Heteroatom Chemistry
Volume 17, Number 5, 2006

Regioselective Formation of a 2,3-Oxaphosphabicyclo[2.2.2]octene 3-oxide in Baeyer–Villiger Type Oxidation; a Dual Pathway for Its Fragmentation

Stefan Jankowski,[1] János Kovács,[2] Krzysztof Huben,[1] Michał Błaszczyk,[3] Marek Główka,[3] and György Keglevich[2]

[1]*Institute of Organic Chemistry, Department of Chemistry, Technical University of Lodz, Zeromskiego 116, 90-924 Lodz, Poland*

[2]*Department of Organic Chemical Technology, Budapest University of Technology and Economics, 1521 Budapest, Hungary*

[3]*Institute of General and Ecological Chemistry, Department of Chemistry, Technical University of Lodz, Zeromskiego 116, 90-924 Lodz, Poland*

Received 17 November 2005; revised 16 December 2005

ABSTRACT: *The O-insertion reaction of a 7-phosphanorbornene (**3**) unsubstituted on the double-bond gave the corresponding 2,3-oxaphosphabicyclo-[2.2.2]octene oxide (**4a**) in a regioselective manner that was useful in the fragmentation-related phosphonylation of alcohols. Both the UV-light mediated and the thermoinduced phosponylation accomplished on the bridged P-heterocycle (**4a**) were found to be sensitive toward steric factors, suggesting that beside the well-known elimination–addition reaction path taking place via metaphosphonate (**11**), a competitive novel addition–elimination route involving an intermediate with a pentacoordinated P-atom (**12**) is also present. This was confirmed by the kinetic consideration of our experimental data.* © 2006 Wiley Periodicals, Inc. Heteroatom Chem 17:369–375, 2006; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20213

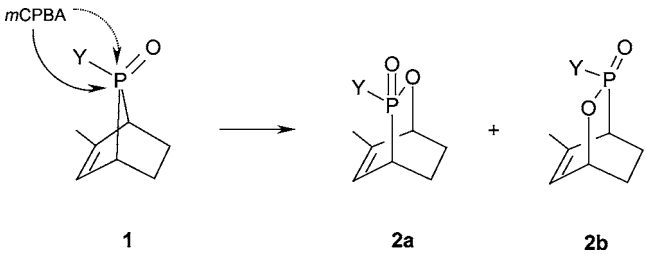
Correspondence to: Stefan Jankowski; e-mail: jankowsk@p.lodz.pl.

Contract grant sponsor: OTKA.
Contract grant number: T 042479.
Contract grant sponsor: Technical University of Lodz.
Contract grant number: D.S. I-18/10.

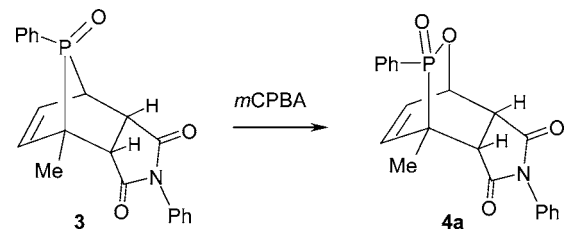
INTRODUCTION

The Baeyer–Villiger reaction has been applied for the oxidation of ketones with peroxides into esters or lactones for over a hundred years [1]. For acyclic substrates, a general preference for the migration of the most electron-rich alkyl group (more substituted carbon) was established from the literature examples. The steric effects can, however, compete with electronic effects in the oxidation of norbornanone derivatives [2–4].

The 7-phosphanorbornene oxides **1** can be easily transformed by *m*CPBA into oxaphosphabicyclo[2.2.2]octenes **2** [5]. The peroxyacid attacks the phosphorus atom from both sides affording the products as two regioisomers (Scheme 1). The ratio of regioisomers **2** depends on the P-substituent (Y = EtO [6], Et_2N [7], MesNH [8], and P-aryl [9]). Recently, the steric effect of the P-aryl substituents (Y = 2,4,6-tri-isopropylphenyl, mesityl or



SCHEME 1



SCHEME 2

4-methylphenyl) of 7-phosphanorbornene 7-oxides on reaction with *m*CPBA was studied in detail [9]. When a 7-phosphanorbornene labeled with O-18 in the P=O group was treated with *m*CPBA, the product of O-insertion possessed the heavy atom in the phosphoryl group [9]. The oxygen atom in the bridge came from the peracid, as was observed in tracer experiments involving the peracetic acid oxidation of ketones [10,11]. This observation is consistent with the involvement of the "Criegee" mechanism [12].

As a continuation of our studies on the Baeyer–Villiger oxidation of 7-phosphanorbornene 7-oxides, we decided to examine the effect of substitution of carbon atoms adjacent to the phosphorus atom. For this, 7-phosphanobornene model compound **3**, bearing one methyl group attached to the carbon atom adjacent to the phosphorus atom and synthesized from a 2-methyl-2,5-dihydro-1*H*-phosphole oxide, was chosen to be the starting material [13].

RESULTS AND DISCUSSION

O-Insertion into 7-Phosphanorbornene Framework

The O-insertion into 7-phosphanorbornene **3** by means of *m*CPBA was carried out according to a known procedure [6–9]. Two differences in comparison with the oxidation of P-phenyl 7-phosphanorbornene derivative with a methyl group attached to the double bond in position 3 were observed. The O-insertion reaction was about five times slower, and only a single product was detected by ^{31}P NMR. ^{13}C NMR spectrum of the product suggested structure **4a** revealing a ^{2}J(PC) coupling constant of 0.6 Hz on the skeletal methyl group of the product that is in agreement with literature example [14]. This coupling could also be a ^{3}J(PC) in the other isomer (**4b**) [15]. Single crystal X-ray analysis of the product confirmed, however, the structure represented by **4a** (Scheme 2, Fig. 1).

Theoretically, two possible intermediates of the oxidation of **3** are **5a** or **5b**, possessing one of the P—C bonds and the peroxy group in apical positions. The migration of the peroxy group into equatorial position (as in **6a** and **6b**) is followed by the insertion of the oxygen atom into the P—C bond that is in apical position (Scheme 3). Our observation that only product **4a** was formed is on the contrary to the rule of the preference for the higher bonding carbon migration. In the Baeyer–Villiger type oxidation of 7-phosphanorbornene **3** the steric effect dominates. In pentacovalent intermediate **6a**, the more crowded carbon atom prefers the equatorial position and is intact against the attack of the peroxy group. Faster migration of the less substituted carbon atom during the

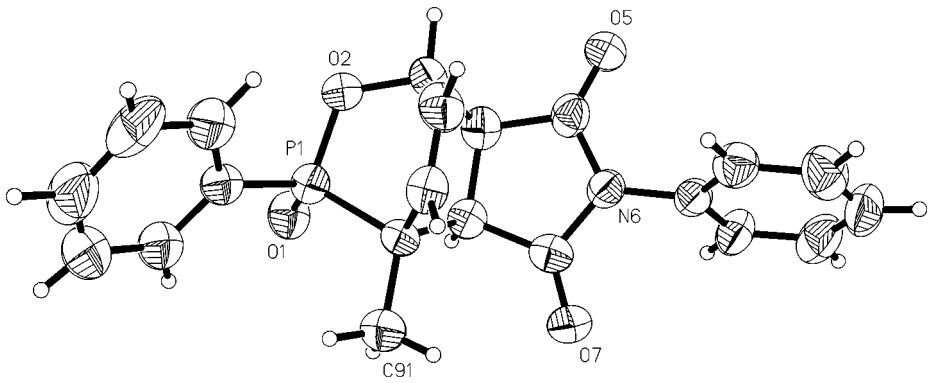
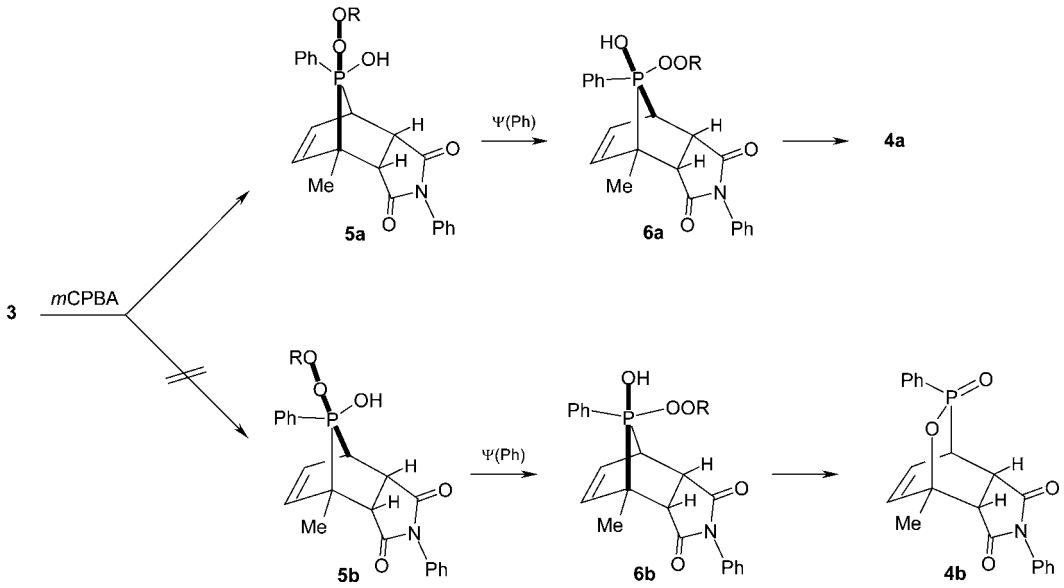


FIGURE 1 The view of molecular structure of **4a** in solid state.

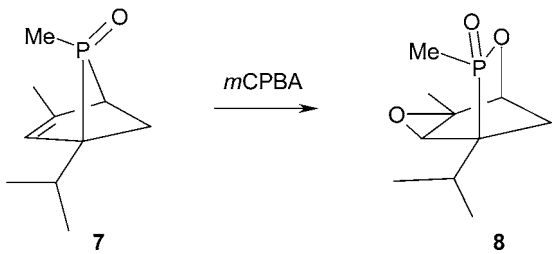


SCHEME 3

Baeyer–Villiger oxidation of *trans*-2,2,3-trimethyl-1-phenylphosphetane 1-oxide and 5-isopropyl-2,6-dimethyl-6-phosphabicyclo[3.1.1]hept-2-ene 6-oxide (**7**) was observed previously by Quin et al. [16]. The result of the second example is shown in Scheme 4. Compound **7** is similar to **3**, but as the epoxidation occurred faster than the oxygen insertion, the product of the reaction with *m*CPBA was identified as **8**.

Fragmentation Reactions of 2,3-Oxaphosphabicyclo[2.2.2]octane 2-oxide **4a** *in the Presence of Alcohols*

The fragmentation of 2,3-oxaphosphabicyclo[2.2.2]-octane **4a** was accomplished by thermolysis in toluene at 110°C, in the presence of methanol or *tert*-butyl alcohol, or by irradiation at 254 nm in acetonitrile or in 1,2-dichloroethane in the presence of an alcohol to give phosphonic acid ester **9**. Esterification of **9** with diazomethane gave phosphonate **10** (Scheme 5).

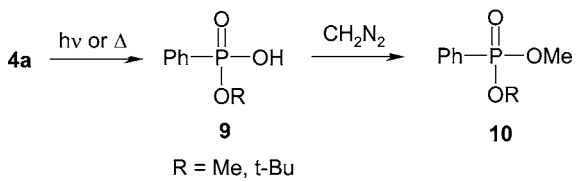


SCHEME 4 [15]

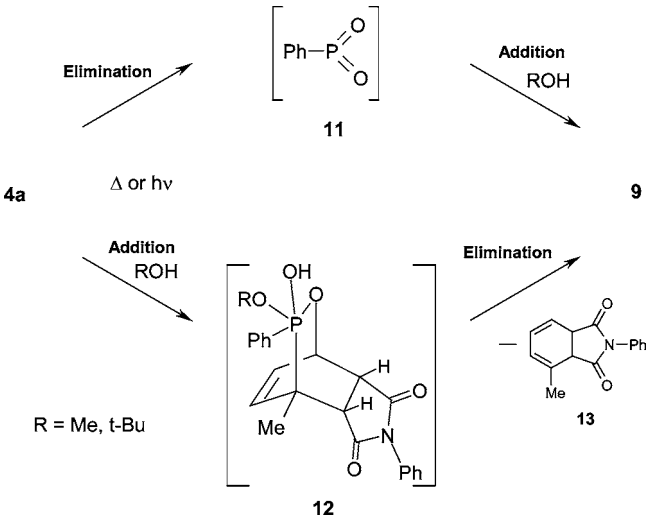
The necessary time for the consumption of the oxaphosphabicyclooctene oxide (**4a**) increased when methanol was replaced by *tert*-butanol regardless of the method (thermal or photochemical) applied. Thermolysis of **4a** was about twice as fast as that of *P*-phenyl-2,3-oxaphosphabicyclo[2.2.2]octene derivative with a methyl group attached to the double bond in position 3 [9].

Regarding the photolysis, the fragmentation-related phosphorylation was ca. 3.5 times slower using *tert*-butanol instead of methanol. Photolysis of substrate **4a** in the presence of an equimolar mixture of methanol and *tert*-butanol led to a mixture containing the corresponding phosphonic acid esters (**9**, R = Me and *t*-Bu) in a ratio of 77/23. Using *tert*-butanol in a twofold quantity, the ratio of the methyl and *tert*-butyl esters was 67/33. It can be seen that the steric hindrance brings about a significant discrimination between the alcohols.

Thermolysis of cycloadduct **4a** in the presence of a 1:2 mixture of methanol and *tert*-butanol afforded a mixture containing the corresponding phosphonic acid esters (**9**, R = Me and *t*-Bu) in a nearly equal



SCHEME 5



SCHEME 6

ratio. This means that the phosphorylation of *tert*-butanol is significantly slower than that of methanol.

Mechanism of the Fragmentation-Related Phosphorylations of Alcohols by 2,3-Oxaphosphabicyclo[2.2.2]octene 2-oxide (**4a**)

It is known that the fragmentation-related phosphorylation of a nucleophile by an oxaphosphabicyclo[2.2.2]octene oxide takes place according to an elimination–addition (EA) mechanism, which is not sensitive to the structure and concentration of the alcohol (Scheme 6). Our experience that the reaction is significantly slower using *tert*-butanol instead of methanol may, however, suggest the involvement of the addition–elimination (EA) bimolecular reaction path via an intermediate with a pentacoordinate phosphorus atom formed by the addition of the alcohol on the P=O group (Scheme 6). The sensitivity of the reaction rate to the steric effect of an alcohol may be in accord with the novel AE mechanism. As a matter of fact, it seems to be probable that the fragmentation-related phosphorylation proceeds according to concurrent EA and AE reaction paths. This situation has recently been substantiated for the fragmentation-related phosphorylations of nucleophiles by 2-phosphabicyclo[2.2.2]octene 2-oxide derivatives, where the AE components have never been assumed before [9].

The effect of alcohol concentration on the yield of photolysis was examined in detail in small scale. Acetonitrile solutions of **4a** with different concentrations of alcohols were irradiated in a quartz NMR tube placed in Rayonet reactor for 40 min. The relative amount of phosphonic derivative **9**, R = Me

TABLE 1 Photolysis of **4a** in the Presence of Alcohol ROH in Acetonitrile (Irradiation Time 40 min)

	Yield[a] *of* **9** *(%)*	
Alcohol Concentration (M)	*R = Me*	*R = t-Bu*
0.68	71	
0.75		44
0.90	73	
1.13		61
1.50	76	74
2.25		74

[a]Established on the basis of relative ^{31}P NMR intensities of product **9** and substrate **4a**.

obtained in reaction with methanol was constant (ca. 73%) over the concentration range of 0.68–1.5 M. In the presence of a sterically hindered alcohol, such as *t*-BuOH, the product percentage was found to increase up till 74% in the cases, when the concentration of alcohol was 1.5 M or higher (Table 1). This experience is in accord with the involvement of two mechanisms (elimination–addition and addition–elimination). The proposed kinetic scheme is outlined in Scheme 7.

According to Scheme 7, light absorption by **4a** produces an excited molecule **4a***, which is fragmented to metaphosphonate **11** (k_1), deactivated (k_{-1}) or reacts with alcohol (k_3) to form pentacoordinated intermediate **12**. Application of the stationary steady-state approximation for **4a***, **11**, and **12** gives the rate of product formation as shown by Eq. (5):

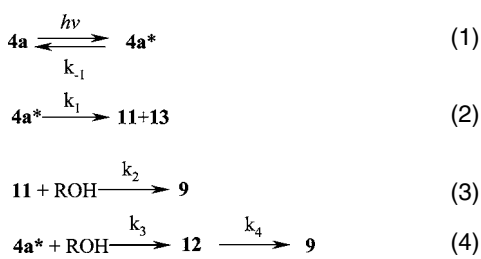
$$\mathrm{d}[\mathbf{9}]/\mathrm{d}t = I(k_1 + k_3[\mathrm{ROH}])/(k_{-1} + k_1 + k_3[\mathrm{ROH}]) \quad (5)$$

where I is the rate of light absorption.

If the alcohol concentration is low enough to allow for $k_{-1} + k_1 \gg k_3[\mathrm{ROH}]$, Eq. (5) is simplified to Eq. (6):

$$\mathrm{d}[\mathbf{9}]/\mathrm{d}t = I(k_1 + k_3[\mathrm{ROH}])/(k_{-1} + k_1) \quad (6)$$

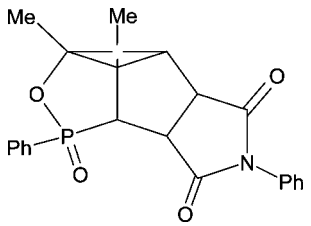
According to Eq. (6), the rate of photolysis depends on the rate constants of uni- and bimolecular processes, and increases with the alcohol concentration.



SCHEME 7

When the alcohol concentration is high enough that the ratio $(k_1 + k_3[\mathrm{ROH}])/(k_{-1} + k_1 + k_3[\mathrm{ROH}])$ becomes close to unity, Eq. (5) is simplified to $\mathrm{d}[\mathbf{9}]/\mathrm{d}t = I$. The rate of product formation becomes constant and is limited only by the rate of light absorption. The light absorption-controlled value is reached faster in the case of methanol than with tertiary butanol, as in the bimolecular reaction (4) one can expect that ${}^{\mathrm{MeOH}}k_3 > {}^{t\text{-BuOH}}k_3$. This means that indeed concurrent EA and AE mechanisms operate, and the model can be adequately described by Eq. (5).

Photolysis of **4a** in 1,2-dichloroethane was carried out in the Rayonet reactor. The photochemically mediated fragmentation afforded phosphonic acid esters **9** in lower yield (50–60%) than achieved in acetonitrile. Two additional signals in ^{31}P NMR spectra were observed in the ratio of about 1:3. The minor signal appeared at δ 78.9 ($C_2H_4Cl_2$) that was independent on the alcohol used. The other signals were recorded at δ 45.9 (methanol, $C_2H_4Cl_2$) and δ 38.5 (*tert*-butanol, $C_2H_4Cl_2$). The rearrangement of 2,3-oxaphosphabicyclo[2.2.2]octenes (1-adamantyl, neopentyl, ethyl as O-substituents, phenyl) accompanying usual metaphosphate extrusion was observed at -75°C and at room temperature with very low yield [17,18]. It was concluded that after the C—O bond cleavage, the oxygen atom can be transferred to the adjacent sp^2 carbon resulting in a cyclopropane ring. According to this mechanism **4a** may rearrange to **14** (Scheme 8), in which the cyclic phosphinate ring can be opened in the presence of an alcohol as it is in **15**. The downfield shift at δ_P 78.9 signal seems to be appropriate for structure **14** and the upfield signals at δ_P 45.9 (R = Me) or 38.5 (R = t-Bu) to **15**. The chemical shift of δ_P 65.6 measured in propionitrile at -75°C for compound **16** [17] supports our assignment in regard to structure **14**. The suggested rearrangement needs further examination.



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In conclusion, steric reasons allowed the regioselective synthesis of a new 2,3-oxaphosphabicyclo[2.2.2]octenes 3-oxide, a useful precursor in the phosphonylation of alcohols. An alternative mechanism of novel type was substantiated for the fragmentation-related phosphorylation no matter if it is thermo- or photoinduced. The involvement of the concurrent EA and AE mechanisms is supported by the experimental data.

EXPERIMENTAL

NMR spectra were recorded on a Bruker Avance DPX 250 spectrometer at 101.20 MHz (^{31}P) and 250.13 MHz (^{1}H), 62.86 MHz (^{13}C) in $CDCl_3$, using tetramethylsilane as internal and 85% H_3PO_4 as external standard. Solvents were dried as follows: CH_3CN and 1,2-dichloroethane by distillation from P_2O_5; toluene, by distillation over Na in the presence of benzophenone.

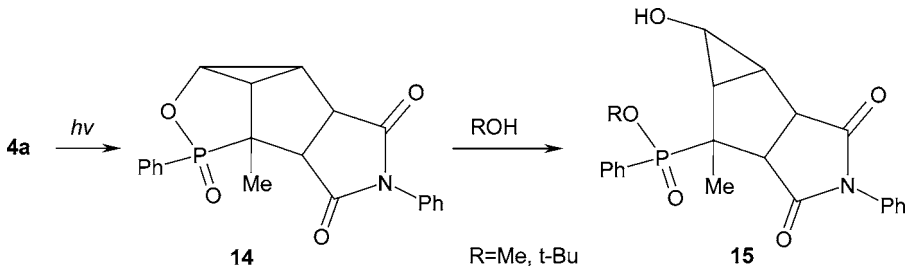
*7-Methyl-2,8-diphenyl-3a,4,7,7a-tetrahydro-1H-4,7-(epoxyphosphano)isoindole-1,3-dione 8-oxide (**4a**)*

2.5 g (purity 77%, 8.7 mmol) of *m*CPBA was added to the 20 cm^3 of dry $CHCl_3$ solution of 0.80 g (2.2 mmol) of 7-phosphanorbornene **3** [13]. The solution was stirred at room temperature and monitored by ^{31}P NMR. After the competition of the reaction, 2.5 g (43.1 mmol) of KF was added and the mixture was stirred at room temperature for 2 h. The suspension was filtered off (Celite 500), and the volatile components were removed. The crude product was purified by crystallization from AcOEt to give product **4a** in 0.56 g (67%).

Colorless solid, mp 182–184°C (ethyl acetate); IR, ν_{max} (CCl_4) 2928, 1708, 1392, 1232, 1200, 984, 784 cm^{-1}; ^{31}P NMR ($CDCl_3$) δ 37.5; ^{13}C NMR ($CDCl_3$) δ 14.4 ($J = 0.6$, $C_7 - Me$), 40.1 ($J = 6.3$, C_{7a}), 41.1 ($J = 67.9$, C_7), 47.6 ($J = 12.6$, C_{3a}), 71.8 ($J = 8.8$, C_4), 126.2 ($C_{2''}$),[a] 128.5 ($J = 13.2$, $C_{3'}$),[b] 128.8 ($J = 83.0$, $C_{1'}$), 129.0 ($C_{4'}$),129.2 ($C_{3''}$),[a] 131.2 ($C_{1''}$), 131.6 ($J = 9.4$, C_5), 133.2 ($C_{4''}$), 134.2 ($J = 9.4$, $C_{2'}$),[b] 138.6 ($J = 6.3$, C_6), 173.0 (C_3), 174.5 ($J = 15.1$, C_1); [a,b] may be reversed; ^{1}H NMR ($CDCl_3$) δ 1.72 (3H, d, $J = 17.0$ Hz, C_7CH_3), 3.78 (1H, t, $J = 8.2$ Hz, $C_{7a}H$), 4.23 (1H, dd, $J = 8.2$, 3.8 Hz, $C_{3a}H$), 5.60 (1H, m, C_4H), 6.17 (1H, t, $J = 8.2$ Hz, C_6H), 6.68 (1H, dt, $J = 8.2$, 5.5 Hz, C_5H), 7.19–7.16 (2H, m, H_{Ar}), 7.64–7.37 (6H, m, H_{Ar}), 7.76–7.68 (2H, m, H_{Ar}); IR, ν_{max} (CCl_4) 2928, 1708, 1392, 1232, 1200, 984, 784 cm^{-1}; MS, $(M+H)^+_{found} = 380.1038$, $C_{21}H_{19}NO_4P$ requires 380.1052.

*Photolysis of 2,3-Oxaphosphabicyclo[2.2.2]-octene Oxide (**4a**)*

1. The solution of 0.3 mmol of cycloadduct **4a** in the mixture of 40 cm^3 of acetonitrile and 4 cm^3 of alcohol was irradiated with a higher light intensity source 125 W mercury lamp in a photochemical



SCHEME 8

quartz reactor for 30 min at 26°C. Volatile components were removed in vacuo to give phosphonic acid-ester **9**.

9, R = Me: Yield, 92%; ^{31}P NMR ($CDCl_3$) δ 22.0 (δ_P^{lit}19.6 [19], 20.1 [20]); MS, $(M+H)^+ = 173$.

9, R = *t*-Bu: Yield, 83%; ^{31}P NMR ($CDCl_3$) δ 17.6 (δ_P^{lit} 11.6 crude sample in *tert*-BuOH [19]): MS, $(M+H)^+ = 215$.

The diethylether solution of phosphonic acid-ester **9** was reacted with an excess of diazomethane in diethylether at 26°C. The crude products obtained after evaporating the volatile components was purified by flash column chromatography on silica gel to furnish 4-methyl-1,3-dimethylene-2-phenyl-2,3,3a,7a-tetrahydro-1*H*-isoindole **13** (chloroform/hexane, 2:1) and phosphonic ester **10** (3% methanol in chloroform).

10, R = Me: Yield, 89%; ^{31}P NMR ($CDCl_3$) δ 22.3 (δ_P^{lit} 22.5 [21]); MS, $(M+H)^+ = 187$.

10, R = *t*-Bu: Yield, 74%; ^{31}P NMR ($CDCl_3$) δ 16.4; ^{13}C NMR ($CDCl_3$) δ 30.4 ($C(CH_3)$), 52.2 ($J = 5.7$, O—CH_3), 83.5 ($J = 8.1$, $C(CH_3)$), 128.4 ($J = 15.4$, $C_{3'}$), 131.6 ($J = 9.9$, $C_{2'}$), 132.2 ($J = 6.7$, $C_{4'}$); ^{1}H NMR ($CDCl_3$) δ 1.58 (s, 9H, $C(CH_3)_3$), 3.67 (d, $J = 11.0$, 3H, O—CH_3), 7.32–7.61 (m, 3H, Ar—H), 7.75–7.86 (m, 2H, Ar—H); *m/z* (FAB/NBA) 229 (3, MH^+), 173 (100), 133 (17), 107 (3).

13, 120.5–121.5°C (lit. [22] mp 120–123°C); ^{1}H NMR ($CDCl_3$) δ 2.11 (s, 3H, CH_3), 3.68 (d, 1H, $J = 12.5$ Hz, *H*-C_{3a}), 3.95 (dt, 1H, $J = 12.5$, 2.8 Hz, *H*-C_{7a}), 5.72 (dd, 1H, $J = 9.5$, 3.9 Hz, *H*-C_7), 5.80 (d, 1H, $J = 5.9$ Hz, *H*-C_5), 5.98 (ddd, 1H, $J = 9.5$, 5.9, 2.8 Hz, H-C_6), 7.23–7.53 (m, 5H, Ph).

2. In photolysis carried out in small scale, we applied the Rayonet reactor fitted with eight lamps, and the procedure was reported recently [9].

*Thermolysis of 2,3-Oxaphosphabicyclo[2.2.2]-octene Oxide (***4a***)*

A solution (1 cm^3) of **4a** (0.02 mmol) and MeOH or *tert*-BuOH (2 mmol) in dry toluene was sealed in an NMR tube (5 mm) under argon. The sample was placed in a thermostat set to 110°C, and the reaction was monitored by ^{31}P NMR until the signal of substrate (**4a**) diminished. The reaction time was 25 and 35 min, respectively. The volatile components were removed, and products were identified as phosphonic acid esters **9** (R = Me, *t*-Bu) by ^{31}P NMR.

9, R = Me, ^{31}P NMR ($CDCl_3$) δ 18.9 (δ_P^{lit} 19.6 [19]); MS, $(M+H)^+ = 173$.

9, R = *t*-Bu, ^{31}P NMR ($CDCl_3$) δ 12.3 (δ_P^{lit} 11.6 [19]); MS, $(M+H)^+ = 215$.

The dichloromethane solution of the phosphonic acid ester (**9**) was treated with an excess of diazomethane in diethylether at 0°C. After completion of the esterification, the solution was evaporated to dryness. The crude product so obtained was purified by flash column chromatography (as above) to afford phosphonic ester **10**.

10, R = Me: Yield, 90%; ^{31}P NMR ($CDCl_3$) δ 22.0 (δ_P^{lit} 22.5 [21]); MS, $(M+H)^+ = 187$.

10, R = *t*-Bu: Yield, 81%; ^{31}P NMR ($CDCl_3$) δ 16.1; MS, $(M+H)^+ = 229$.

Crystal Data of **4a**

$C_{21}H_{18}NO_4P$, $M = 379.33$, colorless prism, monoclinic, $P2_1/c$, $Z = 4$, $a = 11.150(2)$, $b = 6.876(1)$, $c = 25.877(3)$ Å, $\beta = 113.42(1)°$, $V = 1820.6(4)$ Å^3, $T = 291$ K, μ (Mo Kα) = 0.178 mm^{-1}, $R_1 = 0.0736$ for 1660 observed, $wR_2 = 0.139$ for all 3198 reflections collected, $S = 1.063$. Data collection: KM-4 diffractometer with CCD detector. Hydrogen atoms were refined in riding positions with free isotropic temperature parameters.

Crystallographic data for the structural analysis has been deposited with Cambridge Crystallographic Data Centre, CCDC No. 284210 for compound **4a**. Copies of this information can be obtained free of charge from: The Director CCDC, 12 Union Road, Cambridge, CB2 1EZ UK. Fax. (Int code) +44(1223)336-033 or Email: deposit@cccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk.

ACKNOWLEDGMENTS

Authors are thankful to Dr. Tomasz Nonas (TU of Lodz) for his assistance in the preliminary experiments.

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