

# Synthesis and characterization of chalcone-substituted phosphazenes

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**Abstract:** A series of mono[(*E*)-1-(4-alkyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one]cyclotriphosphazenes and hexakis[(*E*)-1-(4-alkyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one]cyclotriphosphazenes have been synthesized. A convenient synthetic method was performed from the reaction of hexachlorocyclotriphosphazenes with 1 and 6 equiv. of (*E*)-1-(4-alkyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one (**2a–2c**) to afford (**3a–3c**) in 17%–19% and (**4a–4c**) in 70%–82%, respectively. The compounds differ in the length of alkyl groups,  $C_nH_{2n+1}$ , where  $n = 10, 12$ , and  $14$ .

**Key words:** hexachlorocyclotriphosphazenes, chalcones, alkyloxy, condensation.

**Résumé :** On a réalisé la synthèse d'une série de mono[(*E*)-1-(4-alkyloxyphényl)-3-(4-hydroxyphényl)prop-2-én-1-one]cyclotriphosphazènes et de hexakis[(*E*)-1-(4-alkyloxyphényl)-3-(4-hydroxyphényl)prop-2-én-1-one]cyclotriphosphazènes. On a exécuté une méthode de synthèse appropriée par réaction d'hexachlorocyclotriphosphazènes avec 1 et 6 equiv. de (*E*)-1-(4-alkyloxyphényl)-3-(4-hydroxyphényl)prop-2-én-1-one (**2a–2c**) qui ont conduit respectivement aux produits (**3a–3c**) avec des rendements allant de 17 à 19 % et aux produits (**4a–4c**) avec des rendements allant de 70 à 82 %. Les composés diffèrent par la longueur des groupes alkyles,  $C_nH_{2n+1}$ , dans lesquels  $n = 10, 12$  et  $14$ .

**Mots-clés :** hexachlorocyclotriphosphazènes, chalcones, alkyloxy, condensation.

## Introduction

Phosphazenes are compounds containing a framework of alternating phosphorus and nitrogen atoms, either in cyclic or linear form.<sup>1</sup> Linear, cyclic, and poly phosphazenes have been widely investigated. These compounds are reported to possess interesting biomedical properties<sup>2</sup> and promising application as effective flame retardants for fiber materials.<sup>3</sup> Nucleophilic substitution reactions on hexachlorocyclotriphosphazenes have been widely reported.<sup>4–6</sup> The synthesis of cyclotriphosphazenes, bearing 4-oxychalcones<sup>6</sup> as side groups, has been studied for photosensitive phosphazenes that could undergo photo-cross-linking reaction under UV irradiation.

In photochemistry, chalcone derivatives were reported to possess outstanding nonlinear optic property for optical communications and optical electronics,<sup>7</sup> liquid crystal displays,<sup>8,9</sup> and alignment film.<sup>10</sup> Chalcones were also reported to promote excellent blue-light transmittance and good crystallability,<sup>11,12</sup> high photosensitivity, and thermal stability for various crystalline electro-optical devices.

Recently, we reported a very convenient method for the preparation of trimeric aryloxyphosphazenes directly from  $[N_3P_3Cl_6]$  and (*E*)-3-(4-(alkyloxy)phenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one using  $K_2CO_3$  in acetone.<sup>13</sup> This prompted us to try the reaction of cyclotriphosphazenes with other para-substituted hydroxy chalcones. We herein describe the synthesis of cyclotriphosphazenes incorporated with hydroxylated chalcones (*E*)-1-[4-(alkyloxy)phenyl]-3-[4-hydroxyphenyl] prop-2-en-1-one

(**2a–2c**), which could be used as model reactions for various crystalline electro-optical devices.

## Results and discussion

The series of chalcone derivatives (*E*)-1-[4-(alkyloxy)phenyl]-3-[4-hydroxyphenyl] prop-2-en-1-one (**2a–2c**) was prepared via Claisen–Schmidt condensation of **1a–1c** and 4-hydroxybenzaldehyde by the route depicted in Scheme 1.

The structural assignments of compounds **2a–2c** were based on the analytical and spectral data. The IR spectra of the hydroxylated chalcones **2a–2c** showed the presence of bands at  $2921$ – $2852\text{ cm}^{-1}$ , which were attributed to the introduction of the long alkyl chain via etherification of 4-hydroxyacetophenone. The presence of a new  $C=O$  stretching frequency at  $1651\text{ cm}^{-1}$  substantiated the formation of the title compound. The chemical structures of **2a–2c** were found to be consistent with  $^1H$  NMR and  $^{13}C$  NMR spectroscopic data and showed the peaks corresponding to the structures. In  $^1H$  NMR spectra, the coupling constant,  $J_{ab} = 15.0$ – $16.0\text{ Hz}$ , indicated all chalcones obtained were in trans-configuration.

The synthetic route for the preparation of mono- $(N_3P_3Cl_5[OC_6H_4CH=CHC(O)C_6H_4OC_nH_{2n+1}])$  (**3a–3c**) and hexa-substituted cyclotriphosphazenes  $(N_3P_3[OC_6H_4CH=CHC(O)C_6H_4OC_nH_{2n+1}]_6)$  (**4a–4c**) is illustrated in Scheme 2.

Mono-substituted cyclotriphosphazenes **3a–3c** were obtained from the reaction of hexachlorocyclotriphosphazenes with 1 equiv. of chalcone derivatives **2a–2c** in the presence of  $K_2CO_3$  in acetone. The higher polarity of the

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