jugated to and coplanar with the benzene ring. For bidentate coordination to occur, the oxygen lone pair(s) must be turned inward and the alkyl or alkoxyalkyl side chain must be turned away. The geometry will vary slightly depending on the extent to which the aryl oxygen is sp^2 hybridized but the side chain will be remote and unable to fully coordinate to the cation as the corresponding lariat ethers do.[6-8]

It appears that when an integral crown ring is present, it binds the cation and the nitro group serves as an additional donor. In contrast, the acyclic compound is a poorer cation binder, increasing the importance of the *ortho* oxygen atom. Once bidentate complexation occurs, the polyethyleneoxy chain cannot fully wrap about the cation so even its weak binding potential cannot be fully realized. It should be noted that the possibility exists that nitroarene-substituted podands having different geometrical arrangements might function as the lariat ethers do. Even so, this is an unusual instance in which acyclic podands fail to afford similar properties to those of the crown ethers.

Experimental Section

Reagents and Solvents. Acetonitrile (MCB, distilled in glass) was stored over Linde 4-A molecular sieves and flask-to-flask distilled from CaH_2 in a vacuum line immediately before use. All solutions were prepared under an inert atmosphere of dry N_2 gas. Tetrabutylammonium perchlorate ($Bu_4N^+ClO_4^-$, TBAP, from MCB) was recrystallized twice from EtOAc and stored in a desiccator. Alkali metal perchlorate salts were recrystallized from deionized water and dried in a vacuum oven at 110 °C for 24 h.

Apparatus. A standard, three-compartment cell, glassy carbon (0.35 cm^2 surface), and Pt wire electrodes were used. $E^{\circ\prime}$ values are reported vs. a saturated aqueous calomel electrode (SCE). The measurements were done on a Bioanalytical Systems (Model CV-1B) apparatus (which does not provide for IR drop compensation) and recorded on a Hewlett-Packard Moseley 7035-B x-y recorder. A 100 mv/s sweep rate was used for all cyclic voltammetry experiments. All transfers were effected by syringe. The electroactive species were present in millimolar concentrations.

Substrates. 2-Nitroanisole (1) was obtained from Aldrich Chemical Co. and distilled prior to use. 2-[(2-Nitrophenoxy)methyl]-15-crown-5 (2) was prepared as previously described[6] from 1-chloro-2-nitrobenzene. 2-(1,4,7,10,13,16,19,22,25-Nonaoxahexaeicosyl)nitrobenzene, also called 2-[methoxyocta(ethoxy)]-nitrobenzene (3), was obtained in 68% yield as a faintly yellow oil by treating $CH_3(OCH_2CH_2)_8OH$ (PEG monomethyl ether, MW 350) with NaH and 1-chloro-2-nitrobenzene in THF solution. Attempted distillation of 3 (or 4, see below) led to decomposition. Osmometric molecular weight determination (494 ± 15 daltons) showed that an average of 7.8 ethyleneoxy units were present in the side chain. A previous, independent molecular weight determination[4d] on the starting ether gave a molecular weight of 400, suggesting approximately 8 ethyleneoxy units. The analysis below is calculated for $n = 8$. Anal. Calcd for $C_{23}H_{39}NO_{11}$: C, 54.65; H, 7.72; N, 2.77. Found: C, 54.45; H, 7.66; N, 3.23. Note that the nitrogen value is high (+0.46) as suggested by the integral and molecular weight data. ^1H NMR ($CDCl_3$), best fit of integral is for $n = 7$: 8.0-6.8 (m, 4 H), 4.4-3.5 (m, 28 H), 3.35 (s, 3 H). IR (neat, strong bands) 2900, 1600, 1525, 1350, 1280, 1120 cm^{-1}. Compound 4 was obtained similarly in 61% yield as a faintly yellow oil. Its ^1H NMR and IR spectral properties were essentially identical with those of 3. Anal. Calcd for $C_{31}H_{55}NO_{15}$: C, 54.63; H, 8.08; N, 2.06. Found: C, 54.36; H, 8.30; N, 2.40.

Registry No. 1, 91-23-6; 2, 87453-20-1; 3, 92670-57-0; 4, 92670-58-1; $CH_3(OCH_2CH_2)_8OH$, 25990-96-9; 1-chloro-2-nitrobenzene, 88-73-3.

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Stereospecific Cobalt-Mediated Enediyne Cyclization Involving a Tetrasubstituted Double Bond: One-Step Construction of the Hydrophenanthrene Nucleus Incorporating Two Adjacent Quaternary Centers

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Received June 29, 1984

We have recently reported that intramolecular enediyne [2 + 2 + 2] cycloadditions are effected by (η^5-cyclopentadienyl)cobalt dicarbonyl [$CpCo(CO)_2$] with pronounced chemo-, regio-, and stereoselectivity, giving rise to complex polycycles from acyclic starting materials, including steroids.[1] The range of double bonds employed in these transformations encompassed 1-mono-,[1b] 1,1-[1c] or 1,2-di-,[1d] and 1,1,2-trisubstituted[1d] patterns. We report in this note that *tetrasubstituted* double bonds appear to be equally capable of entering the cyclization manifold, showing no sign of steric hindrance, unlike other cycloaddition reactions, such as the Diels-Alder, [3 + 2], and other cyclization strategies. The reaction proceeds with complete stereospecificity with respect to the original alkene moiety and also the complexed cobalt center and allows the one-step construction of a tricyclic diene containing two adjacent quaternary carbons starting from acyclic material.

Scheme I outlines an efficient stereospecific approach to the synthesis of starting enediyne 7, employing five metals (Li, Al, Zr, Mg, Pd) other than cobalt in various steps, a powerful demonstration of the utility of both main group and transition metals in organic synthesis.

The sequence chosen en route to starting material begins with the protected 5-hexyn-1-ol 1[2,3] which is alkylated with 4-bromobutanol tetrahydropyranyl ether[3] to give 2. Attempted carboalumination[4] of this compound was unsuccessful, only starting material being recovered. The protecting groups were therefore removed to furnish diol 3 which was added to a solution of 1 equiv of Cp_2ZrCl_2 and 7 equiv of trimethylaluminum in dichloroethane. The resulting lemon yellow solution was heated at 50 °C for 48 h. At this point the product could be trapped at −30 °C with 3 equiv of iodine in THF to give 4, in addition to a small amount of a product assumed to be derived from hydrolysis of the vinylaluminum intermediate. The second methyl group was introduced by initially protecting 4 and subsequently exposing it to palladium-catalyzed (10%) Grignard coupling conditions[5] to furnish 5. Deprotection was followed by an unsuccessful attempt to turn the resulting enediol into the corresponding diiodide directly.[6] Similarly, ditosylate formation from the diol was sluggish and gave low yields. Finally, dimesylate formation oc-

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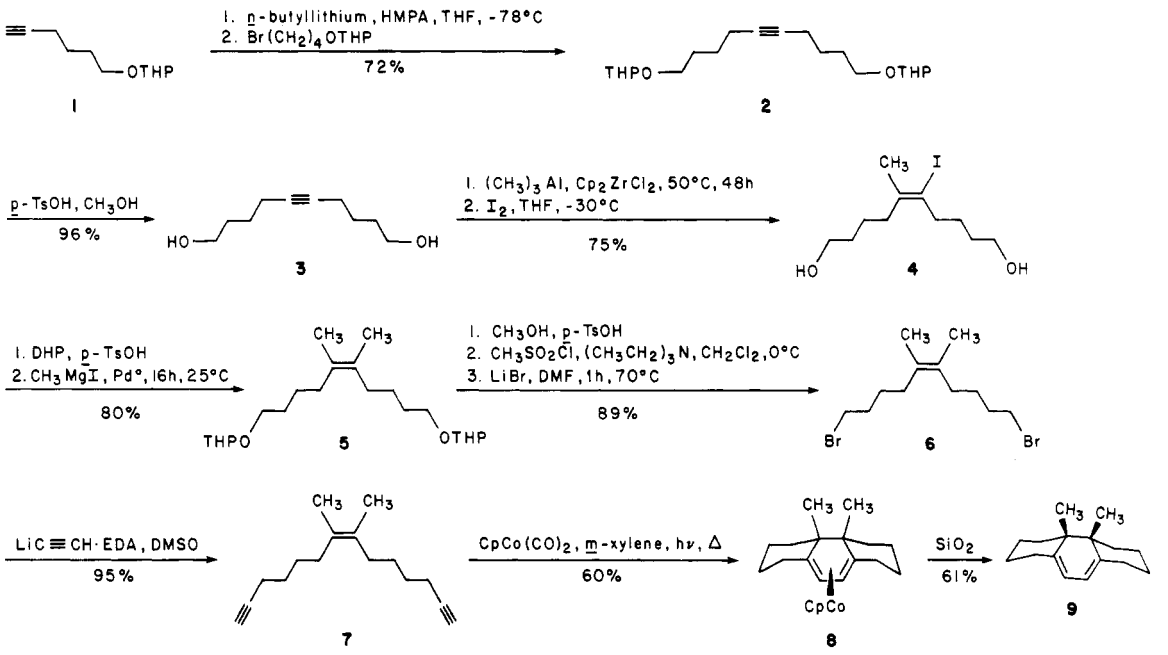
0022-3263/84/1949-5010$01.50/0

Scheme I



curred rapidly and efficiently to set the stage for the uneventful dibromide **6** generation. 7,8-Dimethyltetradec-7-enediyne (**7**) was obtained by reaction of **6** with lithium acetylide–ethylenediamine complex[7] in Me_2SO.

Cyclization was carried out under standard conditions[1] in *m*-xylene heated to 120 °C and irradiated with an ordinary slide projector lamp to give the orange-red complex **8**. The ^1H NMR spectrum clearly revealed that only one isomer had formed, with the metal and the two methyl groups located on the same side of the molecule, as evidenced by the highly characteristic deshielded proton absorptions[1c,d] at δ 1.21. The cyclization yield is better than shown because the free ligand **9** is also formed in this transformation (20–25%), indicating that the [2 + 2 + 2] cycloaddition step may be turned catalytic by appropriate modification of the reaction conditions.[8] The suggested lability of the complex with respect to demetalation is also indicated by the conversion of **8** to **9** on filtration through silica gel.

The free ligand **9** is very air-sensitive and reacts with oxygen to give a paraffin wax like compound which quickly turns yellow with decomposition. A mass spectrum of this product showed a peak at *m/e* 248 (free ligand + O_2). It is possible that **9** adds oxygen in a 1,4-manner to give an endoperoxide[9] which further decomposes.

The reported work, although preliminary in its scope, provides a considerable extension of the use of cobalt-mediated [2 + 2 + 2] cycloadditions in the construction of even highly hindered polycyclic systems, of obvious use in total and other synthesis.

Experimental Section

The general procedures followed in the execution of this work are the same as those described in ref 1b,c.

5-Decyne-1,10-diol Bis(tetrahydropyranyl ether) (2). To a solution of the tetrahydropyranyl ether 1[2,3] (5.46 g, 30 mmol) in dry THF (75 mL) was added at −78 °C under nitrogen 1 equiv of *n*-butyllithium in hexane within 30 min. The temperature was allowed to rise to 0 °C within 30 min and then lowered to −78°C. One equivalent of HMPA (5.4 g) was added, followed by 4-bromobutanol tetrahydropyranyl ether[3] (7.11 g, 30 mmol) within 30 min. The mixture was stirred for 12 h under nitrogen. Aqueous workup and chromatography on alumina (activity III) (ether–pentane, 1:9, as eluent) left **2** (7.3 g, 73%): colorless oil; IR (neat) 2980, 2860, 2840, 1440, 1420, 1150, 1120, 1090, 1060, 1030 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 4.55 (t, J = 3.4, 2 H), 3.90–3.65 (m, 4 H), 3.55–3.30 (m, 4 H), 2.16 (t, J = 6.7, 4 H), 1.9–1.4 (m, 20 H).

5-Decyne-1,10-diol (3). A solution of **2** (3.38 g, 10 mmol) and *p*-toluenesulfonic acid (10 mg) in methanol (20 mL) was stirred at 22 °C. The reaction was monitored by TLC. When all the starting material had disappeared, potassium carbonate (1 g) was added and the mixture was stirred for an additional hour. After filtration, the volatiles were removed and the crude product was chromatographed on silica gel (ether as eluent) to give **3** (1.64 g, 96%): colorless oil; IR (neat) 3340, 2940, 2865, 1060 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 3.64 (t, J = 6.5, 4 H), 2.17 (t, J = 6.8, 4 H), 1.7–1.45 (m, 8 H). Anal. Calcd for $C_{10}H_{18}O_2$: C, 70.55; H, 10.65. Found: C, 70.36; H, 10.29.

5-Iodo-6-methyl-5-decene-1,10-diol (4). To a slurry of bis(cyclopentadienyl)zirconium dichloride (1.5 g, 5 mmol) in 1,2-dichloroethane (25 mL, freshly distilled from CaH_2) was added 7 equiv of trimethylaluminum (3.6 mL, 35 mmol) (pyrophoric!). To the lemon yellow solution thus obtained was added dropwise 5-decyne-1,10-diol (**3**) (0.85 g, 5 mmol) in dichloroethane (7 mL) at 0 °C. A rapid evolution of methane occurred. The mixture was stirred 48 h at 50 °C. Subsequently, iodine (2.83 g, 11 mmol) in freshly distilled THF (10 mL) was added dropwise within 30 min at −30 °C. The mixture was hydrolyzed with 3 N HCl at 0 °C and subjected to aqueous workup. Filtration through silica gel to remove the remainder of the zirconium was followed by chromatography on silica (ether–ethylacetate, 4:1, as eluent) to give **4** (1.20 g, 75%): colorless oil; IR (neat) 3340, 2950, 2860, 1635, 1215, 1060, 790 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 3.65 (m, 4 H), 2.56 (t, J = 7, 2 H), 2.23 (t, J = 7, 2 H), 1.91 (s, 3 H), 1.56 (m, 8 H); MS, *m/e* (relative intensity) 185 (M^+ − I, 62), 167 (71), 149 (84), 77 (100). Anal. Calcd for $C_{11}H_{21}IO_2$: C, 42.32; H, 6.78. Found: C, 42.65; H, 6.75.

5-Iodo-6-methyl-5-decene-1,10-diol Bis(tetrahydropyranyl ether). To a solution of **4** (0.936 g, 3 mmol) in freshly distilled dihydropyran (7 mL) was added at 0 °C *p*-toluenesulfonic acid (5 mg). The reaction mixture was allowed to warm to room temperature and the reaction was monitored by TLC. When all the starting material had disappeared, potassium carbonate (0.5 g) was added. After evaporation of the volatile materials, the residue was purified on alumina (activity III) to give the diether (1.30 g, 90%): colorless oil; IR (neat) 2940, 2860, 1635, 1455, 1440,

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1450, 1140, 1120, 1070, 1035, 1020 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 4.55 (m, 2 H), 3.9–3.65 (m, 4 H), 3.55–3.30 (m, 4 H), 2.55 (bt, J = 7, 2 H), 2.20 (bt, J = 7, 2 H), 1.90 (s, 3 H), 1.65–1.4 (m, 20 H); MS, m/e (relative intensity) 353 (M^+ – I, 22), 269 (31), 185 (37), 149 (40), 85 (75), 53 (100); HRMS calcd for $C_{21}H_{37}O_4$ 353.2691, found 353.2696.

5,6-Dimethyl-5-decene-1,10-diol Bis(tetrahydropyranyl ether) (5). In a flask was placed tetrakis(triphenylphosphine)-palladium (0.140 g, 0.116 mmol) under N_2. A solution of the above iodo diether (0.96 g, 2 mmol) in freshly distilled benzene (35 mL) was added, and the mixture stirred at room temperature for 30 min. Three equivalents of methylmagnesium iodide in ether were injected into the flash with a syringe over 12 h. After aqueous workup, chromatography on alumina (activity III) eluting with ether–petroleum ether (1:9) gave **5** (0.602 g, 80%): colorless oil; IR (neat) 2940, 2860, 1455, 1440, 1350, 1140, 1120, 1070, 1035 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 4.55 (t, J = 3.75, 2 H), 3.85–3.70 (m, 4 H), 3.50–3.33 (m, 4 H), 2.02 (t, J = 7.5, 4 H), 1.59 (s, 6 H) 1.8–1.4 (m, 20 H); MS, m/e (relative intensity) 368 (M^+, 5), 200 (8.2), 167 (3.2), 149 (20), 85 (100); HRMS calcd for $C_{22}H_{40}O_4$ 368.2927, found 368.2928.

5,6-Dimethyl-5-decene-1,10-diol. To a solution of **5** (0.60 g, 1.6 mmol) in methanol (1 mL) was added p-toluenesulfonic acid (5 mg) at 0 °C. The temperature was allowed to rise to room temperature, the reaction mixture was stirred for 12 h, and potassium carbonate (0.5 g) was added. Chromatography on silica gel with ether as eluent gave the desired diol (0.31 g, 97%): colorless oil; IR (neat) 3320, 2930, 2860, 1450, 1360, 1060, 1035 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 3.63 (t, J = 6.1, 4 H), 2.03 (t, J = 7.5, 4 H), 1.60 (s, 6 H), 1.6–1.35 (m, 8 H); MS, m/e 200 (relative intensity) (M^+, 14), 192 (9), 149 (16), 55 (100); HRMS calcd for $C_{12}H_{24}O_2$ 200.1776, found 200.1780. Anal. Calcd for $C_{12}H_{24}O_2$: C, 71.95; H, 12.08. Found: C, 71.58, H, 11.88.

5,6-Dimethyl-5-decene-1,10-diol Dimesylate. To a solution of the enediol (0.20 g, 1.0 mmol) in freshly distilled methylene chloride (10 mL) at 0 °C were added under N_2 triethylamine (0.303 g, 3.0 mmol) and then methanesulfonyl chloride (0.286 g, 2.5 mmol) via syringe within 5 min. The mixture was stirred for 15 min at 0 °C and then worked up with cold water to give the crude dimesylate, pure by TLC and ^1H NMR (0.345 g, 96%): colorless oil; IR (neat) 3020, 2940, 2860, 1380, 1170, 990, 930 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 4.20 (t, J = 7, 4 H), 2.95 (s, 6 H), 2.00 (t, J = 7, 4 H), 1.60 (s, 6 H), 1.7–1.4 (m, 8 H).

1,10-Dibromo-5,6-dimethyl-5-decene (6). In a round-bottomed flask were placed the dimesylate (0.32 g, 0.90 mmol), lithium bromide (0.39 g, 4.5 mmol), and freshly distilled DMF (10 mL). The flask was continuously purged with a flow of N_2 and the mixture heated to 70 °C for 1 h. The resulting solution was worked up with water and the crude product chromatographed on silica with ether–petroleum ether (1:9) as eluent to give **6** (0.272 g, 93%): colorless liquid; IR (neat) 2960, 2945, 2860, 1455, 1445, 1250 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 3.40 (t, J = 6.8, 4 H), 2.01 (t, J = 7.5, 4 H), 1.82 (quin, J = 7, 4 H), 1.60 (s, 6 H), 1.47 (quin, J = 7, 4 H).

7,8-Dimethyltetradec-7-ene-1,13-diyne (7). In a round-bottomed flask were placed under N_2 lithium acetylide–ethylenediamine complex (0.276 g, 3 mmol) and freshly distilled Me_2SO (2 mL). To this slurry was added the dibromide **6** (0.245 g, 0.75 mmol) in Me_2SO (2 mL) at 10 °C within 5 min. The mixture was stirred under N_2 for 2.5 h at room temperature, then quenched with cold water, and worked up. The crude compound was chromatographed on silica gel with ether–petroleum ether (1:9) as eluent to give enediyne **7** (0.145 g, 95%): colorless oil; IR (neat) 3300, 2940, 2860, 2120, 1455, 1440 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 2.18 (td, J = 7, 2.7, 4 H), 2.00 (t, J = 7, 4 H), 1.92 (t, J = 2.7, 2 H), 1.60 (s, 6 H), 1.53–1.43 (m, 8 H); MS, m/e (relative intensity) 216 (M^+, 8), 201 (8.3), 164 (25), 145 (11), 84 (100); HRMS calcd for $C_{16}H_{24}$ 216.1879, found 216.1882.

(η^5-Cyclopentadienyl)(8a,9,10,10a-η^4-*endo,endo*-*cis*-1,2,3,4,4a,4b,5,6,7,8-decahydro-4a,4b-dimethylphenanthrene)cobalt (8). A degassed solution of **7** (38.9 mg, 0.18 mmol) in m-xylene (20 mL) was heated to 120 °C and irradiated (visible light, GE-ENH, 250 W) in the presence of $CpCo(CO)_2$ (36 mg, 0.20 mmol). After 1.5 h the solvent was removed in vacuo (0.05 torr) and the residue chromatographed on alumina (activity II) under N_2 with degassed hexane as eluent to give the complex **8** (37 mg, 60%): red orange oil; ^1H NMR (250 MHz, C_6D_6) δ 4.57 (s, 2 H), 4.50 (s, 5 H), 2.4–1.75 (m, 6 H), 1.65 (m, 4 H), 1.45–1.25 (m, 4 H), 1.21 (s, 6 H), 1.00 (m, 4 H); MS, m/e (relative intensity) 340 (M^+, 88), 325 (10), 310 (14), 272 (39), 216 (41), 201 (94), 187 (13), 173 (42), 159 (100); HRMS calcd for $C_{21}H_{29}Co$ 340.1601, found 340.1593.

***cis*-1,2,3,4,4a,4b,5,6,7,8-Decahydro-4a,4b-dimethylphenanthrene (9).** The cobalt complex **8** (36 mg, 0.11 mmol) was filtered through silica gel (10 g) with degassed hexane as eluent under N_2 to give the free ligand **9** (14 mg, 61%): viscous, colorless oil; IR (neat) 3030, 2920, 2860, 1650, 1600 cm^{-1}; ^1H NMR (250 MHz, C_6D_6) δ 5.55 (s, 2 H), 2.37 (ddd, J = 4.7, 13.1, 2 H), 2.18 (ddd, J = 1.5, 3, 13, 2 H), 1.82 (bd, J = 11, 2 H) 1.75–1.25 (m, 10 H), 1.02 (s, 6 H); MS, m/e (relative intensity) 216 (M^+, 60), 201 (84), 187 (27), 159 (100); HRMS calcd for $C_{16}H_{24}$ 216.1877, found 216.1874.

Acknowledgment. This work was supported by the NSF CHE-82-00049. M.M. was the recipient of a NATO Science Fellowship (1981–1982). K.P.C.V. was a Camille and Henry Dreyfus Teacher–Scholar (1978–1983).

Registry No. 1, 1720-37-2; **2**, 92937-82-1; **3**, 92937-83-2; **4**, 92937-84-3; **4**·2THP, 92937-89-8; **5**, 92937-85-4; **6**, 92937-86-5; **7**, 92937-87-6; **8**, 92957-39-6; **9**, 92937-88-7; Cp_2ZrCl_2, 1291-32-3; $(CH_3)_3Al$, 75-24-1; $(Ph_3P)_4Pd$, 14221-01-3; CH_3MgI, 917-64-6; LiC≡CH, 1111-64-4; $CpCo(CO)_2$, 12078-25-0; 4-bromobutanol tetrahydropyranyl ether, 31608-22-7; dihydropyran, 110-87-2; 5,6-dimethyl-5-decene-1,10-diol, 92937-90-1; 5,6-dimethyl-5-decene-1,10-diol dimesylate, 92937-91-2; ethylenediamine, 107-15-3.

Reaction of Bis(2,4-dinitrophenyl) Phosphate with Hydrophobic Ammonium Ions

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Received April 23, 1984

The overall hydrolysis of diaryl phosphates is a two-step reaction,[1] and reaction of bis(2,4-dinitrophenyl) phosphate (bis-2,4-DNPP) in aqueous alkali is shown in Scheme I.[2]

The first step involves nucleophilic attack upon the phosphoryl group and the second step is spontaneous heterolysis, which is written as generating short-lived metaphosphate ion.[1-5]

Both steps of the reaction are speeded by cationic micelles which assist attack of OH^- by bringing the reactants into close proximity and assist spontaneous dephosphorylation by exerting a medium effect.[6]

Functional micelles which contain nucleophilic groups are effective reagents in deacylation, dephosphorylation, and nucleophilic addition and substitution.[7-10] Nonmi-

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