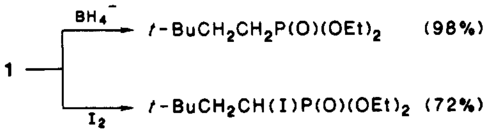
$$t\text{-BuHgCl (4 equiv)} + \text{VP} \xrightarrow{h\nu} \underset{\mathbf{1}}{t\text{-BuCH}_2\text{CH(HgCl)P(O)(OEt)}_2} \quad (4)$$

reaction 4, the intermediate $t\text{-BuCH}_2\dot{\text{C}}\text{HP(O)(OEt)}_2$ is an acceptor radical which readily enters into reaction 3 with t-BuHgCl with $k > 10^5\ \text{M}^{-1}\ \text{s}^{-1}$.[8] The resulting **1** can be reduced by BH_4^- or cleaved by I_2 in high overall yields



(based on starting alkene). For $CH_2{=}CHSO_2Ph$ in Me_2SO, a kcl of 108 ($[CH_2{=}CHSO_2Ph]_0 = 0.19$ M) was measured at 35–40 °C, and yields of $t\text{-BuCH}_2\text{CH}_2\text{SO}_2\text{Ph}$ and $t\text{-BuCH}_2\text{CH(I)SO}_2\text{Ph}$ of 87% and 75% were obtained. In a similar fashion, $CH_2{=}CHSiPh_3$ yielded $t\text{-BuCH}_2\text{H}_2\text{SiPh}_3$ (68%) and $i\text{-PrCH}_2\text{CH}_2\text{SiPh}_3$ (85%) after BH_4^- reduction (kcl = 14 at 0.12 M $CH_2{=}CHSiPh_3$ in Me_2SO).

Vinyl radicals also react as $A^\bullet$ in reaction 3. Thus, reaction 5 has been observed to occur by a chain process to

$$R^1C{\equiv}CR^2 + t\text{-BuHgCl} \xrightarrow{h\nu} \underset{\mathbf{2}}{t\text{-BuC}(R^1){=}\text{C(HgCl)}R^2} \quad (5)$$

yield the vinylmercurial (**2**) which can be reduced by $NaBH_4$ or cleaved by I_2 (Table I) to form a mixture of the appropriate E and Z isomers.[9]

When addition of $R^\bullet$ to an alkene yields a donor radical, reaction 2 can occur. Thus, in Me_2SO, $CH_2{=}C(C_6H_4OMe\text{-}p)_2$ (0.04 M) reacts with t-BuHgCl (0.12 M) in a photostimulated process with an initial kcl of 18 to form $t\text{-BuCH}{=}C(C_6H_4OMe)_2$ (85%) with only traces of $t\text{-BuCH}_2\text{CH}(C_6H_4OMe)_2$.[10]

Reaction of alkenes with RHgCl and BH_4^- leads to reaction 6,[11] provided the alkene can compete effectively with

$$\text{RHgCl} + CH_2{=}CHE \xrightarrow[CH_2Cl_2]{NaBH_4} RCH_2CH_2E + Hg^0 \quad (6)$$

RHgH in the trapping of $R^\bullet$.[6] Some relative reactivities of pertinent alkenes and alkynes in reaction 6 at 25 °C are $CH_2{=}C(Cl)CN$ (52); $CH_2{=}CHCN$ (3.8); $CH_2{=}CHSO_2Ph$ (2.5); $CH_2{=}CHCO_2Et$ (1.0); $EtO_2CC{\equiv}CCO_2Et$ (0.9); VP (0.53);[12] $CH_2{=}CPh_2$ (0.21); $HC{\equiv}CCO_2Et$ (0.19); $CH_2{=}CHPh$ (0.15); $CH_2{=}C(C_6H_4OMe\text{-}p)_2$ (0.11);[13] $CH_2{=}CCl_2$ (0.06); $CH_2{=}CHSPh$ (0.03); $HC{\equiv}CPh$ (0.02); $EtO_2CC{\equiv}CPh$ (0.014); $CH_2{=}CHSOPh$ (0.005); $CH_2{=}CHOEt$, $CH_2{=}CHSiMe_3$, $CH_2{=}CHSnBu_3$, $CH_2{=}CHCMe_3$, $CH_2{=}CHNH_2$, norbornene (all <0.005). When applied to a reactive alkene such as VP, both reactions 6 and 4 (followed by BH_4^-) give high yields of $RCH_2CH_2P(O)(OEt)_2$. However, when applied to less reactive substrates such as $PhC{\equiv}CH$ or norbornene, the Giese reaction gives low yield (8–10% for $PhC{\equiv}CH$, 0% for norbornene) because $R^\bullet$ is preferentially trapped by RHgH.[6] In such cases, the two-step process involving addition of RHgCl to the double or triple bond followed by $NaBH_4$ reduction is preferable.

Alkenes which lead to adduct radicals without strong polar character can yield products of nonchain reactions. Thus, $CH_2{=}CPh_2$ readily traps $t\text{-Bu}^\bullet$ from the photolysis of t-BuHgCl, but the resulting $t\text{-BuCH}_2\dot{\text{C}}\text{Ph}_2$ mainly disproportionate to form $t\text{-BuCH}_2\text{CHPh}_2$ and $t\text{-BuCH}{=}\text{CPh}_2$ (~1:1 ratio).[10] Here, trapping of $t\text{-BuCH}_2\dot{\text{C}}\text{Ph}_2$ by HgCl (from the photolysis of t-BuHgCl) would undoubtedly be reversible and not lead to a stable organometallic. On the other hand, excess norbornene (4 equiv in Me_2SO) forms an adduct radical with $t\text{-Bu}^\bullet$ which is cleanly trapped by HgCl to form a new organomercurial (isolated in 50% yield after 24 h irradiation) which can be reduced by $NaBH_4$ to *exo*-2-*tert*-butylnorbornane.[14] The rate of consumption of norbornene determined by ^1H NMR is equal to the rate of the photochemical dissociation of t-BuHgCl as measured by the "inhibition" period observed in the presence of $(t\text{-Bu})_2NO^\bullet$.

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[8] Based on k_p for polymerization of VP of $1 \times 10^2\ \text{M}^{-1}\ \text{s}^{-1}$ at 40 °C (Levin, Y. A.; Breus, A. A.; Ivanov, B. E. *Dokl. Akad. Nauk SSSR* **1977**, *236*, 154); a 3-fold excess of t-BuHgCl eliminates products derived from attack of $t\text{-BuCH}_2\dot{\text{C}}\text{HP(O)(OEt)}_2$ upon VP. The chain transfer rate constant of VP radical with CCl_4 is $>3 \times 10^2\ \text{M}^{-1}\ \text{s}^{-1}$ (Raynal, S. *Phosphorous Sulfur* **1981**, *11*, 287), but in competition with 10 M CCl_4, 0.1 M t-BuHgCl is also an effective trap for $RCH_2\dot{C}HP(O)(OEt)_2$. Reaction 4 in CCl_4 solution yields $Cl_3CCH_2CH(HgCl)P(O)(OEt)_2$ in 52% (based on the yield of $Cl_3CCH_2CH_2P(O)(OEt)_2$ after BH_4^- reduction).

[9] For example, $t\text{-BuCH}{=}\text{CHPh}$ (Table I) is formed with Z/E = 79/21, the stereoselectivity being controlled by attack of RHgH upon linear $t\text{-BuCH}{=}\dot{\text{C}}\text{Ph}$. Assignment is based upon the known decarboxylation (Cu_2O, quinoline) of $t\text{-BuC}(CO_2H){=}\text{CHPh}$ to predominantly $(Z)\text{-}t\text{-BuCH}{=}\text{CHPh}$ (Buckles, R. E.; Wheeler, N. G. *Organic Synthesis*; Wiley: New York, 1983; Collect. Vol. 4, p 857). From the chemical shift of the vinyl H (δ 7.1 (83%), 6.55 (17%)), the major isomer of $t\text{-BuC}(CO_2Et){=}\text{CHPh}$ (Table I) can be assigned as E (i.e., t-Bu and H are trans). For $t\text{-BuC}(CO_2Et){=}\text{CHCO}_2\text{Et}$, the chemical shifts of δ 5.81 (62%) and 6.18 (38%) allow the assignment of the Z isomer (t-Bu and H are cis) as the major product.

[10] Russell, G. A.; Khanna, R. K.; Guo, D. *J. Chem. Soc., Chem. Commun.* **1986**, 632.

[11] Giese, B. *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 753; **1985**, *24*, 553. Giese, B.; Horlen, H. *Tetrahedron* **1985**, *41*, 4025. Giese, B.; Gröninger, K. *Tetrahedron Lett.* **1984**, *25*, 2473.

[12] Attack of $t\text{-Bu}^\bullet$ upon VP has a rate constant of $5.9 \times 10^4\ \text{M}^{-1}\ \text{s}^{-1}$ at 233 K: Baban, J. A.; Roberts, B. P. *J. Chem. Soc., Perkin Trans. 2* **1981**, 161.

[13] Diarylmethyl radicals apparently react slowly with RHgH. Thus, in reaction **6**, $CH_2{=}C(C_6H_4OMe)_2$ yields exclusively $t\text{-BuCH}{=}C(C_6H_4OMe)_2$ and $CH_2{=}C(Ph)_2$ yields either $t\text{-BuCH}_2\text{CHPh}_2$ or a mixture of $t\text{-BuCH}_2\text{CHPh}_2$ and $t\text{-BuCH}{=}\text{CPh}_2$ at low RHgH concentration.

[14] ^1H NMR evidence (300 MHz) favors the *exo*-2-*tert*-butyl-*exo*-3-(chloromercurio)norborane structure with H(3),H(4) δ 2.28–2.32; H(2) δ 1.64 (d), $J_{2,3}$ = 7.44 Hz; H(1) δ 2.52–2.54 (q); H(*exo*-t-Bu) δ 0.89.

Carboxylative Coupling of Propargylic Alcohols with Allyl Chloride

Summary: Palladium-mediated reaction of lithium 2-alkynyl carbonates with allyl chloride affords cyclic carbonates whose alkaline hydrolysis gives keto alcohols in good yields. The combination of the above procedure with the formation of lithium 2-alkynolates from lithioalkynes and carbonyl compounds provides novel use of lithioalkynes as acyl anion equivalents in the reaction of carbonyl compounds.

Sir: Insertion of carbon dioxide into oxiranes[1-7] or 2-

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[1] Matsuda, H.; Ninagawa, A.; Nomura, R.; Tsuchida, T. *Chem. Lett.* **1979**, 573.

[2] Matsuda, H.; Ninagawa, A.; Nomura, R. *Chem. Lett.* **1979**, 1261.

[3] Nomura, R.; Ninagawa, A.; Matsuda, H. *J. Org. Chem.* **1980**, *45*, 3735.

Scheme I

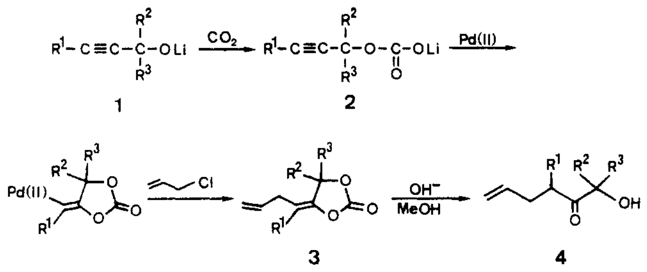


Table I. Preparation of Cyclic Carbonates 3 and Keto Alcohols 4 by Scheme I

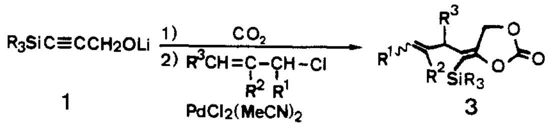
entry	R^1	R^2	R^3	cyclic carbonate[a] product (%)[b]	keto alcohol[a] product (%)[c]
1	Me	H	H	**3a** (64)	**4a** (61)
2	Et	H	H	**3b** (74)	**4b** (59)
3	Bu	H	H	**3c** (66)	**4c** (91)
4	Hex	H	H	**3d** (73)	**4d** (86)
5	Hex	Me	H	**3e** (74)	**4e** (83)
6	Hex	Me	Me	**3f** (50)	**4f** (84)
7	Ph	H	H	**3g** (74)	**4g** (99)
8	Me_3Si	H	H	**3h** (71)	**4h**[d] (51)[d]
9	H	H	H	**3i** (0)	

[a] All compounds described here are new compounds. Structures are determined by ^{1}H NMR (200 MHz) as well as high resolution MS. [b] Yields of cyclic carbonates from propargylic alcohols are shown. [c] Yields of keto alcohols from ethylene carbonates are given. [d] Protodesilylated product was obtained (**4h**, R^1, R^3 = H).

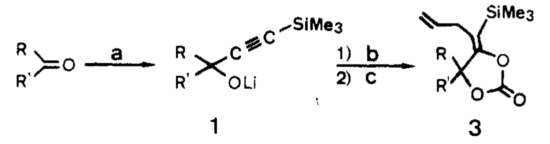
alkynols[8,9] giving cyclic carbonates has attracted considerable attention both by increasing the synthetic value of the products and by the facile fixation of CO_2. Recently reported palladium-catalyzed stereoselective synthesis of butenolides from lithium 4-alkynolates[10] prompted us to think that substitution of CH_2 at the C-2 position with oxygen would give cyclic carbonates bearing stereodefined alkylidene substituents. This report describes the regio- and stereoselective formation of ethylene carbonates **3** by palladium(II)-catalyzed allylative cyclization of lithium 2-alkynyl carbonates **2** which are produced by the reaction of lithium 2-alkyn-1-olates **1** with carbon dioxide (Scheme I).

Carbon dioxide (1 atm, gentle flow for 30 min) was introduced into a THF solution of lithium 2-heptyn-1-olate (**1c**) prepared from 2-heptyn-1-ol (1.12 g, 10.0 mmol in 10 mL of THF) and equimolar butyllithium (5.7 mL of 1.7 M hexane solution). To the resulting solution of lithium 2-heptynyl carbonate (**2c**) were added allyl chloride (15.3 g, 0.2 mol, 20 equiv) and a catalytic amount of $PdCl_2(MeCN)_2$ (130 mg, 0.5 mmol, 0.05 equiv) successively, and the whole was stirred at room temperature for 2 h. Aqueous workup and column chromatography of the reaction mixture afforded (*E*)-(1-allylpentylidene)-2-oxo-1,3-dioxolane (**3c**, 1.30 g, 66% yield)[11] stereoselectively.

Scheme II



Scheme III



a : $LiC\equiv CSiMe_3$
b : CO_2
c : $CH_2=CH-CH_2Cl$, $PdCl_2(MeCN)_2$

Table II. Carboxylate Coupling of 3-Silyl-2-propynols with Substituted Allyl Chlorides by Scheme II

entry	R_3Si	R^1	R^2	R^3	cyclic carbonate[a] product	yield, %
1	Me_3Si	Me	H	H	**3j**	42
2	Me_3Si	H	Me	H	**3k**	24
3	Me_3Si	H	H	Me	**3l**	4
4	$PhMe_2Si$	H	H	H	**3m**	74
5	$PhMe_2Si$	Me	H	H	**3n**	49
6	$PhMe_2Si$	H	Me	H	**3o**	56
7	$PhMe_2Si$	H	H	Me	**3p**	12
8	Ph_2MeSi	H	H	H	**3q**	82
9	Ph_2MeSi	Me	H	H	**3r**	50
10	Ph_2MeSi	H	Me	H	**3s**	62
11	Ph_2MeSi	H	H	Me	**3t**	15

[a] All compounds shown in Table II are new compounds. Structures are determined by ^{1}H NMR (200 MHz) as well as high resolution mass spectrometry.

Analogously, substituted lithium 2-alkynolates **1** afforded the corresponding ethylene carbonates **3** in good yield by successive treatment with carbon dioxide and allyl chloride. Though lithium 2-propynolate did not give adduct **3i**, lithium 3-(trimethylsilyl)-2-propynolate (**1h**) smoothly gave **3h** which afforded **3i** in 50% yield by stereospecific protodesilylation with HI.[12,13] Cyclic carbonates prepared by the above-described procedure are summarized in Table I. Hydrolysis of the above cyclic carbonates **3** afforded 2-oxo-1-alkanols **4** in excellent yields and the results are also in Table I.

Reaction of sodium 2-nonyn-1-olate, in place of lithium alkoxide **1d**, gave cyclic carbonate **3d** in 20% yield, and use of potassium 2-nonyn-1-olate diminished the yield to 13%.

Carboxylative cyclized coupling of lithium 3-(trimethylsilyl)-2-propynolate with substituted allyl chlorides gave satisfactory results; reaction selectively proceeded at the γ position, affording the adduct **3** in good yield (Scheme II). Results are summarized in Table II.

As lithium 2-alkynolates are prepared by the reaction of lithioalkynes with carbonyl compounds, the series of transformations shown in Scheme I affords the novel use of lithioalkynes as acyl anion equivalents. The following example is illustrative: To a THF solution of 1-lithio-2-(trimethylsilyl)ethyne (5.0 mmol, prepared from 5.0

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(5) Baba, A.; Kashiwagi, H.; Matsuda, H. *Tetrahedron Lett.* **1985**, *26*, 1323.

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(9) Ishikawa, J., Inoue, Y.; Hashimoto, H. *Abstracts of Papers*, 52nd Annual Meeting of the Chemical Society of Japan, 1986, 4W04.

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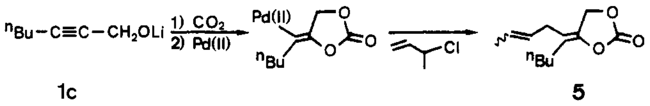
(11) Homogeneity of the product was determined by capillary GLC and stereochemistry was estimated by assuming the analogous stereoselective mechanism discussed in ref 10.

(12) Utimoto, K.; Kitai, M.; Nozaki, H. *Tetrahedron Lett.* **1975**, 2825.

(13) Homogeneity of the product was determined both by GLC and by ^{1}H NMR: olefinic proton at 5.37 (1 H, tt, *J* = 8.0, 2.7 Hz). Calculated value (ref 14) of olefinic proton of **3i** is 5.25 and the value for geometric isomer is 4.72.

(14) Pascual, C.; Meier, J.; Simon, W. *Helv. Chim. Acta* **1966**, *49*, 164.

Scheme IV



mmol of (trimethylsilyl)ethyne and 5.0 mmol of BuLi in THF solution) was added cyclopentanone (420 mg, 5.0 mmol) at −78 °C, and the whole was stirred at 0 °C for 1 h. Carbon dioxide (gas) was blown through the reaction mixture for 30 min at −78 °C. Allyl chloride (8.3 mL, 100 mmol) and $PdCl_2(MeCN)_2$ (65 mg, 0.25 mmol) were added to the reaction mixture maintained at −78 °C, and then the whole was stirred at 0 °C for 4 h. Water was added to the reaction mixture and the whole was extracted with ether. Ethereal solution was washed with brine, dried over Na_2SO_4, and concentrated. Column chromatography of the residue (silica gel/hexane–ethyl acetate) afforded cyclic carbonate **3u** (R,R′ = $-(CH_2)_4-$, 710 mg, 53% yield) and 1-[(trimethylsilyl)ethynyl]-1-cyclopentanol (210 mg, 23% yield). Alkaline hydrolysis of the carbonate **3u** afforded the corresponding keto alcohol **4u** (Scheme III). Analogously, cyclic carbonates **3** were prepared: **3v** (R,R′ = $-(CH_2)_5-$, 19% from cyclohexanone),[15] **3w** (R = Ph, R′ = H, 78% from benzaldehyde),[16] **3x** (R = R′ = H, 47% from paraformaldehyde).

In contrast to the above results with 3-(trimethylsilyl)-2-propynolate, cyclized coupling of lithium 2-alkynyl carbonates with substituted allyl chlorides gave unsatisfactory results: reaction of lithium 2-heptyn-1-olate (**1c**) with 3-chloro-1-butene gave **5**[17] as an *E*/*Z* mixture in 10% yield and reaction with 1-chloro-2-butene did nott give any coupling product (Scheme IV).

The above described palladium-catalyzed three-component reaction of 2-alkyn-1-olates **1**, carbon dioxide, and allyl chloride opens both a novel access to cyclic carbonates **3** and use of lithioalkynes as acyl anion equivalents.

Acknowledgment. We thank The Ministry of Education, Science and Culture for Grant-in-Aid for Developmental Scientific Research (61850153).

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[15] 1-[(Trimethylsilyl)ethynyl]-1-cyclohexanol was obtained in 59% yield. Carboxylative coupling of the above lithio alcoholate with allyl chloride gave **3v** in 59% yield.

[16] The corresponding keto alcohol was obtained in 58% yield by the hydrolysis in aqueous sodium carbonate.

[17] Structures of the two isomers were determined by ^1H NMR (200 MHz) and the ratio of the two isomer was determined by GLC as a well as NMR.

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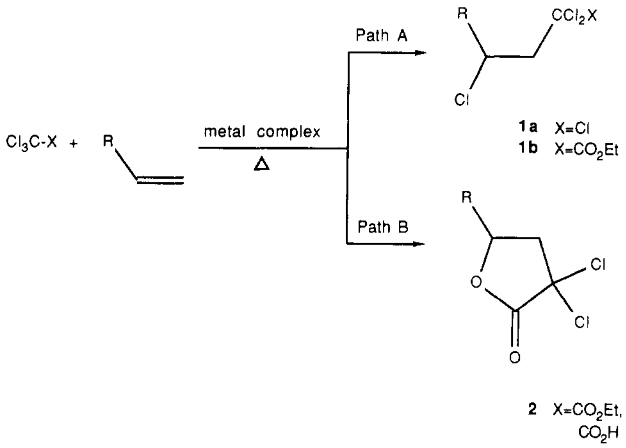
Transition Metal Promoted Intramolecular Cyclizations of α,α-Dichloro Esters and Acids

Summary: Carbocyclic α,γ-dichloro esters or annulated α-chloro γ-lactones can be selectively synthesized via a radical process starting from olefinic α,α-dichloro esters and acids by appropriate choice of transition-metal catalyst and substrate.

Sir: The addition of halocarbons to alkenes catalyzed by various transition-metal compounds has been known for many years.[1] This process, which has been shown to occur through metal-complexed radicals,[2] regioselectively affords adducts such as **1a** (path A, Scheme I). More recently, trichloroacetates were found to undergo analogous radical additions to give α,α,γ-trichloro esters **1b** and/or α,α-dichloro γ-lactones **2**.[3] Interestingly, with some metal catalysts (Cu^{1+}, $Co_2(CO)_8$, Ru^{2+}, etc.) the products of path A predominated, whereas with other catalysts ($[CpMo(CO)_3]_2$, $[CpFe(CO)_2]_2$) path B lactonic products were preferentially formed. Allylic trichloroacetates[4] and trichloroacetamides[5] can also be cyclized intramolecularly via a path A process to afford chlorinated lactones and lactams, respectively. These cyclizations show the regiochemical specificities expected for a free radical reaction.[6] We now report a novel, efficient intramolecular version of this methodology for the synthesis of highly functionalized carbocyclic systems. As discussed below, we have found that the appropriate choice of metal catalyst, or of substrate (i.e. chlorinated ester or carboxylic acid), allows one to efficiently control the nature of the cyclization product (i.e. path A vs. path B).

Scheme I



Initial experiments were carried out with α,α-dichloro ester **3a** and acid **3b**, which were readily prepared by alkylation of 5-bromo-1-pentene with lithio ethyl dichloroacetate[7] and dilithio ethyl dichloroacetic acid,[8] respectively. Cyclizations were conducted in benzene (0.5–1.5 M in substrate) at 160–165 °C in a sealed tube for several hours. Scheme II shows some typical results obtained with this system. α,α-Dichloro ester **3a** cyclized in the expected exo 5-hexenyl radical mode[6] with $RuCl_2(PPh_3)_3$[3c] and $FeCl_2$-

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[1] (a) Elzinga, J.; Hogeveen, H. *J. Org. Chem.* **1980**, *45*, 3957. (b) Bellus, D. *Pure Appl. Chem.* **1985**, *57*, 1827 and references cited therein.

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