Cycloaddition of *N-tert*-Butyl-*C*-phenylnitrone with Methyl 2-Methyl-2,3-butadienoate. A solution containing 1.00 g of *N-tert*-butyl-*C*-phenylnitrone and 0.67 g of methyl 2-methyl-2,3-butadienoate (**45**) in 15 mL of benzene was heated in a sealed tube at 80 °C for 8 h. Concentration of the solution under reduced pressure followed by silica gel chromatography using a 2% ethyl acetate–hexane mixture as the eluent gave 70 mg (20%) of *N-tert*-butyl-5-methyl-4-methylene-3-phenyl-5-carbomethoxyisoxazolidine (**48**) as a clear oil: IR (neat) 2995, 1745, 1455, 1370, 1285, 1225, 1130, 990, 910, 865, 760, and 705 cm^{-1}; NMR ($CDCl_3$, 90 MHz) δ 1.05 (s, 9 H), 1.70 (s, 3 H), 3.80 (s, 3 H), 4.40 (d, 1 H, J = 2.0 Hz), 4.55 (t, 1 H, J = 2.0 Hz), 5.10 (d, 1 H, J = 2.0 Hz), and 7.18–7.38 (m, 5 H); ^{13}C NMR ($CDCl_3$, 200 MHz) δ 24.72, 26.26, 52.55, 58.69, 67.78, 82.04, 108.29, 127.43, 128.33, 128.53, 141.84, 159.99, and 172.41; MS, m/e 289 (M^+), 274, 233, 174, 132, 115, and 84; HRMS calcd for $C_{17}H_{23}NO_3$ 289.1679, found 289.1678.

Acknowledgment. We gratefully acknowledge the National Cancer Institute for generous support of this research. The high-field NMR spectrometer used in these studies was made possible through a NSF equipment grant.

Registry No. 10, 3376-24-7; **11**, 108817-65-8; **12**, 108817-66-9; **13**, 108817-67-0; **14**, 41012-82-2; **15**, 43044-81-1; **16**, 43044-82-2; **17**, 1137-96-8; **18**, 3376-23-6; **19**, 108817-68-1; **20**, 108817-69-2; **21**, 108817-70-5; **24**, 108817-71-6; **25**, 108817-72-7; **26**, 108817-73-8; **27**, 108817-74-9; **28**, 108817-75-0; **29**, 108817-76-1; **31**, 108817-77-2; **32**, 107402-99-3; **33**, 108817-78-3; **34**, 108817-79-4; **35**, 108817-80-7; **36**, 108817-81-8; **37**, 108817-82-9; **38**, 108817-83-0; **39**, 108817-84-1; **40**, 3317-61-1; **41**, 108817-85-2; **42**, 4567-18-4; **44a**, 108817-87-4; **44b**, 108817-91-0; **45**, 18913-37-6; **46a**, 108817-88-5; **46b**, 108817-93-2; **48**, 108817-89-6; $CH_2{=}C{=}CHC(O)OMe$, 108817-89-6; $PhSO_2CH{=}CH_2$, 5535-48-8; fumaronitrile, 764-42-1; α-acetoxyacrylonitrile, 3061-65-2; cyanoallene, 1001-56-5; dimethyl 2,3-pentadienedioate, 1712-36-3; (phenylsulfonyl)propadiene, 2525-42-0; *N*-methyl-*C*-dimethylnitrone, 72552-73-9; 3a,4,5,6-tetrahydro-2,6,6-trimethylpyrrolo[1,2-*b*]isoxazole-3-carbonitrile, 108817-90-9; 3a,4,5,6-tetrahydro-2,3a,6,6-tetramethylpyrrolo[1,2-*b*]isoxazole-3-carbonitrile, 108817-92-1.

Notes

Direct Synthesis of Ethers via Zinc Chloride Mediated Etherification of Alcohols in Dichloroethane

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Received January 23, 1987

Although the Williamson ether synthesis is one of the most widely used methods for synthesis of ethers, it requires initial conversion of alcohols into halides or tosylates and their displacement with alkoxides.[1] Several synthetic methods for the direct synthesis of ethers from alcohols are available, but each method has certain limitations with regard to scope and reaction conditions.[2]

Recently, Lau reported reductive deoxygenation of aromatic aldehydes and ketones with zinc iodide/sodium cyanoborohydride and briefly mentioned the preparation of bis(diphenylmethyl) ether from benzhydrol using zinc iodide in dichloroethane without describing precise reaction conditions and yield.[3,4] This information prompts us to report our results on zinc chloride mediated etherification of alcohols. It has been reported that benzylic, allylic, and tertiary alcohols can be activated with zinc halides to generate carbocationic species, which are trapped with thiols,[5] thio acids,[6] selenols,[7] and hydrides.[3] Similar behavior has been noted with benzylic, allylic, and tertiary halides when using zinc halides.[8]

We have found that the success of direct synthesis of ethers from alcohols is dependent critcally on solvents and Lewis acids. First, solvent effects were examined with α-methylbenzyl alcohol as a model compound with 1 equiv of zinc chloride. In dichloroethane, bis(α–methylbenzyl) ether was obtained in 91% yield at room temperature in 4 h, whereas no reaction occurred in other solvents such as tetrahydrofuran, ethyl ether, and acetonitrile. Among several Lewis acids tested in dichloroethane, zinc chloride gave the best results and is generally recommended. The reaction of α–methylbenzyl alcohol with 1 equiv of zinc iodide at room temperature for 24 h resulted in a less than 20% yield, and the reaction required 6 h in refluxing dichloroethane for completion.[9,10] This result is in contrast with previous reports in which zinc iodide is more effective than zinc chloride.[5,6] Furthermore, the use of boron trifluoride etherate[11] gave a mixture of several products along with the polymerized products, whereas the use of titanium

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[1] Barton, D., Ollis, W. D., Ed. *Comprehensive Organic Chemistry*; Pergamon: Elmsford, NY, 1979; Vol. 1, p 819 and references cited therein.

[2] (a) Curtin, D. Y.; Lescowitz, S. *J. Am. Chem. Soc.* **1951**, *73*, 2630. (b) Traynellis, V. J.; Hergenrother, W. L.; Hanson, H. T.; Valicenti, J. A. *J. Org. Chem.* **1964**, *29*, 123. (c) Emert, J.; Goldenberg, M.; Chiu, G. L.; Valeri, A. *J. Org. Chem.* **1977**, *42*, 2012. (d) Noda, I.; Horita, K.; Oikawa, Y.; Yonemitsu, O. *Tetrahedron Lett.* **1986**, *27*, 1917.

[3] Lau, C. K.; Dufresne, C.; Belanger, P. C.; Pietre, S.; Scheigetz, J. *J. Org. Chem.* **1986**, *51*, 3038.

[4] In contrast with reported deoxygenation of benzylic alcohols by zinc iodide/sodium cyanoborohydride, the reaction of α-methylbenzyl alcohol with 1 equiv of zinc chloride and 2 equiv of sodium cyanoborohydride in dichloroethane at room temperature for 4 h gave bis(α-methylbenzyl) ether in 80% yield without the formation of ethylbenzene.

[5] Guindon, Y.; Grenette, R.; Fortin, R.; Rokach, J. *J. Org. Chem.* **1983**, *48*, 1357.

[6] Gauthier, J. Y.; Bourdon, F.; Young, R. N. *Tetrahedron Lett.* **1986**, *27*, 15.

[7] Clarembeau, M.; Krief, A. *Tetrahedron Lett.* **1984**, *25*, 3625.

[8] (a) Miller, J. A. *Tetrahedron Lett.* **1975**, 2959. (b) Gurudutt, K. N.; Ravindranath, B.; Srinivas, P. *Tetrahedron* **1982**, *38*, 1843. (c) Kim, S.; Kim, Y. J.; Ahn, K. H. *Tetrahedron Lett.* **1983**, *24*, 3369. (d) Kim, S.; Hong, C. Y.; Yang, S. *Angew. Chem.* **1983**, *95*, 568.

[9] In order to explain the relatively low reactivity of zinc iodide, one of the reviewers suggested that trace amounts of water due to the hygroscopic nature of zinc chloride might participate for the reaction to occur rapidly. However, the use of commercially available zinc chloride without drying and wet zinc iodide [after anhydrous zinc iodide (2 mmol) was stirred in dichloroethane (10 mL) containing water (10 μl) at room temperature for 30 min, α-methylbenzyl alcohol was added] did not significantly change the reaction rates, as compared with the use of anhydrous zinc chloride and anhydrous zinc iodide, respectively.

[10] The use of zinc bromide required 8 h at room temperature for completion.

[11] (a) Mandal, A. K.; Mahajan, S. W. *Tetrahedron Lett.* **1985**, *26*, 3863. (b) Eugene, K. L.; Zarate, E. A.; Youngs, W. J.; Salomon, R. G. *J. Am. Chem. Soc.* **1986**, *108*, 1311.

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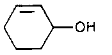
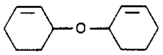
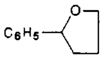
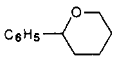
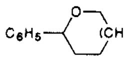
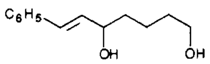
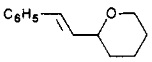
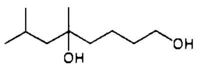
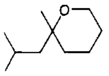
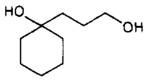
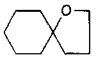
Table I. Preparation of Acyclic and Cyclic Ethers from Alcohols[a]

alcohol	temp, °C	time, h	ether	yield,[b] %	^{1}H NMR spectral data and physical constants (mp, °C; bp, °C/mmHg)[c]
$C_6H_5CH(CH_3)OH$	rt	4	$C_6H_5(CH_3)CHOCH(CH_3)C_6H_5$	91	1.39 (d, *J* = 6.5, 3 H), 1.43 (d, *J* = 6.5, 3 H), 4.27 (q, *J* = 6.5, 1 H), 4.51 (q, *J* = 6.5, 1 H), 7.27 (s, 5 H), 7.32 (s, 5 H); bp 146–152/10 (lit.[2c] bp 144–150/10)
$(C_6H_5)_2CHOH$	rt	2.5	$(C_6H_5)_2CHOCH(C_6H_5)_2$	95	5.45 (s, 2 H), 7.18–7.43 (m, 20 H); mp 89–91 (lit.[d] mp 90–105)
$C_6H_5CH_2OH$	80	72	$C_6H_5CH_2OCH_2C_6H_5$	63 (29)[e]	4.58 (s, 4 H), 7.36 (s, 10 H); bp 162–166/15 (lit.[f] bp 164–166/15)
p-$MeOC_6H_4CH_2OH$[i]	rt	24	*p*-$MeOC_6H_4CH_2OCH_2C_6H_4OMe$-*p*	54	3.80 (s, 6 H), 4.46 (s, 4 H), 6.85 (d, *J* = 8, 4 H), 7.29 (d, *J* = 8, 4 H); mp 37–38 (lit.[2c] mp 38–38.7)
	rt	3		66	1.30–2.20 (m, 12 H), 3.80–4.09 (m, 2 H), 5.81 (br s, 4 H); bp 72–76/1.2
$C_6H_5CH(CH_3)OH + CH_3CH_2OH$[g]	80	2	$C_6H_5CH(CH_3)OCH_2CH_3$	90	1.18 (t, *J* = 7, 3 H), 1.41 (d, *J* = 6, 3 H), 3.35 (q, *J* = 7, 2 H), 4.40 (q, *J* = 6, 1 H), 7.29 (br s, 5 H); bp 86–92/15 (lit.[f] 88–90/15)
$C_6H_5CH(CH_3)OH + C_6H_5CH_2OH$	rt	12	$C_6H_5CH(CH_3)OCH_2C_6H_5$	69	1.43 (d, *J* = 6, 3 H), 4.10–4.60 (m, 3 H), 7.23 (s, 5 H), 7.30 (s, 5 H); bp 88–92/0.5 (lit.[f] bp 87–89/0.5)
$C_6H_5CH(CH_3)OH + CH_2{=}CHCH_2OH$	rt	16	$C_6H_5CH(CH_3)OCH_2CH{=}CH_2$	70[h]	1.44 (d, *J* = 6, 3 H), 3.89 (d, *J* = 5, 2 H), 4.41 (q, *J* = 6, 1 H), 4.96–5.40 (m, 2 H), 5.59–6.20 (m, 1 H), 7.27 (s, 5 H); bp 86–92/10
$C_6H_5CH(OH)(CH_2)_3OH$	80	1		64	1.53–2.50 (m, 4 H), 3.68–4.29 (m, 2 H), 4.83 (t, *J* = 6, 1 H), 7.30 (s, 5 H); bp 90–96/20
$C_6H_5CH(OH)(CH_2)_4OH$	80	3		84	1.20–2.05 (m, 6 H), 3.35–3.75 (m, 2 H), 3.98–4.43 (m, 1 H), 7.33 (s, 5 H); bp 115–120/13 (lit.[j] bp 115/131)
$C_6H_5CH(OH)(CH_2)_{10}OH$	80	2		46	1.18—1.68 (m, 16 H), 1.96–2.37 (m, 2 H), 3.42 (t, *J* = 6, 2 H), 6.28–6.35 (m, 1 H), 7.20–7.39 (m, 5 H); mp 36–38
	rt	12		71	1.40–2.11 (m, 6 H), 3.40–4.31 (m, 3 H), 6.08–6.98 (m, 2 H), 7.25–7.60 (m, 5 H); bp 70–76/3.5
	80	1		90	1.05 (d, *J* = 6.5, 6 H), 1.10–2.15 (m, 12 H), 3.60–3.81 (m, 2 H); bp 86–93/20
	80	1		89	1.12–2.15 (m, 14 H), 3.91 (t, *J* = 6, 2 H); bp 82–88/20

[a] The reaction was normally carried out with equimolar amounts of an alcohol and zinc chloride in dichloroethane. [b] Isolated yields. [c] Satisfactory mass spectral data were obtained. [d] Wang, C.-H. *J. Org. Chem.* **1963**, *28*, 2914. [e] The yield of recovered benzyl alcohol. [f] McKillop, A.; Ford, M. E. *Tetrahedron* **1974**, *30*, 2467. [g] Ethyl alcohol (2 equiv) was used. [h] Bis(α-methylbenzyl) ether was also obtained in 20% yield. [i] Zinc chloride (0.1 equiv) was used. [j] Crisan, C. *Chem. Abstr.* **1957**, *51*, 5061f.

tetrachloride gave α-methylbenzyl chloride as a major product. Aluminum trichloride and boron tribromide were ineffective. Thus, remaining reactions were generally carried out with equimolar amounts of an alcohol and zinc chloride in dichloroethane.

Table I summarizes some experimental results and illustrates the efficiency, applicability, and scope of the present method. Benzhydrol was cleanly converted into bis(diphenylmethyl) ether in 95% yield at room temperature within 2.5 h, whereas the use of zinc iodide required 24 h at room temperature for completion of the reaction. Etherification of benzyl alcohol proceeded very slowly, yielding 63% of dibenzyl ether along with 29% of recovered benzyl alcohol after refluxing in dichloroethane for 72 h. In the case of *p*-methoxybenzyl alcohol, polymerized products were obtained, as previously reported by Emert.[2c] However, bis(*p*-methoxybenzyl) ether could be prepared in 54% yield along with a small amount of polymerized products by use of 0.1 equiv of zinc chloride. An allylic alcohol, 2-cyclohexen-1-ol, worked well. Tertiary alcohols such as 3-ethylheptan-3-ol and 2-phenylhexan-2-ol failed to give the corresponding ethers, and olefinic products were obtained. Preparation of unsymmetrical ethers has been examined with α-methylbenzyl alcohol as a model compound. Reaction of α-methylbenzyl alcohol with ethyl alcohol and benzyl alcohol under similar conditions afforded the corresponding unsymmetrical ethers in high yields without the formation of bis(α-methylbenzyl) ether. However, the use of allyl alcohol gave 1-propenyl α-methylbenzyl ether in 70% yield along with 20% of bis(α-methylbenzyl) ether. Furthermore, the present method can be applied to prepare cyclic ethers under mild conditions. As shown in Table I, when diols containing benzylic, allylic, and tertiary alkyl alcohols were treated with zinc chloride in dichloroethane, tetrahydrofuran, and tetrahydropyran derivatives were obtained in high yields, although 12-membered cyclic ether was obtained in a moderate yield. It is noteworthy that diols containing tertiary alkyl alcohols were smoothly cyclized to the cyclic ethers in high yields without the formation of olefinic products observed in etherification of tertiary alkyl alcohols.

Experimental Section[12]

Typical Procedure for Preparation of Acyclic Ethers. To a stirred solution of α-methylbenzyl alcohol (245 mg, 2.0 mmol) in dichloroethane (10 mL) at room temperature was added anhydrous zinc chloride (270 mg, 2.0 mmol). The reaction mixture was stirred at room temperature for 4 h, diluted with dichloromethane (30 mL), and washed with water and brine. The organic layer was dried over anhydrous $\mathrm{MgSO_4}$ and evaporated to dryness. The residue was distilled with a Kugelrohr apparatus to afford bis(α-methylbenzyl) ether (206 mg, 91%) as a colorless oil. The spectral data and boiling point were identical with those of reported data.

Typical Procedure for Preparation of Cyclic Ethers. To a stirred solution of 1-phenyl-1,5-pentanediol (182 mg, 1.0 mmol) in dichloroethane (20 mL) at room temperature was added zinc chloride (135 mg, 1.0 mmol), and the reaction mixture was stirred at 80 °C for 2.5 h. The usual workup and distillation with a Kugelrohr apparatus gave 2-phenyltetrahydropyran (136 mg, 84%). The spectral data[13] and boiling point were in agreement with reported data.

Acknowledgment. This investigation was supported in part by a grant from the Korea Science and Engineering Foundation. We are grateful to Sung Soo Kim for the experimental assistance.

Registry No. $\mathrm{PhCH(Me)OH}$, 98-85-1; $\mathrm{Ph_2CHOH}$, 91-01-0; $\mathrm{PhCH_2OH}$, 100-51-6; *p*-$\mathrm{MeOC_6H_4CH_2OH}$, 105-13-5; EtOH, 64-17-5; $\mathrm{CH_2{=}CHCH_2OH}$, 107-18-6; $\mathrm{PhCH(OH)(CH_2)_3OH}$, 4850-50-4; $\mathrm{PhCH(OH)(CH_2)_4OH}$, 1011-61-6; $\mathrm{PhCH(OH)(CH_2)_{10}OH}$, 109217-58-5; $\mathrm{PhCH{=}CHCH(OH)(CH_2)_4OH}$, 17303-68-3; *i*-$\mathrm{BuC(OH)(Me)(CH_2)_4OH}$, 109217-59-6; $\mathrm{PhCH(Me)OCH(Me)Ph}$, 93-96-9; $\mathrm{Ph_2CHOCHPh_2}$, 574-42-5; $\mathrm{PhCH_2OCH_2Ph}$, 103-50-4; *p*-$\mathrm{MeOC_6H_4CH_2OCH_2C_6H_4}$-*p*-OMe, 5405-95-8; $\mathrm{PhCH(Me)OEt}$, 3299-05-6; $\mathrm{PhCH(Me)OCh_2Ph}$, 2040-37-1; $\mathrm{PhCH(Me)OCH_2CH{=}CH_2}$, 27122-63-0; $\overline{\mathrm{CH(Ph)(CH_2)_3O}}$, 16133-83-8; $\overline{\mathrm{CH(Ph)(CH_2)_4O}}$, 4203-44-5; $\overline{\mathrm{CH(Ph)(CH_2)_{10}O}}$, 109217-60-9; $\mathrm{PhCH{=}CH\overline{CH(CH_2)_4O}}$, 70028-17-0; *i*-$\mathrm{Bu\overline{C(Me)(CH_2)_4O}}$, 109217-61-0; 2-cyclohexen-1-ol, 822-67-3; 1-hydroxy-1-cyclohexanepropanol, 6963-45-7; di(2-cyclohexenyl) ether, 15129-33-6; 1-oxaspiro[4.5]decane, 176-91-0.

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[12] ^{1}H NMR spectra were recorded in $\mathrm{CDCl_3}$ with a Varian T-60A or FT-80A spectrometer, and chemical shifts are expressed as δ units relative to tetramethylsilane. Coupling constants are given in hertz. Infrared spectra were measured as a neat film or KBr pellets on a Perkim-Elmer 267 spectrometer. Mass spectra were obtained on a Hewlett-Packard 5895A spectrometer. Melting points were taken on an Electrothermal apparatus, and reported boiling points are those observed during distillation with a Kugelrohr apparatus.

[13] Fujisaka, T.; Nojima, M.; Kusabayashi, S. *J. Org. Chem.* **1985**, *50*, 275.

Synthesis of α-Iodo Ketones

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Received January 12, 1987

A number of methods for the direct and indirect preparation of α-iodo ketone from the ketones, enol acetates, and enol silyl ethers have been reported.[1] Although methods such as lead tetraacetate/metal iodide,[2] pyridinium chlorochromate/iodine,[3] silver acetate/iodine,[4] thallium(I) acetate/iodine,[5] and electrolysis[6] represent significant advances, new and efficient methods for the preparation of α-iodo ketones are still desirable. Recently, during our study of natural products synthesis, we discovered a convenient new procedure for the preparation of α-iodo ketones from the ketones or enol silyl ethers under mild reaction conditions.

Results and Discussion

Treatment of cyclohexanone (**1**) with trimethylsilyl iodide/hexamethyldisilazane[7] followed by *m*–chloroperbenzoic acid (MCPBA) oxidation afforded 2-iodo-1-cyclohexanone (**5**) cleanly. Apparently, trimethylsilyl iodide/hexamethyldisilazane reacted with cyclohexanone to give the enol silyl ether (**2**) and hexamethyldisilazane hydroiodide salt (**3**). Treatment of the reaction mixture with MCPBA oxidized the iodide ion to an "$\mathrm{I^+}$" species which reacted with the enol silyl ether to give the α-iodo ketone upon aqueous workup, presumably via intermediate **4** as shown in Scheme I. Addition of sodium iodide to the reaction mixture before the treatment with MCPBA increased the yield of α-iodo ketone by 10–20% (Table I).

Alternatively, performed enol silyl ethers could be treated sequentially with sodium iodide and then MCPBA to give α-iodo ketones in slightly lower yields (see Table II). In contrast, it has been reported that direct treatment of ketone enolate or enol silyl ether with molecular iodine does not give useful yield of iodo ketones.[4] Additionally, we also found that enamine **19** (Table II) could also be treated with NaI/MCPBA followed by hydrolysis to give α-iodo ketone **5**. With the enol silyl ethers of the aromatic ketones **17** and **18**, only the α-iodo ketones **21**[9] and **22** were obtained. Ring iodination reaction did not occur by this procedure.

In summary, we have discovered a convenient procedure for the preparation of α-iodo ketones. This method is complementary to the existing procedures and sometimes could be the method of choice because of its simplicity and high yield.

Experimental Section

General experimental conditions and instruments used in analyses were identical with those described in our earlier work.[8] *m*-Chloroperbenzoic acid and hexamethyldisilazane were purchased from Aldrich Co. All the known iodo ketones were identified by comparison of their IR, ^{1}H NMR, and MS spectra with the published data.

General Procedures for the Preparation of α-Iodo Ketones from Ketones. To a solution of ketone (1 mmol) in dry dichloromethane (5 mL) was added hexamethyldisilazane (1.2 mmol), and then trimethylsilyl iodide (1.1 mmol) was added dropwise at 0 °C. A solution of sodium iodide (1.0 mmol) in THF (3 mL) was then added. To this reaction mixture was added dropwise a solution of MCPBA (1.2 mmol) in dry $\mathrm{CH_2Cl_2}$ (3 mL) at 0 °C, and the reaction was stirred for 10 min. The reaction solution was diluted with ether (50 mL), washed with 10% HCl (5 mL), saturated sodium thiosulfate solution (10 mL), and

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[1] (a) Ringold, H. J.; Stork, G. *J. Am. Chem. Soc.* **1958**, *81*, 250. (b) Julian, P. L.; Karpel, W. J. *J. Am. Chem. Soc.* **1950**, *72*, 362. (c) Djerassi, C.; Lenk, C. T. *J. Am. Chem. Soc.* **1953**, *75*, 3493. (d) Djerassi, C.; Lenk, C. T. *J. Am. Chem. Soc.* **1954**, *76*, 1722. (e) Djerassi, C.; Grossman, J.; Thomas, G. H. *J. Am. Chem. Soc.* **1955**, *77*, 3826. (f) Halpern, O.; Djerassi, C. *J. Am. Chem. Soc.* **1959**, *81*, 439. (g) Rothman, E. S.; Perlstein, T.; Wall, M. E. *J. Org. Chem.* **1960**, *75*, 1966.

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