β Elimination of a phosphonate group from an alkoxyl radical — Intramolecular acylation using acylphosphonate derivatives as carbonyl group acceptors[1]

Chang Ho Cho and Sunggak Kim

Abstract: The possibility of β elimination of a phosphonate group in radical reactions was studied. The facile β elimination of the phosphonate group from an alkoxyl radical was observed for the first time, whereas the β elimination of the phosphonate group from an aminyl and an alkyl radical did not occur. On the basis of our findings, the use of an acylphosphonate as a carbonyl group radical acceptor was investigated. Radical cyclization of the acylphosphonate in the presence of hexamethylditin in benzene at 300 nm for 2 h gave a cyclopentanone or a cyclohexanone derivative in good yield without the formation of a direct reduction product. The reaction can be carried out in the presence of a catalytic amount of hexamethylditin (0.2 equiv.) under similar conditions. In addition, an alkyl phosphonothiolformate group can act as an alkylthiocarbonyl group equivalent radical acceptor, providing ready access to a thiolactone synthesis.

Key words: radical, β elimination, acylation, cyclization, acylphosphonate.

Résumé : On a étudié la possibilité d'une élimination β d'un groupe phosphonate au cours de réactions radicalaires. On a observé pour la première fois une élimination β facile d'un groupe phosphonate à partir d'un radical alkoxyle alors qu'il ne se produit pas d'élimination β d'un groupe phosphonate à partir d'un radical aminyle ou d'un radical alkyle. Sur la base de ces observations, on a étudié l'utilisation d'un acylphosphonate comme groupe carbonyle accepteur de radical. La cyclisation radicalaire de l'acylphosphonate, en présence d'hexaméthyldiétain, dans le benzène, à 300 nm, pendant deux heures, conduit à la formation d'une cyclopentanone ou d'une cyclohexanone, avec un bon rendement, sans formation d'un produit de réduction directe. Il est possible d'effectuer la réaction dans des conditions semblables, en présence d'une quantité catalytique d'hexaméthyldiétain (0,2 équiv.). De plus, un groupe phosphonothiolformiate d'alkyle peut agir comme groupe alkylthiocarbonyle équivalent à l'accepteur radicalaire et fournir ainsi un accès facile à une synthèse de thiolactone.

Mots clés : radical, β élimination, acylation, cyclisation, acylphosphonate.

[Traduit par la Rédaction]

Introduction

The carbonyl group is one of the central functional groups in organic chemistry and can be readily prepared by treatment of carboxylic acid derivatives with organometallic compounds (1). However, the radical version of this conventional acylation reaction has not been successful to date because additions of alkyl radicals to C=O bonds are extremely difficult owing to the high π-bond strengths of C=O bonds (2, 3). Since β fragmentations of alkoxyl radicals are much faster than the additions of alkyl radicals to carbonyl groups, it is anticipated that carbonyl group derivatives cannot be used effectively as radical acceptors to achieve radical-mediated acylations. Thus, a limited number of carbonyl group radical acceptors has been utilized in intramolecular acylations and includes acyl sulfides, acyl selenides (4), and acylgermanes (5, 6). It is noteworthy that a mixed carboxylic phosphonic anhydride is a good radical acceptor in radical cyclizations but fails to function as the carbonyl group acceptor because the β fragmentation of the alkoxyl radical cleaves the carbon–carbon bond rather than the carbon–oxygen bond, as shown in Scheme 1 (7). In addition, several indirect approaches using a nitrile (8) and an oxime ether group have been developed (9, 10). In the case of intermolecular acylation, the use of carbonyl group derivatives as acceptors is uncommon and only several intermolecular radical carboxylations have been reported (11). We developed highly efficient indirect acylation approaches using sulfonyl oxime ethers (10). This indirect approach is at-

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Dedicated to Professor Howard Alper in recognition of his outstanding contribution to organic chemistry.

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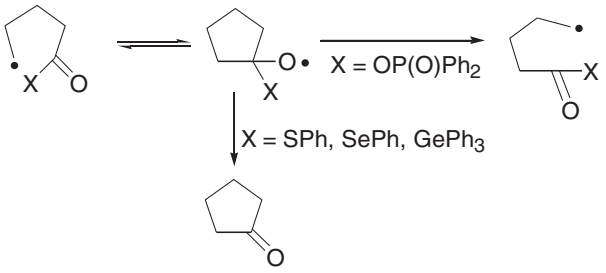
Scheme 1. Intramolecular acylation approach.



tractive since the carbonyl group can be easily generated by oxidative cleavage or by hydrolysis.

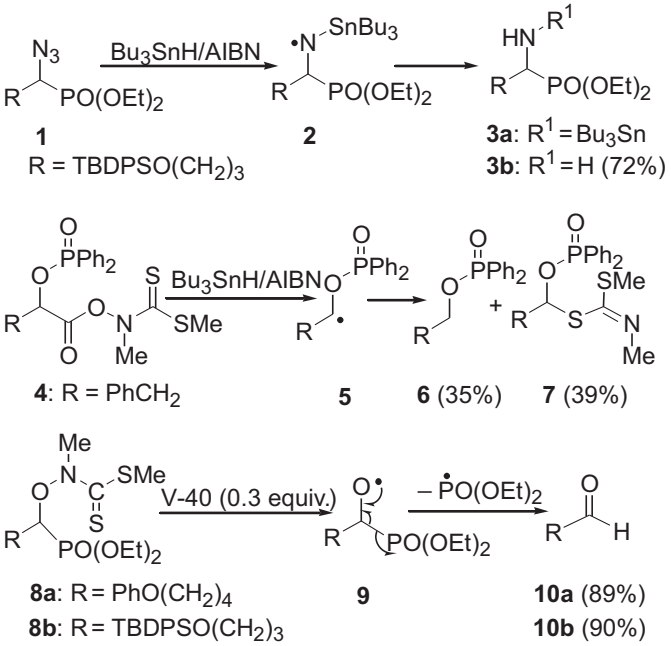
In connection with our continued interest on radical acylation, we have searched for other efficient carbonyl group radical acceptors and investigated the possibility of using a phosphonate as a leaving group in radical reactions (12).

Results and discussion

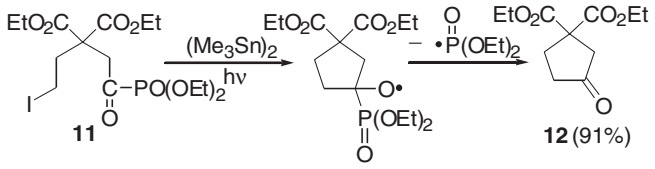
The β elimination of organophosphorous groups has not been well-studied and several reports have appeared only in recent years. The β elimination of a phosphinate group was observed during a rearomatization process (13). The β elimination of a diphenylphosphinoyl group has been known in radical allylation (14) and in tandem radical reactions involving cyclization and the subsequent β elimination of the diphenylphosphinoyl group (15).

The ease of β elimination generally depends on the nature of the π bond formed and the strength of the σ bond broken. Thus, it is expected that the β elimination would be easier when the π bond formed is stronger (16). Based on this rationale, we initially studied the β elimination of a diethyl phosphonate group from an aminyl radical (Scheme 2). When azidophosphonate **1** was treated with Bu_3SnH–AIBN in refluxing benzene for 2 h (17), **3b** was isolated in 72% yield, clearly indicating that the β elimination of the phosphonate group from the aminyl radical in radical intermediate **2** did not occur, probably because of the relatively weak π-bond strength of C=N bonds. Thus, our attention was given next to the formation of a carbonyl bond as a result of the β elimination of the phosphonate group, and we studied the possibility of β elimination of the diethyl phosphonate group from the alkyl radical using thiohydroxamate ester **4** (18). Radical reaction of **4** with Bu_3SnH–AIBN in refluxing benzene for 4 h afforded a mixture of **6** and **7** roughly in an equal ratio without the β elimination of the phosphonate group. Apparently, the phosphorous–oxygen bond in radical intermediate **5** is strong and did not undergo cleavage. Thus, **5** reacted with Bu_3SnH and the starting thiohydroxamate ester **4** to afford a mixture of **6** and **7**, respectively. Finally, the β elimination of a phosphonate group from the alkoxyl radical was examined. As shown in Scheme 2, when **8a** was treated with V-40 (1,1′-azobis(cyclohexane-1-carbonitrile)) as initiator in chlorobenzene for 3 h (19), aldehyde **10a** was isolated in 89% yield. A similar result was obtained with **8b**. Evidently, the β elimination is energetically favorable because of the formation of a strong carbonyl bond along with cleavage of a relatively weak carbon–phosphorous bond.

Scheme 2. β Elimination of a diethyl phosphonate group.



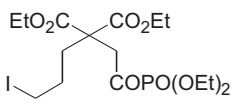
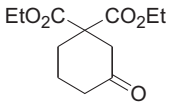
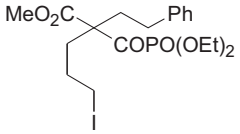
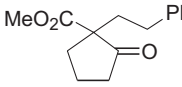
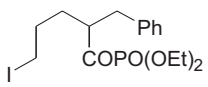
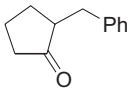
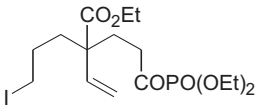
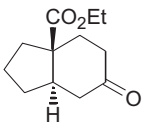
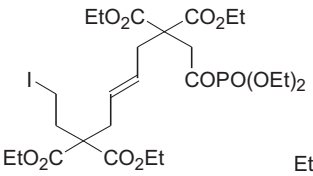
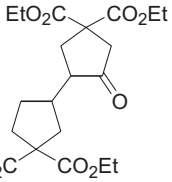
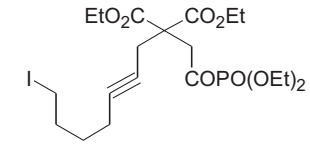
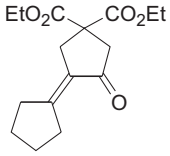
Scheme 3. Radical cyclization of an acylphosphonate.



Based on our findings, we studied the feasibility of using an acylphosphonate as a carbonyl group radical acceptor in intramolecular radical acylations (Scheme 3) (12). Acyl and bis(acyl)phosphine oxides have been employed as photochemical sources of acyl radicals for use as initiators in polymerization reactions (20). Also, acylphosphonates were used as acylating agents of alcohols, amines, and enolates (21). Acyldiethylphosphonates were conveniently prepared by treatment of acid chlorides with triethyl phosphite in dichloromethane at room temperature and were quite stable to silica gel column chromatographic purification (22). Radical cyclization of acylphosphonate **11** in the presence of hexamethylditin at 300 nm (1.1 equiv.) for 2 h in benzene gave cyclopentanone **12** in 91% yield without the formation of the direct reduction product. The reaction can be carried out in the presence of a catalytic amount of hexamethylditin (0.2 equiv.) to afford **12** in 88% yield under similar conditions because the initially generated phosphonate radical reacts with an alkyl iodide to generate an alkyl radical. Thus, the remaining reactions were carried out in benzene using 0.2 equiv. of hexamethylditin at 300 nm for 3 to 4 h.

Table 1 summarizes the experimental results and illustrates the efficiency of the acylphosphonates as the carbonyl group radical acceptor. The reaction was normally clean, yielding the corresponding cyclopentanones and cyclohexanones in high yield. For most of the cases observed in this study, there was no indication of the presence of the direct reduction product and other side products. Tandem radical

Table 1. Radical cyclization of acylphosphonates.[a]

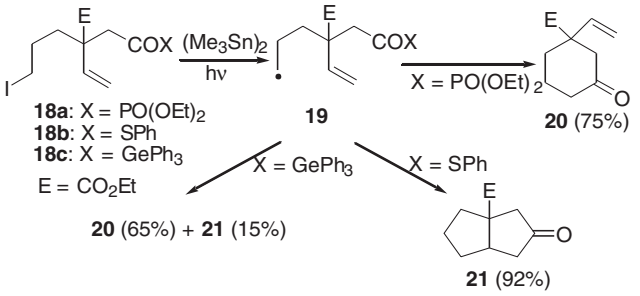
Entry	Substrate	Product	Yield (%)[b]
1			71 (82)[c]
2			61
3			72
4			69
5			79[d]
6			77

[a]The reaction was carried out with 0.2 equiv. of $(Me_3Sn)_2$ in benzene at 300 nm for 2 h.
[b]The yield was not optimized.
[c]$(Me_3Sn)_2$ (1.1 equiv.).
[d]Stereochemistry of starting material is *E* only. Diastereomeric mixture (1.2:1) of the product.

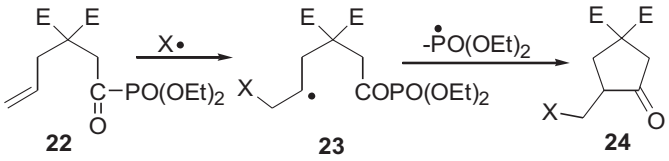
cyclizations worked equally well. Not only alkyl radicals but also alkenyl radicals reacted with acylphosphonates to give cyclopentanone derivatives (Table 1, entries 4–6).

It is noteworthy that reaction of **13a** under similar conditions gave cyclohexanone **16** as a sole product, whereas **13b**, using a thioester as acceptor, gave a mixture of **16** and **17b** in 46% and 33% yield, respectively (4) (eq. [1]). Apparently, the latter is formed as a result of the formation of stable radical intermediate **15b** owing to the facile β fragmentation of the alkoxyl radical **14b**. The result obtained here clearly indicates that the β elimination of the phosphonate group in **9** is very fast and highly efficient. Even more exciting results were obtained with **18** (Scheme 4). When **18a** was treated with hexamethylditin (0.2 equiv.) at 300 nm for 2 h, gratifyingly, **20** was obtained exclusively, whereas **21** was reported to be a sole product from the radical reaction of acyl sulfide **18b** (4). Apparently, 6-exo ring closure to the acylphosphonate group is much faster than 5-exo ring closure to the C=C bond, whereas 5-exo ring closure to the C=C bond is much faster than 6-exo ring closure to the thioester group. To strengthen the efficiency of the acylphosphonate, a comparative experiment was also carried out with acylgermane **18c**. Irradiation of a benzene solution of **18c** in the presence of hexamethylditin (0.2 equiv.) at 300 nm for 3 h afforded a 65:15 mixture of **20** and **21**, indicating higher reactivity of the acylphosphonate relative to the acylgermane as the carbonyl group radical acceptor. Since an approximate rate constant for 5-exo cyclization of a primary alkyl radical to an acylgermane is known to be 7×10^6 s^{-1} (5*c*), the approximate rate constant for the same cyclization to the acylphosphonate would be 3×10^7 s^{-1}. Apparently, the acylphosphonate is regarded as the most reactive among previously reported carbonyl group acceptors.

Scheme 4. Comparative studies of an acyl sulfide, an acylgermane, and an acylphosphonate.

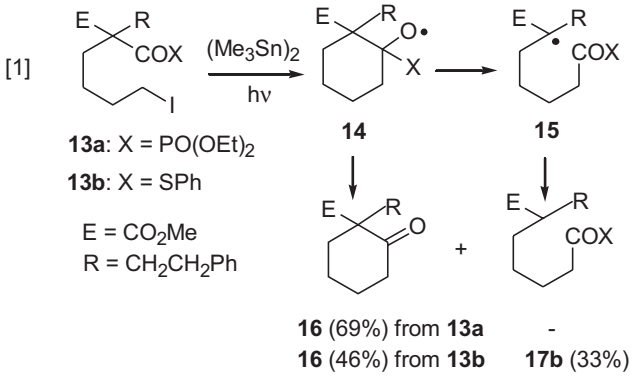


Scheme 5. Radical addition and cyclization of an acylphosphonate.



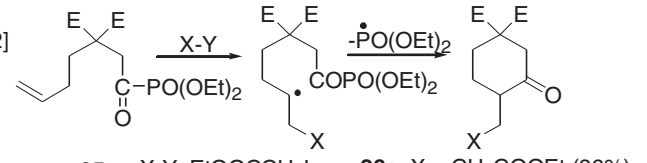
Based on the highly efficient and fast addition of the alkyl radical onto the acylphosphonate, we next studied sequential radical reactions involving intermolecular radical addition to the alkenyl and the alkynyl bond and subsequent cyclization (Scheme 5). This approach provides a ready access to β-functionalized cyclopentanones and cyclohexanones (23). Addition of a phenylsulfanyl radical and a phenylsulfonyl radical to an alkenyl group in **22** was followed by cyclization to afford cyclopentanone **24** along with the generation of the diethyl phosphonate radical for the chain propagation. Similarly, various electrophilic alkyl radicals from activated olefins bearing α-electron-withdrawing groups reacted smoothly with **22** to yield **24** in high yields. Appar-

[1]



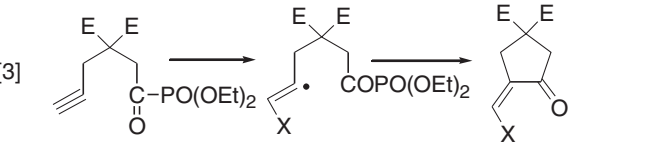
ently, the fast addition of the alkyl radical to the acylphosphonate obviates the problem of the quenching of radical intermediate **23** by the alkyl iodide prior to the cyclization. The experimental results are summarized in Table 2. The present approach can be applied to the formation of cyclohexanone derivatives (eq. [2]). Treatment of **25** with ethyl iodoacetate and hexamethylditin (0.2 equiv.) in benzene at 300 nm for 5 h afforded **26a** in 86% yield. A similar result was obtained with iodomethyl phenyl sulfone under the same conditions. Furthermore, the present approach can be applied to alkynyl-acylphosphonate **27**. When **27** was reacted with thiophenol and phenylsulfonyl bromide in the presence of AIBN in refluxing benzene for 3 h, the desired products **28a** and **28b** were obtained in 86% and 90% yield, respectively (eq. [3]).

[2]



25 X-Y: $EtOOCCH_2I$, $PhSO_2CH_2I$ **26a**: X = CH_2COOEt (86%) **26b**: X = CH_2SO_2Ph (85%)

[3]

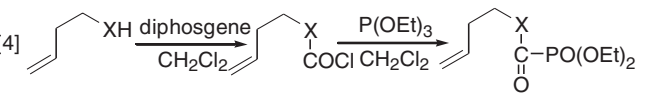


27 PhSH/AIBN **28a**: X = SPh: *E*/*Z* = 1:12, 86%

$PhSO_2Br$/AIBN **28b**: X = SO_2Ph: *E*/*Z* = 1:32, 90%

We next studied several interesting variations using a phosphonoformate and a phosphonothiolformate as radical acceptors to explore whether the present acylation could be further extended to the synthesis of lactone **31a** and thiolactone **31b**, respectively, via intermediate **30** (Scheme 6). Diethylphosphonoformate **29a** was prepared by the treatment of chloroformate **33a** with triethyl phosphite in dichloromethane at room temperature for 2 h (eq. [4]). **29a** was purified by passing it through a short column of silica gel. Similarly, diethylphosphonothiolformate **29b** was prepared from **32b** by a two-step procedure (24). Treatment of **32b** with diphosgene afforded **33b**, which was further treated with triethyl phosphite to give alkyl phosphonothiolformate **29b** in 75% yield.

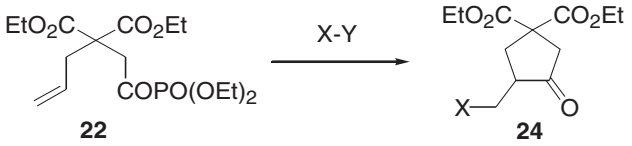
[4]



32a: X = O **32b**: X = S **33** **29a** (83%) **29b** (75%)

Reaction of **29a** with phenylsulfonyl bromide in the presence of AIBN (0.2 equiv.) in benzene at 80 °C for 6 h did not afford lactone **35** but gave addition product **36a** in 84% yield (eq. [5]). When the reaction was carried out with ethyl iodoacetate and hexamethylditin at 300 nm, a similar result

Table 2. Radical addition and cyclization of acylphosphonates.

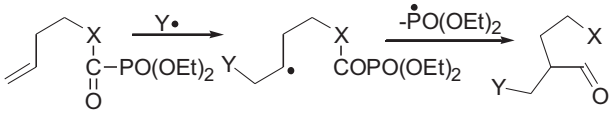


22 **24**

X-Y	Condition[a]	Time (h)	Yield (%)
PhS-H	A	10	89
$(TMS)_3Si$-H	A	2	75
$PhSO_2$-Br	A	2	90
C_6F_{13}-I	B	5	85
$EtOOCCH_2$-I	B	5	78
$EtOOCCH_2$-SC(=S)OEt	B	5	75
$PhSO_2CH_2$-I	B	5	72
$NCCH_2$-I	B	5	73

[a]Method A: AIBN, C_6H_6, reflux; Method B: 0.2 equiv. of $(Me_3Sn)_2$, C_6H_6, *hν* = 300 nm.

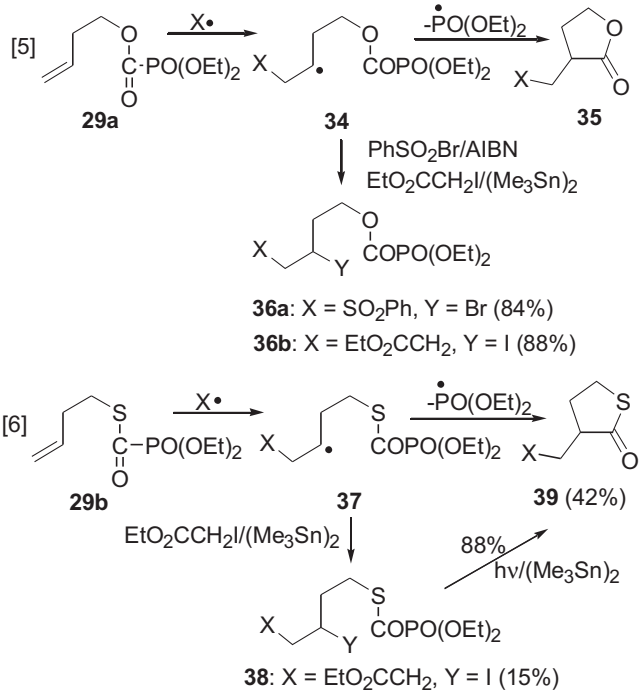
Scheme 6. Radical addition and cyclization of phosphonoformate and phosphonothiolformate.



29a: X = O **29b**: X = S **30** **31**

was obtained, yielding **36b** in 88% yield. It is evident that intermediate radical **34** underwent bromine and iodine atom transfer rather than cyclization. To enforce the cyclization of **36a**, the solution of **36a** was irradiated at 300 nm in the presence of hexamethylditin, but lactone **35** was not formed. The failure of the cyclization may be due to the unfavorable *E*-conformation of **34** and (or) the low reactivity of the alkoxycarbonyl group as a radical acceptor. Since the differences in energy are generally smaller for the *E* and *Z* conformations in thiol esters than for the corresponding carboxylic esters (25), radical cyclization of **29b** was studied. Irradiation of a benzene solution of **29b**, ethyl iodoacetate, and hexamethylditin (0.5 equiv.) at 300 nm for 3 h afforded a mixture of addition product **38** and thiolactone **39** in 15% and 42% yield, respectively (eq. [6]). Furthermore, reaction of **38** in the presence of hexamethylditin at 300 nm for 3 h gave thiolactone **39** in 88% yield. From the results obtained here, two features are noteworthy. The addition of an alkyl radical onto the phosphonothiolformate group occurred smoothly, and the diethyl phosphonate group was eliminated preferentially relative to the alkylthio group.

In conclusion, we have shown the facile β elimination of a phosphonate group from an alkoxyl radical and its application to intramolecular acylation approach, in which we demonstrated that the acylphosphonate was a highly efficient carbonyl group radical acceptor and more reactive than the previously known acyl sulfide and acylgermane. In addition, an alkyl phosphonothiolformate group can act as an



alkylthiocarbonyl group equivalent radical acceptor, providing a ready access to a thiolactone synthesis.[3]

Acknowledgments

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