. The less efficient regioselectivity of sodium diacetoxyborohydride, however, was not expected. The reactivity of (acyloxy)borohydride species is generally believed to decrease as more electron withdrawing ligands become bonded to the central boron atom.[3] Thus, one would expect the re-

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[1] (a) Johnson, M. R.; Rickborn, B. *J. Org. Chem.* 1970, 35, 1041. (b) Experiment performed in our laboratory. (c) Jackson, W. R.; Zurqiyah, A. *J. Chem. Soc.* 1965, 5280. (d) Iqbal, K.; Jackson, W. R. *J. Chem. Soc. C* 1968, 616.

[2] Nutaitis, C. F.; Gribble, G. W. *Tetrahedron Lett.* 1983, 24, 4287.

[3] Gribble, G. W.; Nutaitis, C. F. *Org. Prep. Proc. Int.* 1985, 17, 317.

0022-3263/89/1954-5629$01.50/0