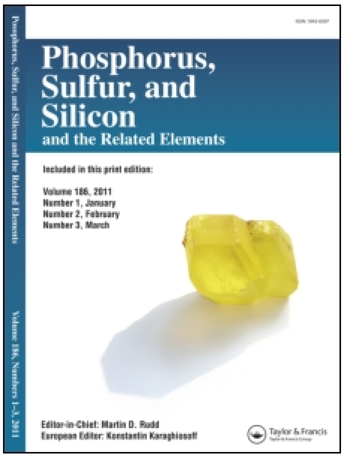

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# REACTION OF ISOQUINOLINETRIONE AND 1,3(2H,4H)-ISOQUINOLINEDION-4-YLIDENE DERIVATIVES WITH TRIALKYL PHOSPHITES

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# REACTION OF ISOQUINOLINETRIONE AND 1,3(2H,4H)-ISOQUINOLINEDION-4-YLIDENE DERIVATIVES WITH TRIALKYL PHOSPHITES

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Alkoxy-substituted 2-aklyl-1,3,4(2H)-isoquinolinetriones (**7**) react with trialkyl phosphites to give the corresponding 1,3(2H,4H)-isoquinolinedion-4-ylidene derivatives **8** and trialkyl phosphates. The reaction of **8** with trialkyl phosphites in the presence of water affords reduction products shown to be 1,3(2H,4H)-isoquinolinedion-4-yl derivatives **9** by the spectral data and X-ray crystallography.

## INTRODUCTION

Although synthesis of alkenes by reductive dimerization of $\alpha,\beta$-unsaturated monocarbonyl compounds, e.g. phthalic anhydride[1] and substituted maleic anhydrides,[2] using trialkyl phosphites has been known, the reductive co-condensation of vicinal triketones using phosphorus triester has not yet been reported. It is known that the reaction of vicinal triketones with trialkyl phosphites is not generally predictable. For example, trialkyl phosphites reacted with diphenylpropanetrione (**1**)[3] to yield 1 : 1 adducts formulated as cyclic unsaturated pentaoxyphosphoranes (**4**) and with quinisatin (**2**)[4] to give the corresponding phosphate derivatives (**5**). But anhydrous alloxan (**3**) reacted with trimethyl phosphite to afford the phosphorylated compound **6** like an *o*-quinone or an $\alpha,\beta$-diketone.[5]

Disubstituted acetylenes bearing groups such as methoxycarbonyl, benzoyl and phenyl can be reduced by triphenylphosphine and water.[6] Dimethyl acetylenedicarboxylate, for example, on treatment with triphenylphosphine and deuterium oxide produces dimethyl [$\alpha\beta$-$^2H_2$] fumarate in 70% yield. Although evidence for trialkyl phosphite as reducing reagent has been obtained,[7] to our knowledge, there have no reports on the direct formation of alkanes from alkenes by trialkyl phosphites and water.

We describe herein the deoxygenative dimerization of isoquinolinetriones (**7**) with trialkyl phosphites and the reduction of 1,3(2H,4H)-isoquinolinedion-4-ylidene

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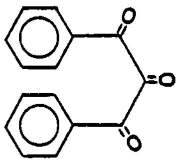

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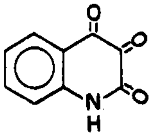

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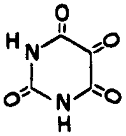

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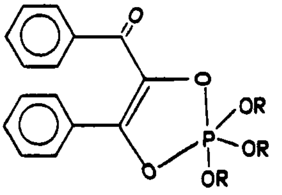

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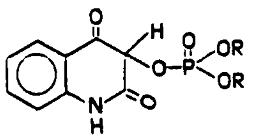

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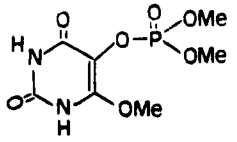


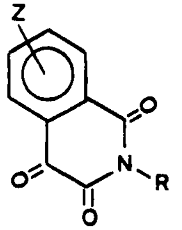
6

derivatives (**8**) with trialkyl phosphites in the presence of water to afford 1,3(2H,4H)-isoquinolinedion-4-yl derivatives (**9**).

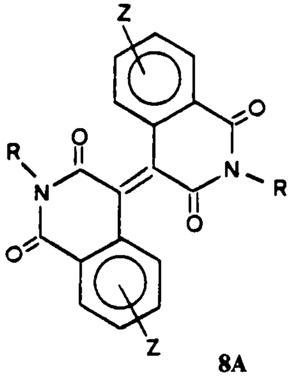
## RESULTS AND DISCUSSION

Trialkyl phosphites, namely, trimethyl-, triisopropyl phosphites react with **7** to give the corresponding dimeric compounds **8** and trialkyl phosphates.

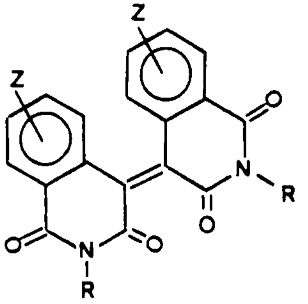
Structural elucidation for compound **8c**, taken as an example, was based upon the following evidence: (i) Elemental analysis and molecular weight determination agreed with the formula $C_{26}H_{26}N_2O_{10}$, (ii) The IR spectrum of **8c** in KBr showed bands at 1718 and 1672 $cm^{-1}$ (C=O, amide), (iii) The UV spectrum of **8c** showed the UV absorption at the longer wavelength compared with that of **7c**, (iv) The $^1H$ NMR spectrum of **8c** disclosed the presence of 6 *N*-methyl protons as a singlet, 18 methoxy protons (6H + 12H) as two singlets and 2 aromatic protons as a singlet. Signals of methyl protons in the $^1H$ NMR spectrum of **8c** are shifted upfield compared with those of **7c** and the signal of *O*-methyl protons at C(5,5′) of **8c** is shifted more upfield than those of other methyl protons of **8c** in comparison with those of methyl protons of **7c**. According to models of the structure **8A** and **8B**, the structure **8A** has a greater preference than structure **8B**. The van der Waals repulsions between two of the carbonyl oxygens at C(3) and C(3′) and, in particular, repulsions between two of the methoxy groups at C(5) and C(5′) in the structure **8B** are stronger than the repulsions between carbonyl oxygen at (3) and methoxy group at C(5′) and repulsions between carbonyl oxygen (3′) and methoxy group at C(5) in the structure **8A**. Thus structure **8B** would be extremely sterically unfavourable.



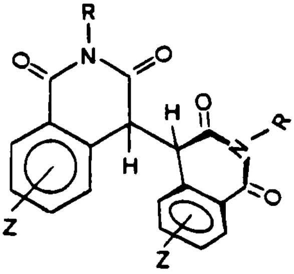
7 a: Z=5,7-OMe, R=Me
b: Z=5,7-OMe, R=c-$C_6H_{11}$
c: Z=5,6,7-OMe, R=Me
d: Z=5,6,7-OMe, R=c-$C_6H_{11}$



**8A**



**8B**



**9**

In the course of the studies on the chemistry of trialkyl phosphites, we found that compounds **8** were reduced readily by treatment with trialkyl phosphites and water to afford the corresponding reduction compounds **9** and trialkyl phosphates. The similarity of formation of the reduction compounds **9** suggests that they are of analogous constitution. These spectroscopic data led to structure **9c**, taken as an example, being tentatively assigned to 1,3(2H,4H)-isoquinolinedion-4-yl derivative, assuming the hydrogenation pattern of **8c**. To confirm this structure an X-ray analysis was performed. The compound **9c** crystallizes in the triclinic space group p$\bar{1}$ with cell dimensions a = 9.298(6), b = 10.567(9), c = 15.053(7) Å, $\alpha = 72.47(9)$, $\beta = 73.71(6)$, $\gamma = 64.92(3)^0$, Z = 2 and $D_c = 1.126$ g $cm^{-3}$. The X-ray intensities were measured with Mo—$K_\alpha$ radiation on a full automatic four-circle diffractometer. The crystal structure **9c** was solved by a combination of Patterson and Fourier techniques and the atomic parameters were adjusted by least-squares calculations Final R is 0.055 for 2977 reflexions. A view of the solid-state conformation of **9c** and atom numbering scheme are shown in the Figure 1. Selected bond distances and bond angles for **9c** are given in Table I.

We assume that the deoxygenative dimerisations of isoquinolinetriones by trialkyl phosphites follow the pathway adumbrated[1,2,9] for phthalic anhydride, so that

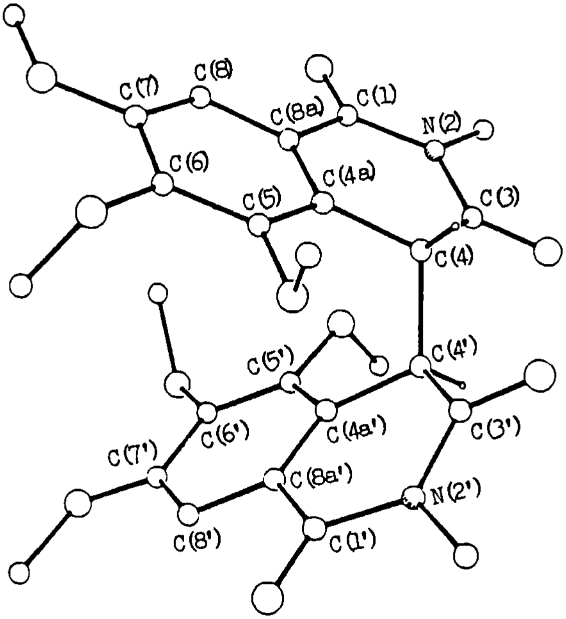


FIGURE 1 Structure of **9c**.

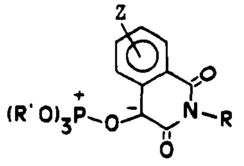
## TABLE I

Selected bond distances (Å) and bond angles (deg) for **9c**

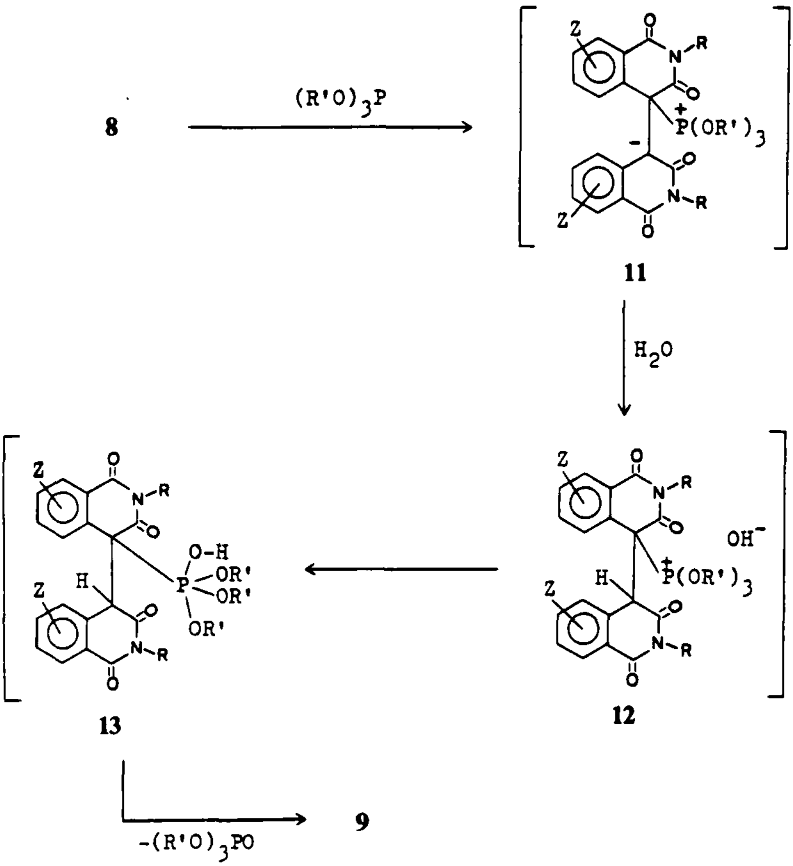
| | | | |
|---|---|---|---|
| | (i) Bond distances | | |
| C(3)—C(4) | 1.515(4) | C(4)—C(4a) | 1.502(5) |
| C(4)—C(4′) | 1.570(3) | C(3)—O(3) | 1.219(5) |
| N(2)—C(3) | 1.379(3) | C(1)—N(2) | 1.388(5) |
| C(4a)—C(8a) | 1.387(3) | C(6)—O(6) | 1.370(4) |
| C(5)—C(6) | 1.387(5) | O(6)—C(6b) | 1.401(5) |
| C(5)—O(5) | 1.374(3) | C(4a)—C(5) | 1.408(4) |
| O(5)—C(5b) | 1.437(3) | C(3′)—C(4′) | 1.519(3) |
| C(4′)—C(4a′) | 1.517(4) | C(3′)—O(3′) | 1.217(4) |
| N(2′)—C(2′) | 1.473(3) | C(1′)—N(2′) | 1.393(4) |
| C(6′)—O(6′) | 1.369(3) | C(5′)—C(6′) | 1.393(4) |
| O(6′)—C(6b′) | 1.423(4) | C(5′)—O(5′) | 1.381(4) |
| C(4a′)—C(5′) | 1.398(3) | O(5′)—C(5b′) | 1.438(4) |
| | (ii) Bond angles | | |
| C(3)—C(4)—C(4a) | 114.8(2) | C(3)—C(4)—C(4′) | 109.2(2) |
| C(4a)—C(4)—C(4′) | 114.5(2) | C(4)—C(3)—O(3) | 119.6(2) |
| C(4)—C(3)—N(2) | 120.2(3) | O(3)—C(3)—N(2) | 120.2(3) |
| C(3)—N(2)—C(2) | 117.0(3) | C(3)—N(2)—C(1) | 124.3(3) |
| C(2)—N(2)—C(1) | 118.0(2) | N(2)—C(1)—O(1) | 119.8(3) |
| N(2)—C(1)—C(8a) | 117.7(2) | O(1)—C(1)—C(8a) | 122.5(3) |
| C(1)—C(8a)—C(8) | 117.1(2) | C(1)—C(8a)—C(4a) | 121.5(3) |
| C(4)—C(4a)—C(8a) | 121.1(2) | C(4)—C(4a)—C(5) | 120.9(2) |
| C(4)—C(4′)—C(3′) | 110.3(2) | C(4)—C(4′)—C(4a′) | 114.6(2) |
| C(3′)—C(4′)—C(4a′) | 113.8(2) | C(4′)—C(3′)—O(3′) | 120.0(3) |
| C(4′)—C(3′)—N(2′) | 119.8(2) | O(3′)—C(3′)—N(2′) | 120.0(2) |
| C(3′)—N(2′)—C(2′) | 117.7(2) | C(3′)—N(2′)—C(1′) | 124.2(2) |
| C(2′)—N(2′)—C(1′) | 117.4(2) | N(2′)—C(1′)—O(1′) | 120.1(2) |
| N(2′)—C(1′)—C(8a′) | 116.9(2) | O(1′)—C(1′)—C(8a′) | 123.0(3) |

1,3(2H,4H)-isoquinolinedion-4-ylidene derivatives (**8**) entails generation of the phosphorane (**10**), which then undergoes a Wittig type reaction with a further isoquinolinetrione molecule.

A possible pathway of the reaction of **8** with trialkyl phosphites and water is shown in Scheme 1.



**10**



Scheme 1

## EXPERIMENTAL

*General.* Melting points were determined with an Electrothermal Melting Point Apparatus and were uncorrected. The $^1H$, $^{31}P$ NMR spectra were obtained on a Varian FT-80A spectrometer. Standards were tetramethylsilane(TMS) for $^1H$ NMR and 85% $H_3PO_4$ for $^{31}P$ NMR. $^{31}P$ chemical shifts downfield from

85% $H_3PO_4$ are designated positive. The IR spectra were determined on a Perkin-Elmer Model 283 B grating spectrophotometer. UV spectra were obtained using a Cary Model 17 spectrophotometer and $CHCl_3$ as solvent. Mass spectra were obtained using a Varian Mat 212 or HP 5985 mass spectrometer. Microanalyses were performed by the Analytical Laboratory, KAIST, South Korea. Triaklyl phosphites were purified by treatment with Na followed by fractional distillation. Anhydrous acetonitrile and chloroform were obtained by the usual procedures. Other chemicals were of reagent grade.

*Materials.* Alkoxy-substituted 2-alkyl-1,3,4(2H)-isoquinolinetriones were synthesized according to the literature.[8]

*General procedure for the reaction of* **7** *with trialkyl phosphites.* To a solution of **7** (7 mmol) in 30 ml of dry chloroform was added dropwise excess trialkyl phosphite (14 mmol) with stirring under nitrogen. As the reaction was elevated to the reflux temperature, the change of the color of the solution to yellow-green was observed. After being stirred for a few days, the solvent was removed under reduced pressure. The liquid that distilled under vacuum was analyzed by $^1H$, $^{31}P$ NMR and IR spectrometry, and was found to contain trialkyl phosphite and trialkyl phosphate. The residue of this distillation showed no absorption in its $^{31}P$ NMR spectrum. The residue was extracted with dichloromethane, filtered, evaporated to dryness. Preparative thin layer chromatography on silica gel, eluting with 10 : 1 chloroform/acetonitrile mixture, gave dimeric compound. The yields of the products are summarized in Table II. The physical and spectral data and analyses of these compounds are as follows:

**8a**: mp > 300°C; $^1H$ NMR($CDCl_3$, $\delta$ ppm) 3.34(6 H, s, 2,2′-$NCH_3$), 3.77(6 H, s, 5,5-$OCH_3$), 3.88(6 H, s, 7,7′-$OCH_3$), 6.55(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 6,6′-H), 7.20(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 8,8′-H); IR(KBr disc, $cm^{-1}$) 1720 and 1671(C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 417($\varepsilon$ 11983); MS(m/e) $M^+$, 466(46.4), 435(100.0), 234(46.4), 85(68.6), 83(92.5). Anal. Calcd. for $C_{24}H_{22}N_2O_8$: C, 61.8; H, 4.7; N, 6.0. Found: C, 61.6; H, 4.7; N, 5.8.

**8b**: mp 245–246°C; $^1H$ NMR($CDCl_3$, $\delta$ ppm) 1.26–2.38(22 H, m, 2,2-*N*-cyclohexyl), 3.13(6 H, s, 5,5′-$OCH_3$), 3.88(6 H, s, 7,7′-$OCH_3$), 6.35(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 6.6′-H), 7.21(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 8,8′-H); IR(KBr, $cm^{-1}$) 1718 and 1672(C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 403($\varepsilon$15050); MS(m/e) $M^+$, 602(100.0), 571(65.4), 489(81.5), 272(34.6), 84(69.1). Anal. Calcd. for $C_{34}H_{38}N_2O_8$: C, 67.8; H, 6.3; N, 4.7. Found: C, 67.9; H, 6.6; N, 4.5.

**8c**: mp > 300°C; $^1H$ NMR($CDCl_3$, $\delta$ ppm) 3.38(6 H, s, 2,2′-$NCH_3$), 3.69(6 H, s, 5,5′-$OCH_3$), 3.96(12 H, s, 6,6′, 7,7′-$OCH_3$), 7.43(2 H, s, 8,8′-H); IR(KBr, $cm^{-1}$) 1718 and 1672 (C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 395 ($\varepsilon$ 14611); MS(m/e) $M^+$, 526(14.5), 495(100.0), 464(26.6), 84(13.9), 57(15.6). Anal. Calcd. for $C_{26}H_{26}N_2O_{10}$: C, 59.3; H, 4.9; N, 5.3. Found: C, 59.0; H, 5.1; N, 5.3.

**8d**: mp > 300°C $^1H$ NMR($CDCl_3$, $\delta$ ppm) 1.26–1.89(22 H, m, 2,2′-*N*-cyclohexyl), 3.68(6 H, s, 5,5′-$OCH_3$), 3.93(6 H, s, 7,7′-$OCH_3$), 3.96(6 H, s, 6,6′-$OCH_3$), 7.38(2 H, s, 8,8′-H); IR(KBr, $cm^{-1}$) 1720 and 1677(C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 397($\varepsilon$ 15392); MS(m/e) $M^+$, 662(6.8), 549(71.8), 332(100.0), 71(54.2), 57(62.8). Anal. Calcd. for $C_{36}H_{42}N_2O_{10}$: C, 65.3; H, 6.3; N, 4.2. Found: C, 65.3; H, 6.1; N, 4.0.

General procedure for the reduction of **8** with trialkyl phosphites and water. A large excess of trialkyl phosphite was slowly added to a solution of **8** in chloroform containing aqueous ethanol. After the

TABLE II

Reaction of isoquinolinetriones with trialkyl phosphites[a]

| Isoquinolinetrione **7** | Dimer product **8** | (R′O)$_3$ R′ | Temp.(°C) | Time (hr) | Yield[b] (%) |
|---|---|---|---|---|---|
| a: Z = 5,7-OMe, R = Me | a | Me | reflux | 96 | 58 |
| | | *i*-Pr | 40 | 80 | 60 |
| b: Z = 5,7-OMe, R = *c*-$C_6H_{11}$ | b | Me | reflux | 96 | 59 |
| | | *i*-Pr | 40 | 80 | 63 |
| c: Z = 5,6,7-OMe, R = Me | c | Me | reflux | 96 | 53 |
| | | *i*-Pr | 40 | 80 | 57 |
| d: Z = 5,6,7-OMe, R = *c*-$C_6H_{11}$ | d | Me | reflux | 96 | 50 |
| | | *i*-Pr | 40 | 80 | 55 |

[a] The molar ratio of $(R'O)_3P$ and the isoquinolinetrone is 7.0 : 1.0.
[b] Yield of chromatographed dimer product.

TABLE III

Reduction of **8** with trialkyl phosphite and water[a]

| **8** | Reduction product | $(R'O)_3P$ R' | Time(hr) at 100°C | Yield[b] (%) |
|---|---|---|---|---|
| a: Z = 5,5′,7,7′-OMe, R = Me | **9a** | Me | 108 | 47 |
| b: Z = 5,5′,7,7′-OMe, R = $c$-$C_6H_{11}$ | **9b** | *i*-Pr | 72 | 55 |
| | | Me | 90 | 51 |
| c: Z = 5,5′,6,6′,7,7′-OMe, R = Me | **9c** | *i*-Pr | 60 | 58 |
| | | Me | 78 | 54 |
| d: Z = 5,5′,6,6′,7,7′-OMe, R = $c$-$C_6C_{11}$ | **9d** | *i*-Pr | 60 | 61 |
| | | Me | 72 | 63 |
| | | *i*-Pr | 48 | 67 |

[a] The molar ratio of $(R'O)_3P$, **8** and water is 7.0 : 1.0 : 1.0.

[b] Yield of chromatographed reduction product.

reaction mixture was stirred at 30°C for 4 hr and at 100°C for a few days, removal of the solvent and distillation of the residue gave trialkyl phosphite and trialkyl phosphate. The residue of this distillation was extracted with chloroform, filtered, evaporated to dryness. Purification (preparative thin layer chromatography) afforded reduction product. The yields of the products are summarized in Table III. The physical and spectral data and analyses of these compounds are as follows:

**9a**: mp 239–240°C; $^1$H NMR($CDCl_3$, $\delta$ ppm) 3.40(6 H, s, 2,2′-$NCH_3$), 3.53(6 H, s, 5,5′-$OCH_3$), 3.79(6 H, s, 7.7′-$OCH_3$), 4.82(2 H, s, 4,4′-H), 6.32(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 6,6′-H), 7.18(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 8,8′-H); IR(KBr, cm$^{-1}$) 1720 and 1675(C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 327($\varepsilon$ 2496); MS(m/e) $M^+$, 468(5.7), 234(100.0), Anal. Calcd. for $C_{24}H_{24}N_2O_8$: C, 61.5; H, 5.1; N, 6.0. Found: C, 61.8; H, 4.8; N, 5.7.

**9b**: mp 236–237°C; $^1$H NMR($CDCl_3$, $\delta$ ppm) 1.25–1.75(22 H, m, 2,2′-*N*-cyclohexyl), 3.52(6 H, s, 5,5′-$OCH_3$), 3.80(6 H, s, 7,7′-$OCH_3$), 4.68(2 H, s, 4,4′-H), 6.29(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 6,6′-H), 7.18(2 H, d, $J_{6-8} = J_{6'-8'} = 2.6$ Hz, 8,8′-H); IR(KBr, cm$^{-1}$) 1720 and 1671 (C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 332($\varepsilon$ 6040); MS(m/e) $M^+$, 604(1.2), 302(100.0). Anal. Calcd. for $C_{34}H_{40}N_2O_8$: C, 67.6; H, 6.6; N, 4.6. Found: C, 67.7; H, 6.4; N, 4.4.

**9c**: mp 221–222°C; $^1$H NMR($CDCl_3$, $\delta$ ppm) 3.35(6 H, s, 2,2′-$NCH_3$), 3.59(6 H, s, 5,5′-$OCH_3$), 3.66(6 H, s, 7,7′-$OCH_3$), 3.81(6 H, s, 6,6′-$OCH_3$), 4.87(2 H, s, 4,4′-H), 7.32(2 H, s, 8,8′-H); IR(KBr, cm$^{-1}$) 1718 and 1670 (C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 315 ($\varepsilon$ 5984); MS(m/e) $M^+$, 528(8.9), 264(100.0). Anal. Calcd. for $C_{26}H_{28}N_2O_{10}$: C, 59.1; H, 5.3; N, 5.3. Found: C, 59.0; H, 5.1; N, 5.5.

**9d**: mp 152–153°C; $^1$H NMR($CDCl_3$, $\delta$ ppm) 0.90–2.33(22 H, m, 2,2′-*N*-cyclohexyl), 3.59 and 3.72(3 H, s, 5 or 5′-$OCH_3$), 3.75(3 H, s, 7 or 7′-$OCH_3$), 3.83(6 H, s, 7 or 7′-$OCH_3$ and 6 or 6′-$OCH_3$), 3.89(3 H, s, 6 or 6′-$OCH_3$), 4.73 and 4.86(1 H, s, 4 or 4′-H), 7.35(2 H, s, 8,8′-H); IR(KBr, cm$^{-1}$) 1718 and 1640 (C=O); UV($CHCl_3$) $\lambda_{max}$(the longest wavelength, nm) = 317 ($\varepsilon$ 3054); MS(m/e) $M^+$, 664(0.9), 332(100.0). Anal. Calcd. for $C_{36}H_{44}N_2O_{10}$: C, 65.1; H, 6.6; N, 4.2. Found: C, 64.9; H, 6.5; N, 4.2

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