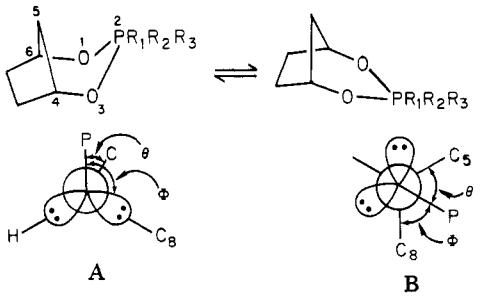
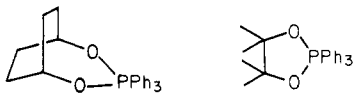
Scheme I



A B

for phosphorus–carbon coupling similar to that observed in P(III) and P(IV) phosphorus derivatives.[12] Construction of an accurate Karplus curve[13] from this data is not possible because of the limited amount of data and the absence of accurate structural information from X-ray crystallography.

We have also examined this reaction kinetically and report the activation barriers for the formation of phosphoranes **5**, **9**, and **10**[14] in Table II. The reaction is en-



9 **10**

thalpy controlled. The activation entropies are approximately the same for all three bimolecular reactions. The increasing ring strain in the peroxides is reflected in a decreasing enthalpy of activation.

Experimental Section

Materials. The synthesis of bicyclic peroxide **4** and 2,3-dioxabicyclo[2.2.2]octane was accomplished as reported previously.[15] Both compounds gave satisfactory spectral and physical data. The triphenylphosphine, methyl diphenylphosphinite, and dimethyl phenylphosphonite were obtained from Aldrich and used without further purification. Trimethyl phosphite was received from Eastman Kodak and distilled off of sodium before use. Benzene was distilled in a N_2 atmosphere off of benzophenone ketyl and then stirred over the disodium salt of EDTA. The *n*-decane was distilled at 5 mmHg and then stirred over EDTA.

Dioxaphosphoranes 5–8. These compounds were synthesized by addition of triphenylphosphine, methyl diphenylphosphinite, dimethyl phenylphosphonite, and trimethyl phosphite via syringe to a serum-capped 10-mm NMR tube containing **4** and benzene at −78 °C. The reaction mixture was then allowed to warm slowly to room temperature and the spectral data was collected.

NMR Measurements. The ^{31}P NMR measurement were made on a JEOL-FX270 MHz instrument at 109.13 MHz. A total of 16 384 points were collected over a spectral width of 50 000 Hz, utilizing a pulse delay of 5 or 10 s. All the chemical shifts are reported relative to 85% H_3PO_4 by substitution. The proton and ^{13}C NMR data were also collected on a JEOL-FX270 MHz instrument and the data referenced to tetramethylsilane by substitution.

Kinetic Measurements. A 1.7-mL sample of 2.0×10^{-2} M **4** in *n*-decane was mixed with 1.7 mL of 2.0×10^{-4} M PPh_3 also in *n*-decane in a 1-mm curvette thermostated at the desired temperature. The psuedo-first-order disappearance of PPh_3 was monitored at 290 nm for at least 3 half-lives.

Data Treatment. Each rate constant was determined by following the decrease in absorbance of PPh_3 at 290 nm for at least 3 half-lives and plotting ln $(A_0 - A_\infty)$ vs. time. All rate constants were obtained by linear regression analysis of the experimental data and resulted in correlation coefficients (r) of greater than −0.997 in each case. The reported rate constants were obtained by dividing the pseudo-first-order rate constants by the concentration of **4** and are the average of three independent determinations.

The activation parameters were determined by plotting ln (k/T) vs. $1/T$ and the confidence limits in the slope and intercept were calculated with the equations of Bennett and Franklin[16] and propagated into the activation parameters at the 95% confidence level.

Acknowledgment. We thank the University of Wyoming for support of this work. We also thank Professor Alfons L. Baumstark (Department of Chemistry, Georgia State University, Atlanta, Ga) for graciously allowing us to quote his activation barrier for the reaction of **10** prior to its publication.

Registry No. **4**, 279-35-6; **5**, 78870-51-6; **6**, 81898-36-4; **7**, 81898-37-5; **8**, 81898-38-6; **9**, 81898-39-7; **10**, 49595-63-3; triphenylphosphine, 603-35-0; methyl diphenylphosphinite, 4020-99-9; dimethyl phenylphosphonite, 2946-61-4; trimethyl phosphite, 121-45-9.

(12) (a) Quin, L. D.; Gallagher, M. J.; Cunkle, G. T.; Chesnut, D. B. *J. Am. Chem. Soc.* **1980**, *102*, 3136. (b) Thiem, J.; Meyer, B. *Org. Mag. Reson.* **1978**, *11*, 50.

(13) (a) Karplus, M. *J. Chem. Phys.* **1959**, *30*, 11. (b) Barfield, M.; Karplus, M. *J. Am. Chem. Soc.* **1969**, *91*, 8305.

(14) The activation barriers for the formation of **10** was measured by Professor Alfons L. Baumstark (Georgia State University).

(15) (a) Porter, N. A.; Gilmore, D. W. *J. Am. Chem. Soc.* **1977**, *99*, 3503. (b) Salomon, R. G.; Salomon, M. F. *Ibid.* **1977**, *99*, 3501. (c) Coughlin, D. J.; Brown, R. S.; Salomon, R. G. *Ibid.* **1979**, *101*, 1533.

(16) Gordon, A. J.; Ford, R. A. "The Chemist Companion"; Wiley-Interscience: New York, 1972; p 491.

Phenylmercuric Hydroxide. A Highly Selective Reagent for the Hydration of Nonconjugated Terminal Alkynes[1]

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Received January 26, 1982

We herein describe an unusual and highly selective method for hydrating nonconjugated terminal alkynes based on the use of phenylmercuric hydroxide (**1**) as a reagent. Unlike classical mercury catalyzed procedures, σ-bonded mercury acetylides are formed initially as stable intermediates and subsequently reacted with water under neutral pH to form the corresponding methyl ketone.[3,4] Isolated yields which have been obtained by using this approach lie in the range of 49–65%.

Addition of phenylmercuric hydroxide to a chloroform (or carbon tetrachloride) solution of 1-heptyne followed by heating for 2 h at 60 °C produced a pale yellow solution. Direct examination by IR, ^1H NMR, and ^{13}C NMR indicated complete conversion to the acetylide (eq 1), accom-

$$PhHgOH + CH_3(CH_2)_4C{\equiv}CH \xrightarrow[CHCl_3]{60\ ^\circ C} PhHgC{\equiv}C(CH_2)_4CH_3 + H_2O \quad (1)$$

(1) Supported by the Division of Basic Energy Sciences of the Department of Energy (Contract EG-77-S-02-4446).

(2) On leave from the Institute of Macromolecular Chemistry of the Czechoslovak Academy of Sciences.

(3) For conventional mercury-catalyzed hydrations, see: Budde, W. L.; Dessy, R. E. *J. Am. Chem. Soc.* **1963**, *85*, 3964. Parshall, G. W. "Homogeneous Catalysis"; Wiley-Interscience: New York, 1980. Stacy, G. N.; Mikulec, R. A. "Organic Syntheses"; Wiley: New York, 1963; Collect. Vol. IV, p 13.

(4) For examples of other mercury reagents used in alkyne hydration, see: Goldberg, H. W.; Aeshbacher, R.; Hardegger, E. *Helv. Chim. Acta* **1943**, *26*, 680. Kagan, H. B.; Marquett, A.; Jacques, J. *Bull. Soc. Chem. Fr.* **1960**, 1979.

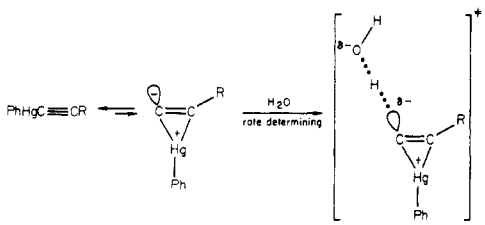
0022-3263/82/1947-3331$01.25/0

$$PhHgC{\equiv}C(CH_2)_4CH_3 \xrightarrow[CHCl_3/H_2O]{60\ ^\circ C} CH_3C(O)(CH_2)_4CH_3 \quad (2)$$

panied by trace amounts of 2-heptanone. Addition of an aqueous phase and subsequent heating (25 h, 60 °C) resulted in the complete decomposition of the acetylide and further formation of 2-heptanone (eq 2).[5] Separation of the organic layer followed by distillation afforded a 56% isolated yield of the ketone. Two crossover experiments demonstrate that hydration of the mercury acetylide is preferred over the parent alkyne (present in small but finite concentrations at equilibrium). Thus, addition of 1 equiv of 1-hexyne to a chloroform solution of the mercury acetylide of 1-heptyne followed by reaction with water resulted in a ratio of 2-heptanone/2-hexanone of 5.5 after 1 h.[6] In a similar conversion, the acetylide of 1-hexyne was hydrated in the presence of 1 equiv of 1-heptyne and yielded a ratio of 2-hexanone/2-heptanone of 4.0.

Compared to conventional hydration procedures, 1 is highly selective for the hydration of nonconjugated terminal alkynes. Attempted hydration of internal acetylenes (e.g., 4-decyne) with 1 afforded only unreacted starting material. Conjugated terminal alkynes such as phenylacetylene, (*p*-methoxyphenyl)acetylene, and ethyl propiolate react smoothly with 1 to form the corresponding acetylides; the latter, however, are remarkably unreactive toward water.[7] If one compares these results with a closely related mercury-catalyzed system [chloroform–water (10 mol % of $HgSO_4$ + H_2SO_4)] at 50 °C, one finds that phenylacetylene undergoes hydration at a rate comparable to that of 1-heptyne. Also, while 4-decyne reacts more slowly than 1-heptyne, the selectivity is not nearly as great as that found with 1. The potential utility of phenylmercuric hydroxide is further demonstrated by its ability to tolerate a variety of functional groups such as acetals, thioacetals, lactones, alkenes, epoxides, and secondary bromides. Thus, hydration of 1-heptyne could be successfully performed in the presence of 2-phenyl-1,3-dithiacyclohexane, acrolein diethyl acetal, ϵ-caprolactone, 1-decene, α-methylstyrene, cyclohexene oxide, or 2-bromooctane without any detectable decomposition of the latter; such functionalities are, however, unstable under the catalytic conditions described above. Finally, it is interesting and significant to note that ethynylcarbinols, e.g., 1-ethynylcyclohexanol and 1-ethynylcyclopentanol, can also be converted to α-hydroxy ketones in good yields with 1. Such transformations play an important role in many natural product syntheses.

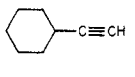
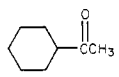
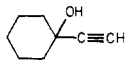
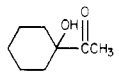
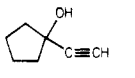
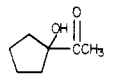
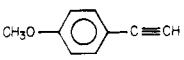
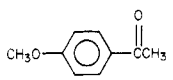
(5) While the mechanistic details of the hydration step remain to be established, the existence of a deuterium isotope effect in the initial hydration rate (k_H/k_D = 1.9, when D_2O is used), together with the ability of mercury to support a positive charge, leads us to suggest the involvement of the following zwitterionic intermediate and transition state:



(6) Analysis of the product mixture (GLC) revealed the presence of a significant quantity of 1-heptyne, indicating that some alkyne–acetylide exchange had occurred. It is presumed that most, if not all, of the 2-hexanone produced is derived from the corresponding acetylide.

(7) After attempted hydration of phenylacetylene failed, the chloroform layer was treated with an aqueous solution containing 1.5 equiv of HCl (1 h, 50 °C). A quantitative recovery of phenylacetylene was thus obtained, along with a 79% isolated yield of phenylmercuric chloride.

Table I. Hydration of Alkynes with Phenylmercuric Hydrixide[a]

reactant	product	isolated yield, %
$CH_3(CH_2)_4C{\equiv}CH$	$CH_3(CH_2)_4C(O)CH_3$	56
$CH_3(CH_2)_3C{\equiv}CH$	$CH_3(CH_2)_3C(O)CH_3$	67[b]
$CH_3(CH_2)_7C{\equiv}CH$	$CH_3(CH_2)_7C(O)CH_3$	55
		49
		65[c]
		57[c]
$CH_3(CH_2)_3$-$C{\equiv}C(CH_2)_3CH_3$	$CH_3(CH_2)_3$-$C(O)(CH_2)_4CH_3$	0
$PhC{\equiv}CH$	$PhC(O)CH_3$	0[d]
		0[d]
$C_2H_5OC(O)C{\equiv}CH$	$C_2H_5OC(O)$-$C(O)CH_3$	0[d]

[a] Reaction of 10 mmol of alkyne with 10 mmol of 1 followed by hydrolysis using procedures similar to those described in the Experimental Section. [b] GLC yield. [c] Reaction of alkyne with 1 in $CHCl_3$ at 50 °C for 3 h; in this case, the addition of water and further heating was unnecessary. [d] Quantitative conversion to mercury acetylide.

The high selectivity toward nonconjugated terminal alkynes which characterizes the procedure described herein should make it a useful supplement to existing hydration methods.

Experimental Section

General Methods. Unless stated otherwise, all reagents and chemicals were obtained commercially. Phenylmercuric hydroxide (Aldrich Chemical Co.) was used without further purification. All ^1H NMR, ^{13}C NMR, and IR spectra were recorded on Varian A-60, JEOL FX 60Q, and Beckman Acculab 7 spectrometers, respectively. Chemical shifts were measured in $CDCl_3$ and expressed in δ relative to tetramethylsilane. Product mixtures were analyzed by GLC on a Hewlett-Packard Model 5830 A flame-ionization instrument (2 ft × 0.125 in. UCW-982 on Chromosorb W column).

General Hydration Procedure. Procedures similar to that described for the conversion of 1-heptyne to 2-heptanone were followed for all of the reactions reported in Table I. Addition of 2.95 g (10 mmol) of phenylmercuric hydroxide to 10 mL of 1.0 M of 1-heptyne in chloroform followed by heating for 2 h at 60 °C produced a pale yellow solution accompanied by the formation of 0.12 g of metallic mercury. Direct analysis of the organic layer indicated the formation of the corresponding acetylide: IR ($CDCl_3$) 3070, 3020, 2990, 2940, 2910, 2840, 2340, 2150 ($\nu_{C{\equiv}C}$), 1720 ($\nu_{C{=}O}$, vw), 1650, 1570, 1460, 1420, 1250, 1160, 1090, 1060, 1030, 1000 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.90 (t, 3 H, CH_3CH_2), 1.40 (m, 8 H, $(CH_2)_4$), 7.30 (m, 5 H, Ph); ^{13}C NMR ($CDCl_3$) δ 19.5 ($J_{^{199}Hg-^{13}C}$ = 3672 Hz, $HgC{\equiv}C$). The acetylenic proton of the parent alkyne could not be detected by the IR ($CDCl_3$) $\nu_{C{\equiv}CH}$ signal at 3300 cm^{-1} and the ^1H NMR ($CDCl_3$) signal at δ 1.9 (t, 1 H, $HC{\equiv}C$, J = 2Hz).

Water (10 mL) was then added to the chloroform solution and the resulting two-phase system heated for 24 h at 60 °C. During this time a small amount of 1 (0.067 g) precipitated from solution and was removed by centrifugation. Direct spectroscopic examination of the organic layer indicated the presence of 2-heptanone and 1: IR ($CDCl_3$) 3020, 2990, 2940, 2910, 2840, 1720 ($\nu_{C{=}O}$, s), 1650, 1560 (Hg associated carbonyl),[8] 1460, 1430, 1260, 1160, 1060,

(8) Murahashi, S.; Nozabura, S.; Fuji, S. *Bull. Chem. Soc. Jpn.* **1965**, *38*, 1840.

1020, 1000, 900, 850 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.90 (t, 3 H, CH_3CH_2), 1.25 (m, 6 H, CH_2), 2.10 (s, 3 H, CH_3CO), 2.40 (t, 2 H, CH_2CO), 7.25 (m, 5 H, Ph). The aliphatic region of the ^{13}C NMR spectrum was identical with that of authentic 2-heptanone.[9] Evaporation of chloroform followed by distillation (Kugelrohr) afforded 0.64 g (56%) of 2-heptanone having IR and ^1H NMR spectra which were identical with those of an authentic sample.

Registry No. 1, 100-57-2; $CH_3(CH_2)_4C{\equiv}CH$, 628-71-7; $CH_3(CH_2)_3C{\equiv}CH$, 693-02-7; $CH_3(CH_2)_7C{\equiv}CH$, 764-93-2; $CH_3(CH_2)_3C{\equiv}C(CH_2)_3CH_3$, 1942-46-7; $PhC{\equiv}CH$, 536-74-3; $C_2H_5OC(O)C{\equiv}CH$, 623-47-2; $CH_3(CH_2)_4C(O)CH_3$, 110-43-0; $CH_3(CH_2)_3C(O)CH_3$, 591-78-6; $CH_3(CH_2)_7C(O)CH_3$, 693-54-9; deuterium, 7782-39-0; cyclohexylacetylene, 931-48-6; (1-hydroxycyclohexyl)acetylene, 78-27-3; 1-ethynylcyclopentanol, 17356-19-3; *p*-ethynylanisole, 768-60-5; acetylcyclohexane, 823-76-7; 1-acetylcyclohexanol, 1123-27-9; 1-acetylcyclopentanol, 17160-89-3; 1-heptynylphenylmercury,, 82080-25-9.

(9) Treatment of such chloroform solutions with aqueous hydrochloric acid (1.5 equiv) for 1 h at 50 °C led to a 79% isolated yield of phenylmercuric chloride (presumably from reaction with regenerated 1). Efforts at developing a catalytic system based on 1 failed, apparently due to the inability of 1 to react further with alkynes after being in contact with an aqueous phase. In a control experiment, 1 showed no reactivity toward 1-heptyne present in a water–chloroform mixture.

Efficient Regiocontrolled Synthesis of Sarkomycin and Homosarkomycin

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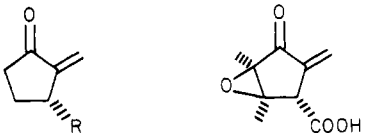
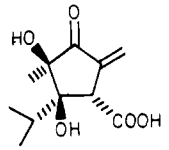
Received January 25, 1982

Sarkomycin (1), parent member of the cyclopentanoid class of antibiotic–antitumor agents which now includes methylenomycin A (3),[2] xanthocidin (4),[3] and pentenomycin I (5),[4] has attracted considerable attention as a synthetic target since its initial isolation in 1953[5] and structural elucidation in 1955.[6,7] By and large, however, published routes to this antitumor agent have been nonregiocontrolled.[8] Indeed, only the very recent synthesis by Marx and Minaskanian (1979),[9] the elegant chiral approach of Boeckman and collaborators (1980),[10] and the palladium-catalyzed cyclization route of Tsuji and Kobayashi (1981),[11] are in fact regiocontrolled. Each of these sequences, however, has the disadvantage of length (ca. 9 or 10 steps).



1, R = COOH
2, R = CH_2COOH

3



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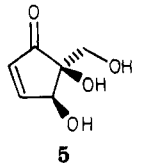
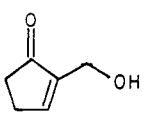
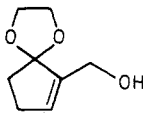


5

In connection wiith our continuing interest in the cyclopentanoid class of antibiotics, we are pleased to record here the synthesis of both sarkomycin (1) and its congener homosarkomycin (2).[12] Both synthetic sequences are short, efficient, and regiocontrolled; furthermore, both take advantage of the ready availability of α-(hydroxymethyl)cyclopentenone (6) or its immediate precursor ketal



6



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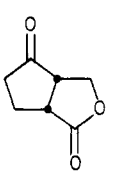

8

7, prepared by application of the α-oxovinyl anion methodology developed recently in our[13] and other laboratories.[14] Finally we note that our approach to sarkomycin takes advantage of the Marx–Minaskanian[9] acid-catalyzed retrolactonization of lactone 8 (i.e., cyclosarkomycin).

Sarkomycin. With cyclosarkomycin (8) as our initial target, conjugate addition of lithium divinylcuprate[15] to α-(hydroxymethyl)cyclopentenone (6) at −78 °C afforded, after the usual workup (saturated aqueous NH_4Cl), unsaturated keto alcohol 9 in 73% yield as an epimeric mixture. Ozonolysis of the latter (−78 °C, CH_2Cl_2), fol-

(1) Camille and Henry Dreyfus Teacher Scholar, 1978–1983; NIH Career Development Awardee, 1980–1985.

(2) Haneishi, T.; Kitahara, N.; Takiguchi, Y.; Arai, M.; Sugawara, S. *J. Antibiot.* **1974**, *27*, 386. Haneishi, T.; Terahara, A.; Arai, M.; Hata, T.; Tamura, C.; *Ibid.* **1974**, *27*, 393. Haneishi, T.; Terahara, A.; Hamano, K.; Arai, M. *Ibid.* **1974**, *27*, 400. For synthesis of 3 see: (a) Scarborough, R. M.; Smith, A. B., III *J. Am. Chem. Soc.* **1977**, *99*, 7085. Also see: Scarborough, R. M.; Toder, B. H.; Smith, A. B., III *J. Am. Chem. Soc.* **1980**, *102*, 3904. (b) Jernow, J.; Tautz, W.; Rosen, P.; Blount, J. F. *J. Org. Chem.* **1979**, *44*, 4210. (c) Koreeda, M.; Liang, Y. P.; Akagi, H. 178th National Meeting of the American Chemical Society, Washington, DC, 1979; American Chemical Society: Washington, DC, 1979; ORGN 38. (d) Takahaski, Y.; Isobe, K.; Hagiwara, H.; Kosugi, H.; Uda, H. *J. Chem. Soc., Chem. Commun.* **1981**, 714.

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