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SECOND ORDER NON-LINEAR OPTICAL POLYPHOSPHAZENES

(Proc. of Symp. on Non-Linear Optics, ACS Meeting, Boston, 1990)

by

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Second-Order Nonlinear Optical Polyphosphazenes

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In this contribution we describe the synthesis and second-order nonlinear optical properties of a series of mixed-substituent poly(organophosphazenes) that possess covalently attached donor-acceptor substituted, conjugated moieties. The general structure of the polymers is $[NP(OCH_2CF_3)_x(OR)_y]_n$, where $OR = -O(CH_2CH_2O)_kC_6H_4-CH=CH-C_6H_4NO_2$, where $k = 1-3$, and $-OCH_2CH_2N(CH_2CH_3)C_6H_4-N=N-C_6H_4NO_2$, and $x + y = 100\%$. The nonlinear optical properties of thin films of the polymers were investigated by using second harmonic generation, giving second-harmonic coefficients, d_{33} , in the range 4.1-34 pm/V.

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The development of polymeric nonlinear optical (NLO) materials is currently an area of intense investigation (1-4). 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 (5-7) or can be covalently attached to a polymer backbone (8-14). The noncentrosymmetric alignment of the nonlinear optical molecules in both approaches is achieved by heating the polymer to its glass transition temperature, at which point the chains have reorientational mobility, followed by application of a strong electric field. In this paper, we will discuss the synthesis and nonlinear optical properties of phosphazene macromolecules that possess covalently attached donor-acceptor substituted, conjugated moieties (15). The structures of the nonlinear optical side groups are illustrated in Figure 1. Polyphosphazenes offer a potential advantage in that the macroscopic properties of the polymer can be tailored by the incorporation of specific substituent groups (16-21).

Figure 1 near here.

Synthesis of Nonlinear Optical Side Groups

Our initial work involved the synthesis of side chains which have the molecular characteristics required for a nonlinear optical response. Compounds 1-3 were prepared by the use of Horner-Emmons-Wadsworth Wittig methodology (22). Compound 4 was commercially available (Aldrich) as the dye, Disperse Red 1. As outlined in Scheme I, in the first step in the synthesis of 1-3, 4-hydroxybenzaldehyde was allowed to react with chloroethanol derivatives in basic ethanol containing potassium iodide for 15 h at reflux. The benzaldehyde product was then allowed to react with diethyl(4-nitrobenzyl)phosphonate in the presence of potassium

tert-butoxide in ethylene glycol dimethyl ether for 15 h at room temperature and 1 h at 85°C to yield the stilbene side groups. Compounds 1-3 were purified by column chromatography and were recrystallized from n-hexane/methylene chloride to yield yellow solids.

Scheme I near here.

Compounds 1-4 were characterized by conventional spectroscopic techniques. For the stilbene compounds 1-3, the trans conformation of the double bond was confirmed by ^1H NMR analysis. For example, in the ^1H NMR spectrum of 1, the olefinic protons were detected as a doublet of doublets resonance at 7.23 and 7.01 ppm, with a trans coupling constant of 16.3 Hz. In addition, the ^{13}C NMR spectra of the stilbene compounds indicated the presence of a single isomer that was consistent with the desired structures. The UV/visible spectra in tetrahydrofuran solution showed a λ_{max} value for 1-3 at 378 nm (ϵ 2.6×10^4) and for 4 at 490 nm (ϵ 3.1×10^4).

Synthesis of Nonlinear Optical Phosphazene Macromolecules

The overall synthetic pathway to mixed-substituent polyphosphazenes 5-9 is described in Scheme II, and the corresponding polymer structures and composition ratios are listed in Table I. Poly(dichlorophosphazene) was prepared by the thermal ring-opening polymerization of the cyclic trimer $(\text{NPCl}_2)_3$, as described in earlier papers (16-18). The substitution reactions of poly(dichlorophosphazene) were carried out in three steps. The synthesis and purification of polymer 6 will be discussed as a representative example. In the first step, sodium trifluoroethoxide was added to

poly(dichlorophosphazene) to replace approximately 50% of the chlorine atoms. In the second step, a stoichiometric deficiency of the sodium salt of 1 was allowed to react with the partially substituted polymer. In the final step, an excess of sodium trifluoroethoxide was added to replace the remaining chlorine atoms in order to obtain a fully derivatized, hydrolytically stable polymer. This three step synthetic procedure was necessary because the direct addition of the sodium salt of 1 to poly(dichlorophosphazene) resulted in the formation of an insoluble, incompletely substituted polymeric precipitate. Polymer 6 was isolated by precipitation from the concentrated THF reaction mixture into water and was purified by dialysis against methanol/water (1:1 v/v) for 7 to 10 days.

Scheme II and Table I near here.

The preparation of soluble, single-substituent polyphosphazenes that contained species 1-4 as side groups could not be accomplished because, as noted previously, the direct addition of the sodium salt of the chromophore to poly(dichlorophosphazene) resulted in the formation of a polymeric, incompletely substituted precipitate. The precipitate was insoluble in refluxing THF as well as warm dioxane, N,N-dimethylformamide, dimethylsulfoxide, nitrobenzene and N-methylpyrrolidinone. This insolubility was attributed to both the extended rigid structure and the intrinsically high polarity of the donor-acceptor substituted, conjugated side chains. Both factors may induce extensive side group stacking and thus lead to the formation of insoluble polymers.

The preparation of soluble polymers containing species 1-4 was accomplished by the use of the polar trifluoroethoxy group as co-substituent.

The partially substituted trifluoroethoxy polymer, prepared in the first step of the polymer synthesis (see Scheme II), provided a polar environment for the incorporation of the chromophoric side chains. However, the maximum loading of the polymers by the chromophores 1-4 was limited by the solubility of the polymeric products. Hence, the side group ratios for polymers 6-9 represent a maximum incorporation range of the chromophore side group by the use of this synthetic scheme.

The preparation of mixed-substituent polymers that contained co-substituents other than trifluoroethoxy groups was also explored. This part of the investigation was carried out in an attempt to tailor the macromolecular properties, for example, glass transition temperature, solubility behavior, morphology, and film-forming ability, in order to optimize the nonlinear optical behavior. However, the aryloxy substituents, including phenoxy, 4-methylphenoxy, and 3-ethylphenoxy, as well as the alkoxy substituent, methoxyethoxyethoxy, all yielded insoluble polymers, even with low incorporation ratios (10-15%) of the chromophore. These results suggest that the highly polar trifluoroethoxy group is a necessary co-substituent for the preparation of soluble polymers containing 1-4 as side chains.

Structural Characterization and Properties of Polyphosphazenes

Characterization of polymers 5-9 was achieved by ^1H and ^{31}P NMR spectroscopy, gel permeation chromatography, differential scanning calorimetry, UV/visible and infrared spectroscopy, and elemental microanalysis. All the polymers were soluble in common organic media, such as tetrahydrofuran, acetone, and methylethyl ketone.

A typical ^{31}P NMR spectrum consisted of a sharp, singlet resonance at -8 ppm, presumably a consequence of the similar environment at the trifluoroethoxy and ethoxy-ether substituted phosphorus atoms in the mixed substituent system. In addition, the singlet resonance indicated a high degree of chlorine replacement. This was supported by the elemental microanalysis data.

The substituent ratios of the polymers were determined by ^1H NMR analysis by a comparison of the integration of the combined aromatic and vinyl resonances, which were generally between 8.4 and 6.8 ppm, with the trifluoroethoxy resonance at 4.5 ppm.

The molecular weights of polymers 5-9 were estimated by gel permeation chromatography to be in the range $M_n = 9.4 \times 10^4$ to 3.2×10^5 , $M_w > 9.3 \times 10^5$, with M_w/M_n values in the region 4-7. UV/visible spectra in tetrahydrofuran showed the same trends as the corresponding side group compounds 1-4, with λ_{max} values in the range 369-378 nm for 5-7 and 468 nm for 8. Infrared spectroscopy of thin films cast on KBr for all of the polymers showed an intense P=N stretching vibration at $1250\text{-}1200\text{ cm}^{-1}$. In addition, the absorbance for the NO_2 unit at ca. 1345 cm^{-1} was detected.

The glass transition temperature (T_g) of the mixed-substituent polyphosphazenes 5-9 varied with the loading of the chromophoric side chain and with the length of the connecting ethyleneoxy spacer group. Species with one ethyleneoxy unit comprising the spacer group generated the highest glass transition temperature. The T_g values were 19°C for 5, 25°C for 6, 25°C for 7, 54°C for 8, and 44°C for 9. No evidence of $T(1)$ or T_m transitions were detected for any of these polymer samples. Hence, the addition of the chromophoric substituent disrupts the microcrystallinity of the

single substituent polymer $[\text{NP}(\text{OCH}_2\text{CF}_3)_2]_n$, which has a T_g at -66°C , a $T(1)$ between 60 and 90°C , and a T_m at 240°C (24).

The colors of the polyphosphazenes corresponded to those of the chromophores employed. Thus, polymers 5-8, which contained chromophores 1-3, were yellow, while polymer 9, which contained chromophore 4, was red.

Evaluation of the Second Order Nonlinear Optical Behavior

Films of polyphosphazenes 5-9 were spin cast onto indium-tin oxide coated glass from a concentrated solution in methylethyl ketone. The solution was first filtered to remove particulate impurities and the films were heated to 80 - 85°C to remove the solvent. The thicknesses and refractive indices of the polymers were obtained from ellipsometric measurements on calibration layers, which were spun on BK7 glass substrates. Measurements on each sample were performed at four different wavelengths (634.8 nm, 753.0 nm, 802.0 nm and 852 nm) in order to minimize the errors in the extrapolated values at 532 and 1064 nm. The thickness of the layers examined ranged from 70 - 250 nm, and were always much less than the coherence lengths, as determined from the refractive index measurements.

The NLO properties of the films were subsequently investigated using second-harmonic generation. A Q-switched Nd:YAG laser ($\lambda = 1064$ nm) 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.

Alignment of the NLO side groups in the films 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. Increasing poling voltage led to an increase in

the harmonic intensity, i.e. maximum alignment was not achieved at this voltage. However, higher voltages occasionally resulted in damage to the sample surface. Hence, for comparison purposes, the voltage was limited to 10 kV. Because of the low glass transition temperatures of these polymers, the poling was carried out at room temperature, concurrent with the second-harmonic generation measurements. This arrangement had the advantage of reproducibility of the measurement condition for each layer. Following removal of the poling field, the second-harmonic signal decayed to zero within a few minutes.

The values of the second-harmonic coefficient, d_{33} , for samples 5-9 are listed in Table II. The values of d_{33} were obtained using the analysis of Jerphagnon and Kurtz (25), and were calculated under the assumption that the degree of alignment of the nonlinear optical chromophores can be described using the isotropic model. Hence, we assumed $d_{33}=3d_{31}$ (4).

Table II near here.

In the series of polymers 5-8, which contain the nitrostilbene side groups 1-3, the trend in the d_{33} value versus loading of the chromophoric side group was well reproduced, with d_{33} values in the range 4.1-5.0 pm/V. Note that the decrease in the spacer length from three to one ethyleneoxy units appeared to have no effect on the d_{33} value. For polymer 9, which contained the high β azo chromophore 4, the d_{33} value was 34 pm/V, which was significantly higher than for the stilbene substituted polymers that contained equivalent side group incorporation ratios. This is partially a consequence of the greater resonant enhancement, given the longer wavelength of the azo chromophore absorption peak.

Conclusions and Future Prospects

The synthetic versatility offered by the phosphazene system has allowed the preparation of polymers that contain nonlinear optical units as pendant side chains. Our future research on nonlinear optical polyphosphazenes will focus on tailoring the macromolecular system to generate higher glass transition temperatures. This, and the stabilized alignment of the chromophoric side groups, should be attainable by the incorporation of a third co-substituent that contains a crosslinkable moiety. Thus, crosslinking of the polymer matrix during the application of an electric field would be expected to stabilize the nonlinear optical character.

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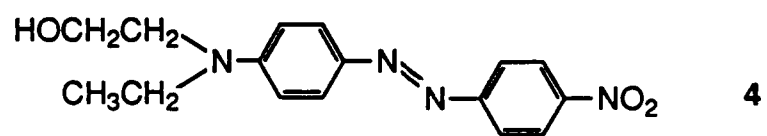
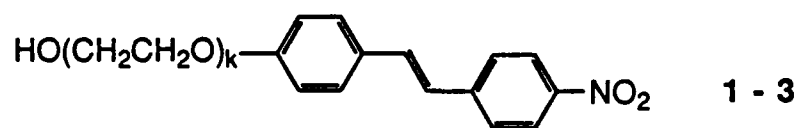
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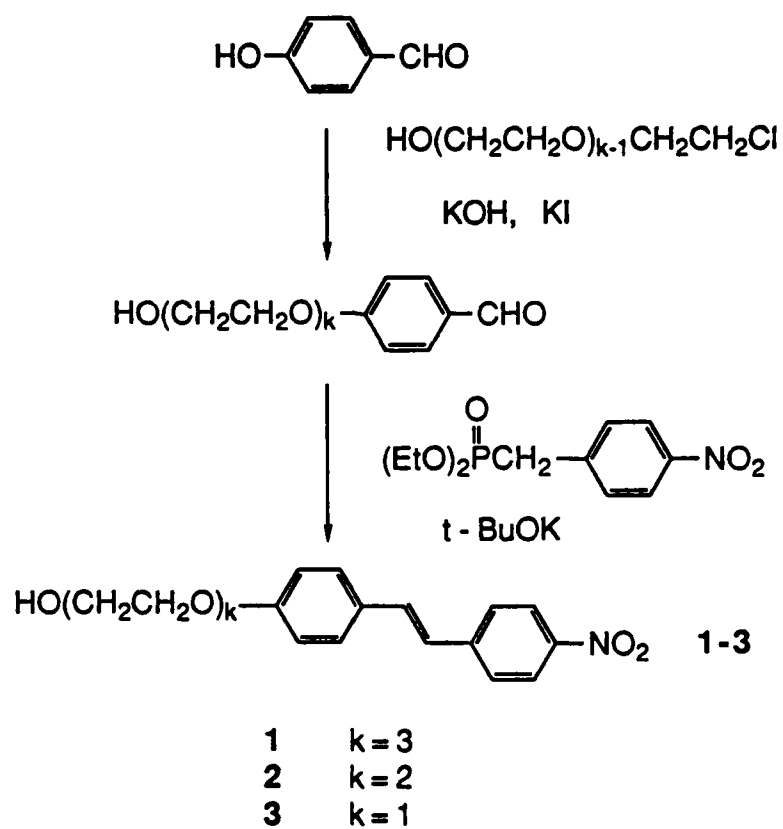
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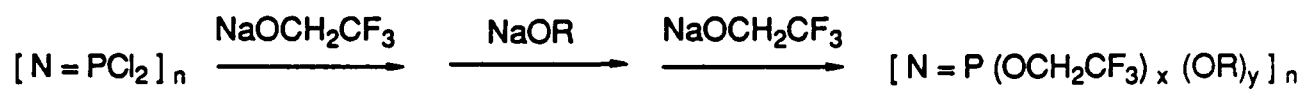
Figure 1



Scheme I



Scheme II



5 - 9

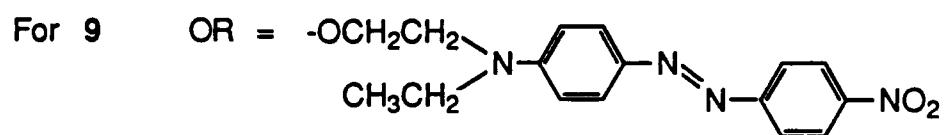
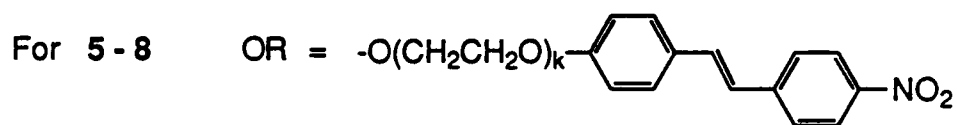


Table I
Polyphosphazene Structures and Composition Ratios

Compd ^a	Side Group Structure ^b	y ^c , %
5	k = 3	26
6	k = 3	36
7	k = 2	39
8	k = 1	31
9	-	33

^aSee Scheme II for general polymer structure

^bSee Figure 1 for general side group structure

^cx + y = 100%

Table II
 d_{33} Coefficients for Polyphosphazenes

Compd ^a	d_{33} , pm/V
5 (y=26%)	4.1
8 (y=31%)	4.7
6 (y=36%)	5.0
7 (y=39%)	5.0
9 (y=33%)	34

^aSee Scheme II and Table I for polymer structures and composition ratios;
Polymers 5-8 arranged in order of increasing value of y

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