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A mixed substituent substituent polyphosphazenes with the structure $[NPOCH_2CF_3)_{64\%}\{O(CH_2CH_2O)_3\text{-}C_6H_4\text{-}CH{=}CH\text{-}C_6H_4\text{-}NO_2\}_{36\%}]_n$ has been synthesized and the second-order nonlinear properties have been investigated. A second-harmonic coefficent of d_{33} = 5.5 pm/V was obtained. Reprints (10)

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A Second-Order Nonlinear Optical Poly(organophosphazene)

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Abstract: A mixed substituent polyphosphazene with the structure $[NP(OCH_2CF_3)_{64\%}\{O(CH_2CH_2O)_3\text{-}C_6H_4\text{-}CH{=}CH\text{-}C_6H_4\text{-}NO_2\}_{36\%}]_n$ has been synthesized and the second-order nonlinear optical properties have been investigated. A second-harmonic coefficient of d_{33} = 5.5 pm/V was obtained.

The development of polymeric nonlinear optical (NLO) materials is currently an area of intense investigation.[1] Polymeric systems which show second harmonic generation (SHG) have conjugated aromatic molecules with electron-donor and acceptor moieties in a noncentrosymmetric array. These nonlinear optical molecules can be doped into a glassy polymer matrix[2] or can be covalently attached to a polymer backbone[3]. Here we report the synthesis and second order nonlinear optical response of polymer **1**, a polyphosphazene in which a nitrostilbene unit is covalently linked to the polymer chain through a tri(ethylene oxide) spacer group. Phosphazene macromolecules offer a potential advantage in that the macroscopic properties of the polymer can be tailored by the incorporation of specific substituent groups.[4,5] Polymer **1** is therefore a prototype which offers many opportunities for further tailoring of the molecular structure to generate an optimum combination of nonlinear optical and physical properties.

Polymer 1 near here.

Our initial work involved the synthesis of a side chain for the polyphosphazene substrate which has molecular characteristics that are required for a nonlinear optical response. As outlined in Scheme I, 4-hydroxybenzaldehyde was allowed to react with 2-[2-(2-chloroethoxy)ethoxy]ethanol in basic ethanol containing potassium iodide for 15 h at reflux to yield 4-[2-(2-(2-hydroxyethoxy)ethoxy)ethoxy]benzaldehyde, **2**. Compound **2** was then allowed to react with diethyl(4-nitrobenzyl)phosphonate and potassium tert-butoxide in ethylene glycol dimethyl ether for 15 h at room temperature to give the desired trans-4-[2-(2-(2-hydroxyethoxy)ethoxy)ethoxy]-4'-nitrostilbene, **3**. Compound **3** was purified by column chromatography and was recrystallized from n-hexane/methylene chloride.[6]

Scheme I near here.

Polymer **1** was synthesized by the procedure described in Scheme II. Poly(dichlorophosphazene) was prepared by the thermal ring-opening polymerization of the cyclic trimer $(NPCl_2)_3$.[4] In the first step in the synthesis of **1**, sodium trifluoroethoxide was added to poly(dichlorophosphazene) to replace approximately 50% of the P-Cl bonds. In the second step, a stoichiometric deficiency of trans-$NaO(CH_2CH_2O)_3$-C_6H_4-CH=CH-C_6H_4-NO_2 was allowed to react with the partially substituted polymer. In the final step, an excess of sodium trifluoroethoxide was added to replace the remaining P-Cl bonds in order to obtain a fully derivatized, hydrolytically stable polymer.[7] Polymer **1** was isolated by precipitation from tetrahydrofuran into water and was purified by dialysis against methanol/water (1:1 v/v) for seven days. The polymer is a yellow elastomeric material that is soluble in common organic solvents such as tetrahydrofuran (THF) and methyl ethyl ketone (MEK). Characterization was achieved by ^{31}P and ^{1}H NMR spectroscopy,[8] infrared and UV/visible spectroscopy, elemental microanalysis, gel permeation chromatography and differential scanning calorimetry.[9] ^{1}H NMR analysis of polymer **1** indicates a 36% incorporation of the nitrostilbene side chain.

Scheme II near here.

Films of polyphosphazene **1** were cast onto indium-tin oxide coated glass from a concentrated solution of MEK. The solution was first filtered to remove particulate impurities and the films were dried in vacuum to remove all of the solvent. The NLO properties of the films were subsequently investigated using second-harmonic generation. A Q-switched Nd:YAG laser (λ = 1.064 μm) with a pulse width of 8 ns and a pulse energy of 10 mJ was used as the source of the

fundamental, and a reference sample of Y-cut quartz (d_{11} = 0.46 pm/V) was used for calibration of the frequency-doubled signal. From measurements of the refractive index at both the fundamental and second-harmonic wavelengths, the coherence length of **1** was calculated to be 2.8 μm. The thin-film thicknesses used were always less than this coherence length, being typically ca. 0.5 μm.

Alignment of the NLO side groups in the layers was achieved by single-point corona poling, with the point source held at +10 kV, at a distance of 1.5 cm from the surface. Poling voltages greater than 10 kV sometimes resulted in damage to the film, manifested as a slight cloudiness. Note, however, that this voltage was still below the saturation point of the signal. The variation of the signal with poling voltages will be discussed more fully in a later publication. Due to the low glass transition temperature of **1** (T_g = 25°C), the poling was carried out at room temperature while the SHG measurements were being made. Upon removal of the voltage, the second-harmonic signal decayed to zero within a few minutes.

The second-harmonic coeffieient of the polymer film, d_{33}, was obtained from a Maker fringe analysis of the data,[10] giving d_{33} = 5.5 pm/V. This value of d_{33} was obtained using the isotropic model for poled polymers, where d_{33}/d_{31} = 3.[2a] Singer et al[3d] have found this model to be appropriate for analysis of their side-chain polymers. Recently, however, Eich et al[3e] have observed deviations from this ratio, possible due to mesogenic interactions among the side groups. The applicability of the isotropic model to poly(organophosphazenes) is currently under investigation.

Given that the degree of alignment was not maximized in this experiment, and that we are using a less efficient donor moiety than other studies of functionalized polymers,[2c,3d] this is a very promising value of d_{33}. Work to attach more efficient donors and to increase the glass transition temperature of the polymer is in progress.

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6. For **3**: mp 66-67°C; λ_{max} (THF) = 378 nm; m/z calcd. 373, m/z found 373; IR

(KBr) 3500-3100 (br, ν(OH)), 1340 cm^{-1} (s, $\nu(NO_2)$); ^1H NMR ($CDCl_3$) δ 8.21 (2H, d, J=8.8 Hz, ArH), 7.61 (2H, d, J=8.7 Hz, ArH), 7.49 (2H, d, J=8.7 Hz, ArH), 7.23 (2H, d, J=16.3 Hz, CH), 7.01 (2H, d, J=16.3 Hz, CH), 6.95 (2H, d, J=8.8 Hz, ArH), 4.18 (2H, t, OCH_2), 3.90 (2H, t, OCH_2), 3.75 (6H, m, OCH_2), 3.65 (2H, t, OCH_2), 2.20 (1H, br s, OH); Yield = 40-55%; Anal. for $C_{20}H_{23}NO_6$, Calcd: C, 64.33; H, 6.21; N, 3.75. Found: C, 63.96; H, 6.20; N, 3.72.

7. This three step synthetic procedure was necessary since the direct addition of trans-$NaO(CH_2CH_2O)_3$-C_6H_4-CH=CH-C_6H_4-NO_2 to poly(dichlorophosphazene) resulted in the formation of an insoluble, incompletely substituted polymeric precipitate.

8. NMR spectra were recorded on a Bruker WP-360 spectrometer. Chemical shifts are relative to 85% H_3PO_4 (^{31}P) or tetramethylsilane (^1H).

9. For **1**: λ_{max} (THF) = 376 nm; ^{31}P NMR (THF/D_2O) δ -8.3; $M_n = 3.2 \times 10^5$, $M_w = 1.4 \times 10^6$, $M_w/M_n = 4$; IR (KBr) 1340 (s, $\nu(NO_2)$), 1280 cm^{-1} (s, ν(P=N)); ^1H NMR (d_6-acetone) δ 8.3-6.9 (m, ArH, CH), 4.5 (br s, OCH_2CF_3), 4.3-3.6 (m, OCH_2); T_g = 25°C; Anal. Found: C, 43.01; H, 4.66; N, 5.13; Cl, <0.11. Analysis is consistent with a 30% incorporation of **3**.

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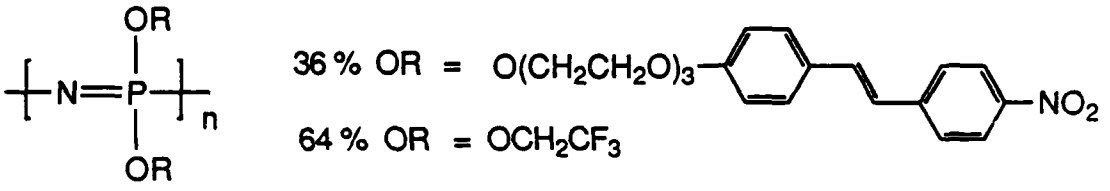

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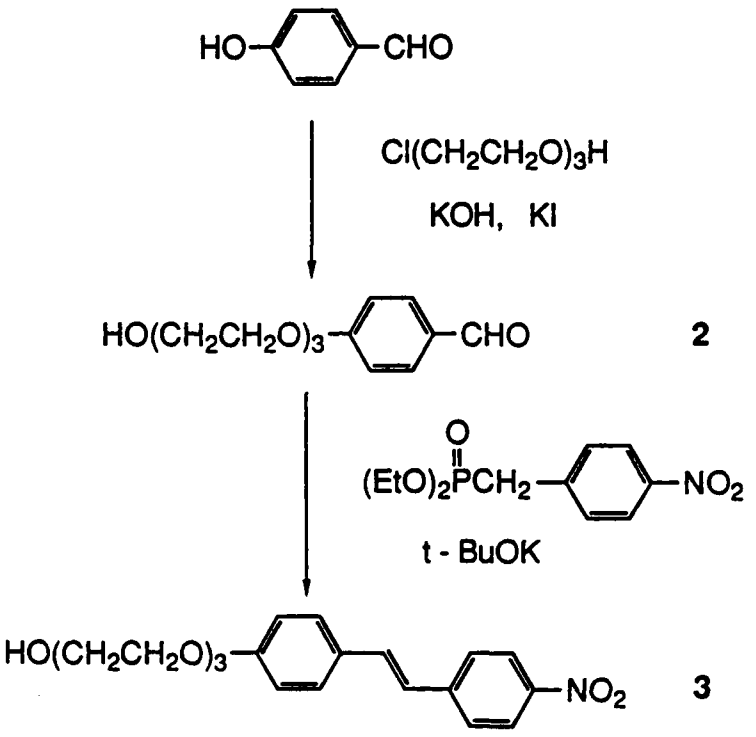
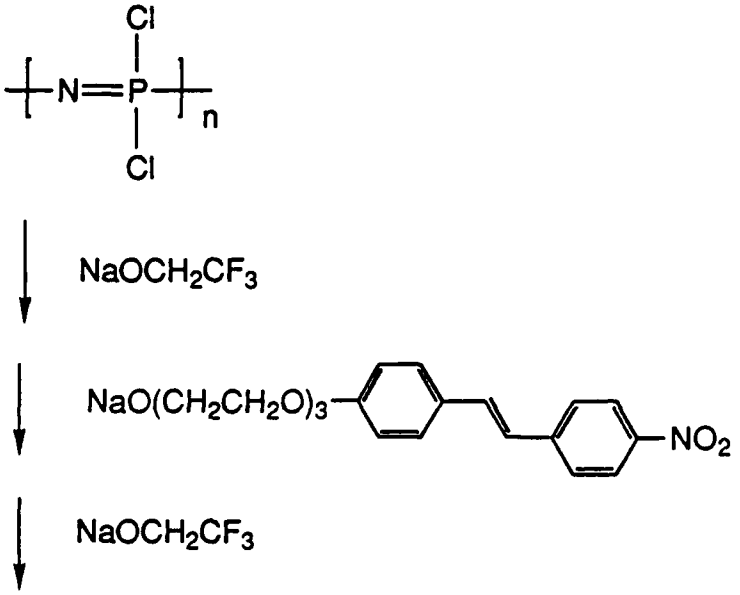
Polymer 1


HO
CHO
Cl(CH2CH2O)3H
KOH, KI
HO(CH2CH2O)3
CHO
2
O
(EtO)2PCH2
NO2
t - BuOK
HO(CH2CH2O)3
NO2
3

Scheme I



Polymer 1

Scheme II

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