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New Synthesis of a Soluble High Molecular Weight Poly(arylene vinylene): Poly[2-methoxy-5-(3,7-dimethyloctyloxy)-*p*-phenylene vinylene]. Polymerization and Device Properties

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ABSTRACT: A novel synthetic approach to high molecular weight poly(arylene vinylene) derivatives has been investigated. Soluble precursors of poly[2-methoxy-5-(3,7-dimethyloctyloxy)-*p*-phenylenevinylene], called OC1C10, were synthesized via a nonionic route. The precursor and the conjugated polymer are discussed along with the electroluminescence and photoluminescence of the latter.

I. Introduction

Conjugated polymers are of great interest for the development of optical[1] and electronic applications. Many of these conjugated polymers are insoluble in common solvents and are infusible at temperatures below the decomposition temperature of the polymer. It is only rather recently that there has been real progress with the development of useful processing routes. In precursor polymer routes, a solution-processable "precursor" polymer is formed, usually as a thin film which is converted in situ to form the conjugated polymer by thermal or chemical treatment to eliminate a volatile side group. The Wessling precursor route[2] is a well-established route for preparing poly(*p*-phenylenevinylene), PPV, and derivatives, in which bis(sulfonium halide) salts of *p*-xylene are treated with an alkali base in water. The mechanism is believed to process via the in situ generation of the actual monomer, a *p*-quinodimethane system. It is still under discussion as to whether the polymerization is ionic[3] or free radical.[4] Control of molecular weight and molecular weight distribution measurements of the ionic precursors is very difficult due to irreversible interactions between the precursor polymer and the gel permeation chromatography column.[4] Another drawback is the instability of the precursor polymer: the sulfonium groups are good leaving groups, which results in unwanted solvent substitutions and gel formation. Moreover, many efforts have been made to develop directly processable conjugated polymers.[5] Such soluble polymers no longer require thermal treatment during device fabrication, which is another drawback of the PPV precursor route.

An alternative route was developed by our research group.[6−8] This novel route is particularly attractive because it produces polymeric intermediates soluble in organic solvents avoiding the ionic precursors. Therefore, polymer characterization and molecular weight determination can be performed more easily. The general idea of this new route is the base-induced polymerization of *p*-arylene derivative which have the following groups: (i) a polarizer group P in the α-position which can stabilize the anion formed on the acid−base equilibrium; (ii) a good leaving group, L, in the α′-position.

These two features allow the formation of *p*-quinodimethane (**3**) (the "real" monomer) represented in Scheme 1 for the example of unsubstituted PPV.[9]

We have reported the unsymmetric monomer synthesis toward synthesis of poly(*p*-phenylenevinylene) in a previous paper.[10] 2-Chloromethyl-5-*n*-butylsulfomethyl-1-(3,7 dimethyloctyloxy)-4-methoxybenzene is synthesized starting from 2,5-bis(chloromethyl)-1-(3,7-dimethyloctyloxy)-4-methoxybenzene (abbreviated BCDM) with an unexpected high selectivity.[11] BCDM is industrially applied as a monomer in dehydrogenation polymerization toward the commercial conjugated OC1C10 polymer. In this paper, polymerization by use of the nonionic procedure with this asymmetric monomer is described. The precursor and the conjugated polymer are characterized, and photoluminescence and electroluminescence properties of the latter are discussed.

II. Polymerization by a Nonionic Procedure

Typical Procedure for the Polymerization of the Asymmetric Monomer Derived from 2,5-Bis(chloromethyl)-1-(3,7-dimethyloctyloxy)-4-methoxybenzene (BCDM). In Scheme 2 the new approach is shown for the synthesis of the polymer precursor **8** of the conjugated polymer OC1C10 using a new nonionic precursor route.

Since the polymerization of α-chloro-α′-alkylsulfinyl-*p*-xylene is best performed in *sec*-butanol the monomer derived from BCDM is also converted in this solvent.[10−11] The polymerization is carried out as described in the Experimental Section. Note that polymerization is not done with a purified monomer but with a crude monomer mixture of BCDM (18%) (**11**), two monosubstituted isomers **13** and **14** (74%) and the disubstituted product **12** (7%). Scheme 3 gives an overview of the possible products after polymerization. The concentration used was 0.10 M.

The reaction mixture consists mainly of precursor polymer **9**, unreacted BCDM (**11**), and unreacted disubstituted product **12**. No solvent-substituted product is detected. Purification of this mixture by precipitation

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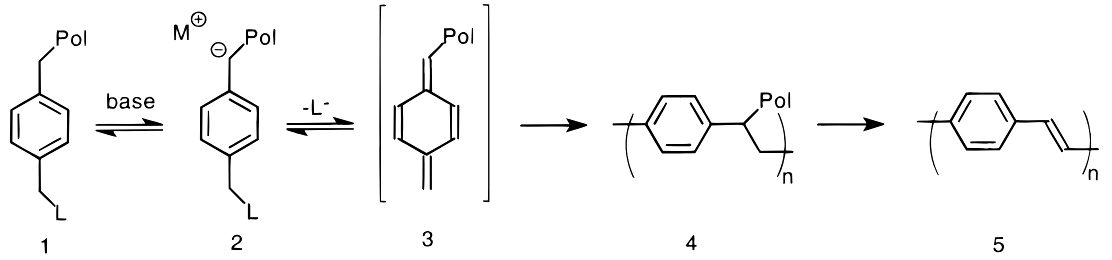
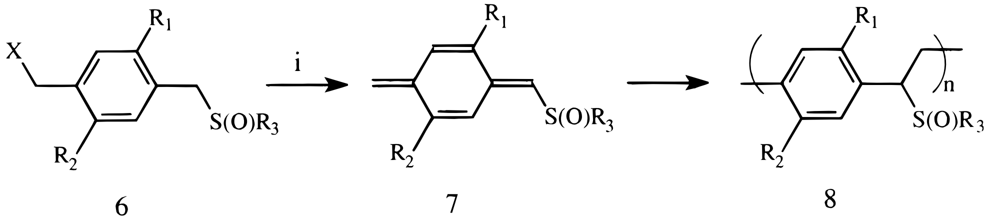
Scheme 1. General Scheme of the PPV Polymerization

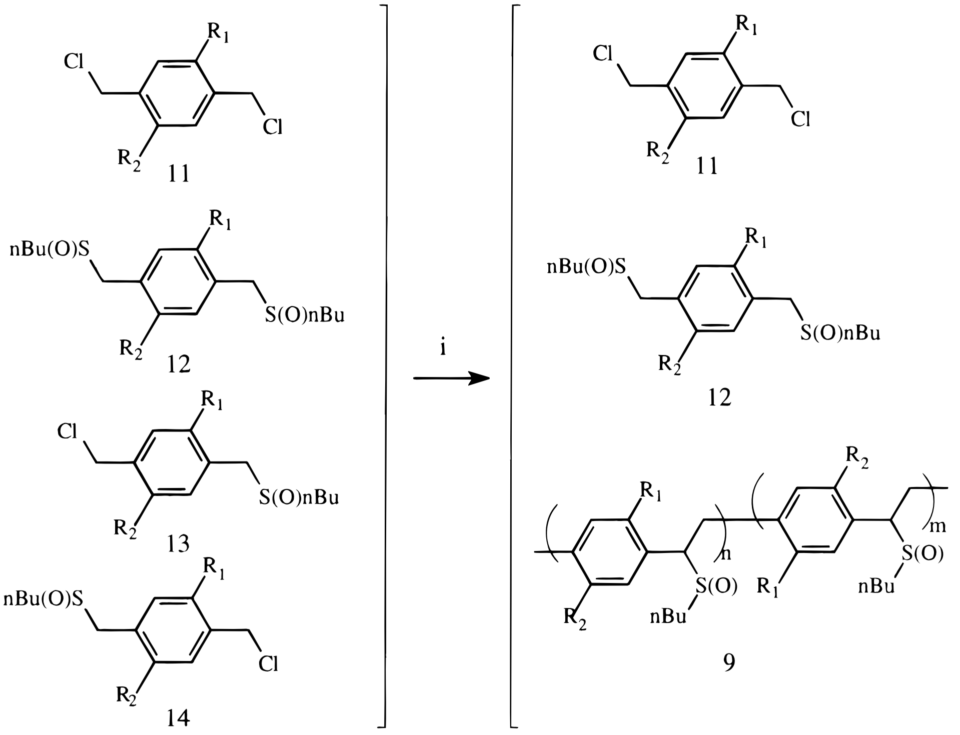


Scheme 2. Polymerization of Asymmetric Monosulfinyl Derivate of BCDM to Precursor Polymer

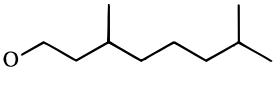


i : 1.0-1.3 eq. NaOtBu, Solvent, 30 °C, 1 h, $R_1 = OC_{10}H_{21}$, $R_2 = OCH_3$, $R_3 = C_4H_9$

Scheme 3. Possible Products after Polymerization



i : 1.3 eq. NaOtBu, solvent, RT, 1 h



R_1 =

$R_2 = OCH_3$

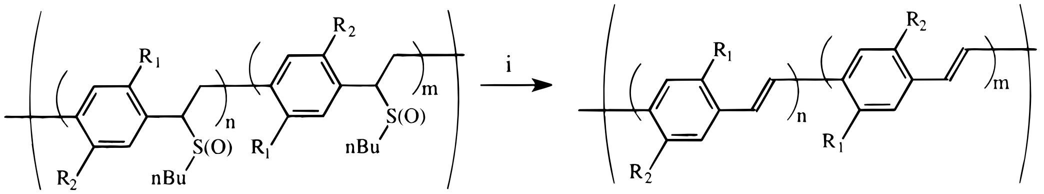
is impossible. The difference in solubility characteristics between the products mentioned is not significant enough for an efficient purification. Furthermore, the precursor polymer probably has a T_g below room temperature, thus complicating isolation of the polymer. Therefore, purification was postponed until the precursor was converted to the conjugated polymer. OC1C10 polymers are actually copolymers of two monomer isomers, **13** and **14**.

Molecular weight determination by size exclusion chromatography, SEC, in THF calibrated with polystyrene standards gave a $\bar{M}_w$ of 307 000 and a dispersity of 2.1 for the precursor polymer.

The concentration during polymerization can be increased to at least 0.15 M. This mainly depends on the solubility of the monomer in the solvent used. This is a significant improvement compared to the polymerization of BCDM by the classical dehydrohalogenation route in which the concentration needs to be much lower.

The polymerization of only one pure monomer isomer, **13**, obtained by chromatographic separation, was car-

Scheme 4. Conversion of Precursor Polymer to OC1C10



9 10

i : toluene, reflux, 3 h.

Table 1. Yields (%), Number-Average Molecular Weights (M_n), Weight-Average Molecular Weight (M_w), and Degree of Polymerization (DP) As Determined by SEC of OC1C10-Gilch and OC1C10-New

polymer	yield[a] (%)	$M_n \times 10^5$	$M_w \times 10^6$	DP
OC1C10-Gilch	52	1.3	1.2	9.2
OC1C10-New	98	1.5	0.56	3.8

[a] Starting from monomer.

ried out, also leading to a homopolymer: the so-called “OC1C10-homopolymer”. The solubility of this homopolymer is extremely reduced compared to that of the copolymer OC1C10, which makes the study of the former material more difficult. [11]

Conversion of Precursor to Conjugated Polymer OC1C10. The conversion of the precursor polymer to the conjugated polymer is carried out by refluxing in toluene (110 °C) for 3 h under nitrogen atmosphere, Scheme 4.

A yield of 3.6 g (98% starting from monomer, isomer mixture **13** + **14**) of conjugated polymer was obtained after precipitation in methanol. Molecular weight determination by SEC in THF against polystyrene standards gave a $\bar{M}_w$ of 560 000 and a polydispersity of 3.8.

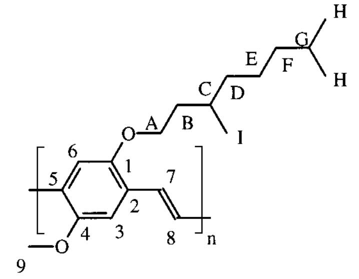
III. Results and Comments

Conjugated polymer OC1C10 obtained by a dehydrohalogenation route (the Gilch procedure) is at this moment used in industry for producing the active layer in LED applications.[12] Therefore, we decided to study the device properties of our OC1C10 obtained by our nonionic polymerization route “OC1C10-New” and to compare its properties with those of the classical OC1C10 used in industry, which is obtained from the Gilch route, “OC1C10-Gilch”.[13]

Synthesis, Spectroscopic, and Thermal Results. The polymerization results of OC1C10 polymers are summarized in Table 1. The number-average molecular weight (M_n) and the weight-average molecular weight (M_w) of these polymers, determined by gel permeation chromatography using polystyrene standards, were in the range of 150 000−130 000 and 560 000−1 200 000 with a polydispersity index of 3.8−9.2.

Structural features of the resulting polymers OC1C10 (OC1C10-Gilch, OC1C10-New) were identified by FT-IR, ^{1}H NMR, and ^{13}C NMR. Absorption peaks at 970 and 3050 cm^{-1} appear after the elimination reaction, which corresponds respectively to the out-of-plane bending and C−H stretch of *trans*-vinylene groups, suggesting that the generated double bonds in the main chain are mainly in the trans configuration. No major difference was observed between OC1C10-Gilch and OC1C10-New in ^{1}H NMR. As the elimination reaction on the precursor polymer proceeded, the methine proton adjacent to the sulfoxy group at 4.55 ppm disappeared and new vinylic protons appeared at 7.45 ppm along with aromatic protons of the backbone at 7.05 ppm. Also, the signal at 3.9 ppm was assigned to the methoxy protons while the methylene protons adjacent to the oxygen atom in the OC10 side chain are observed at 4.1 ppm. ^{13}C NMR APT (attached proton test) and two-dimensional NMR spectra ^{1}H−^{13}C (HETCOR) of OC1C10-New further confirm its molecular structure. All peaks have been attributed as shown in Table 2. The method of synthesis of the polymer does not have an influence on the ^{13}C NMR spectrum.

Table 2. ^{13}C and ^{1}H NMR Chemical Shifts and Assignment of OC1C10-New



chemical shift (ppm)		
^{13}C NMR	^{1}H NMR	assignment
19.8	0.98	I
22.6	0.85	H
24.7	1.30	E
27.9	1.50	G
30.2	1.76	C
36.5	1.9 /1.65	D
37.4	1.12/1.38	F
39.3	1.12	B
56.2/56.4	3.9	9
67.7/67.9	4.15	A
110.5/110.3/108.9/109.6	7.05	3, 6
127.1		2, 5
123.0	7.45	7, 8
151.5/151.1		1, 4

^{1}H NMR wide-line relaxometry is a technique that can be used to study the local mobility at temperature above T_g and phase morphology at temperature below T_g in a polymer on the nanometer level. Despite the loss of chemical shift information, the advantage of wide-line NMR compared to high-resolution ^{13}C CP/MAS solid-state NMR is that it is a fast technique. For example a T_{2H} experiment takes only a few minutes while many hours are needed for a ^{13}C CP/MAS experiment. In wide-line NMR the T_{2H} relaxation time constants are obtained out of an analysis of the FID. In this technique, no line narrowing techniques are used; the proton spectrum consists of a broad signal. Thus. no differentiation can be made between the backbone and the side chain protons. For many years this technique had less success compared to ^{13}C CP/MASS

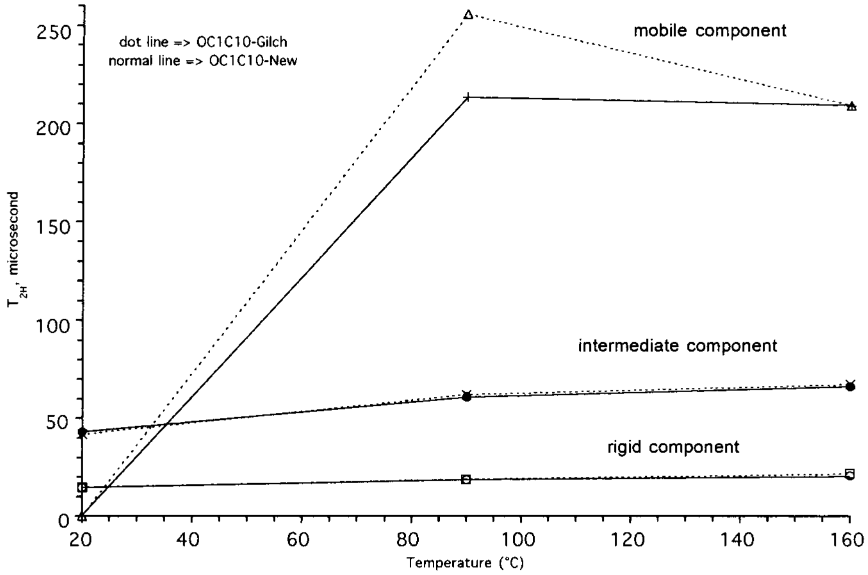


Figure 1. T_{2H} Relaxation times of OC1C10-New and OC1C10-Gilch as a function of the temperature between 20 and 160 °C. A Gaussian (rigid component) and one or two exponential functions were used for the analysis. Dotted line: OC1C10-Gilch. Normal line: OC1C10-New.

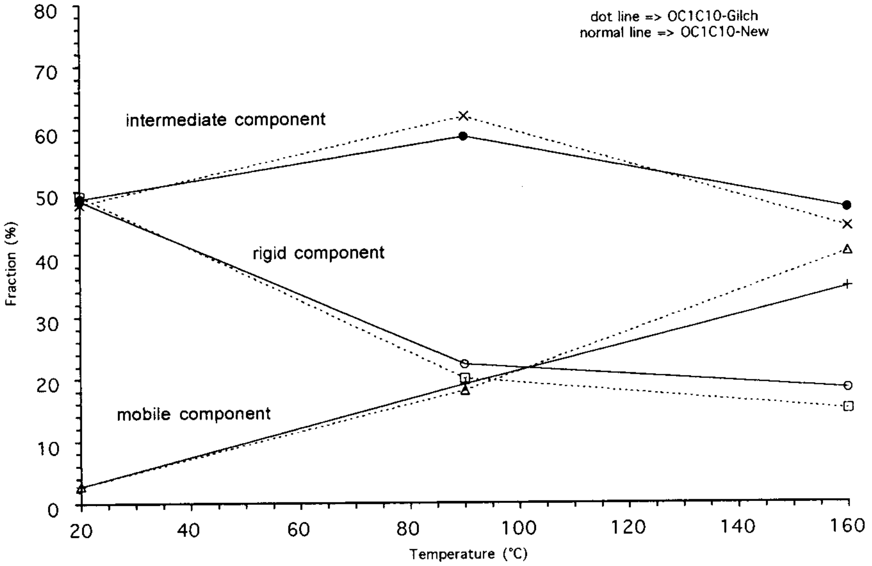


Figure 2. Influence of the temperature on the fractions of the different spin–spin relaxation times of OC1C10-New and OC1C10-Gilch. Dotted line: OC1C10-Gilch. Normal line: OC1C10-New.

NMR because the results are rather difficult to interpret.

T_{2H} was measured for a OC1C10-New sample as well as for a OC1C10-Gilch sample by using the solid echo pulse sequence. Spectra are recorded with a dwell time of 0.5 μs allowing an accurate determination of the echo maximum. A Gaussian and an exponential functions were used for the analysis of the FID.

The number of components needed to fit the FID depends on the temperature. At room temperature, a Gaussian function with a small value of T_{2H} and one exponential function are needed to describe the decay. At higher temperatures, one Gaussian function and two exponential functions are necessary (Tables 3 and 4). The three components will be named as folows: (1) rigid component with a short T_{2H}; (2) mobile component with a long T_{2H}; (3) intermediate component corresponding to a population with a mobility between the rigid and the mobile component. The short T_{2H} relaxation time which is connected to the rigid component is constant over the whole temperature range while the other T_{2H} constants increase with the temperature (Figure 1). The mobility of the polymer chains in the amorphous domains increases with the temperature (specially when temperature exceeds T_g) which explains the increasing spin–spin relaxation time with the temperature.

Table 3. T_{2H} Relaxation Times of OC1C10-Gilch at Various Temperature with the Related Fraction Percentage

	OC1C10-Gilch	OC1C10-Gilch-90 °C	OC1C10-Gilch-160 °C
		Rigid Component	
fraction (%)	49	20	15
T_{2H} (μs)	15	19	22
		Intermediate	
fraction (%)	48	62	45
T_{2H} (μs)	42	62	67
		Mobile	
fraction (%)	none	18	40
T_{2H} (μs)	none	255	209

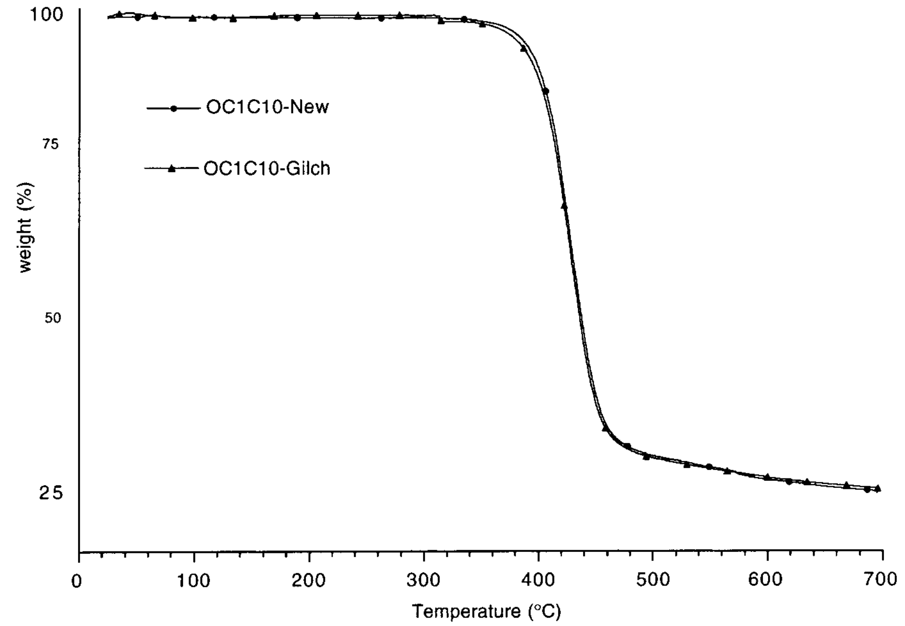


Figure 3. TGA thermograms of different OC1C10 polymers at 10 °C min^{-1}, under N_2 flow.

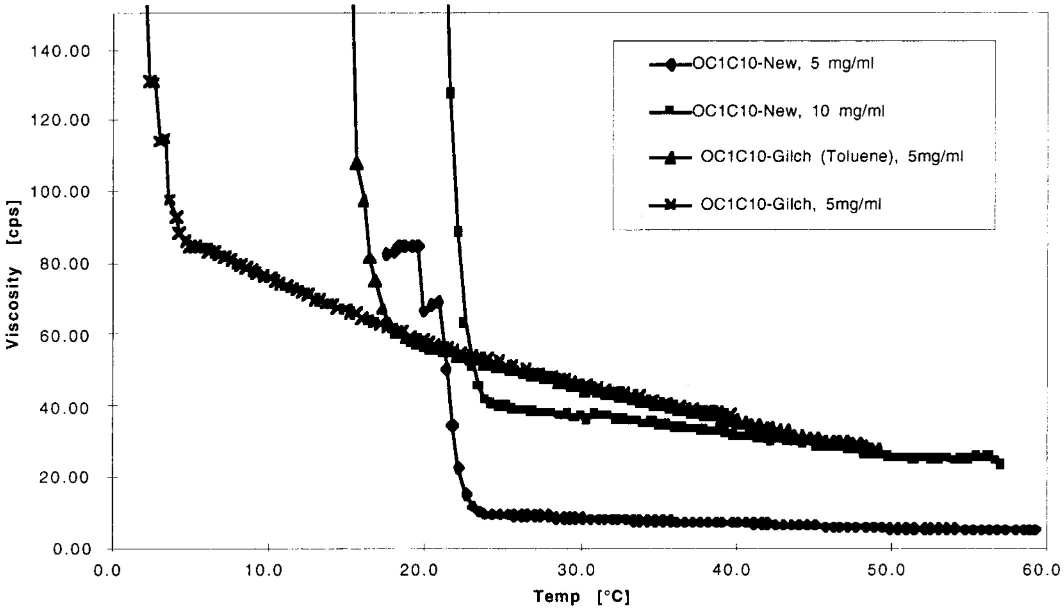


Figure 4. Gelation behavior of different OC1C10 polymers in solution for 70:30 toluene/THF (exception noted).

Table 4. T_{2H} **Relaxation Times of OC1C10-New at Various Temperature and Related Fraction Percentages**

	OC1C10-New	OC1C10-New-90 °C	OC1C10-New-160 °C
	Rigid Component		
fraction (%)	48	22	18
T_{2H} (μs)	14	18	20
	Intermediate		
fraction (%)	49	59	47
T_{2H} (μs)	43	61	66
	Mobile		
fraction (%)	none	19	34
T_{2H} (μs)	none	213	209

For polymers without long side chains one normally observes a single Gaussian component ($T_{2H} \approx 10-15$ μs for both the amorphous and crystalline domains) at temperature below T_g. When temperature exceeds T_g, the amorphous phase starts to differentiate (increase of T_{2H}; additional exponential component) from the crystalline phase, resulting in a clear fractional separation of amorphous and crystalline phase allowing the determination of the degree of crystallinity of the polymer. Because of the presence of the long side chain in OC1C10, straightforward interpretation of each fraction is almost impossible. Nevertheless, the technique still allows a very sensitive comparison of both species toward their molecular mobility.

The temperature has also an influence on the fractions related to these three relaxation times (Figure 2). At room temperature there are only two components, one rigid and one intermediate component. The fraction of the rigid component decreases with the temperature in favor of the two other components. The fraction of the mobile component keeps growing over the whole range of temperature while the fraction of the intermediate component shows a maximun.

Both OC1C10-Gilch and OC1C10-New have a similar behavior. The local mobility of both polymers can be

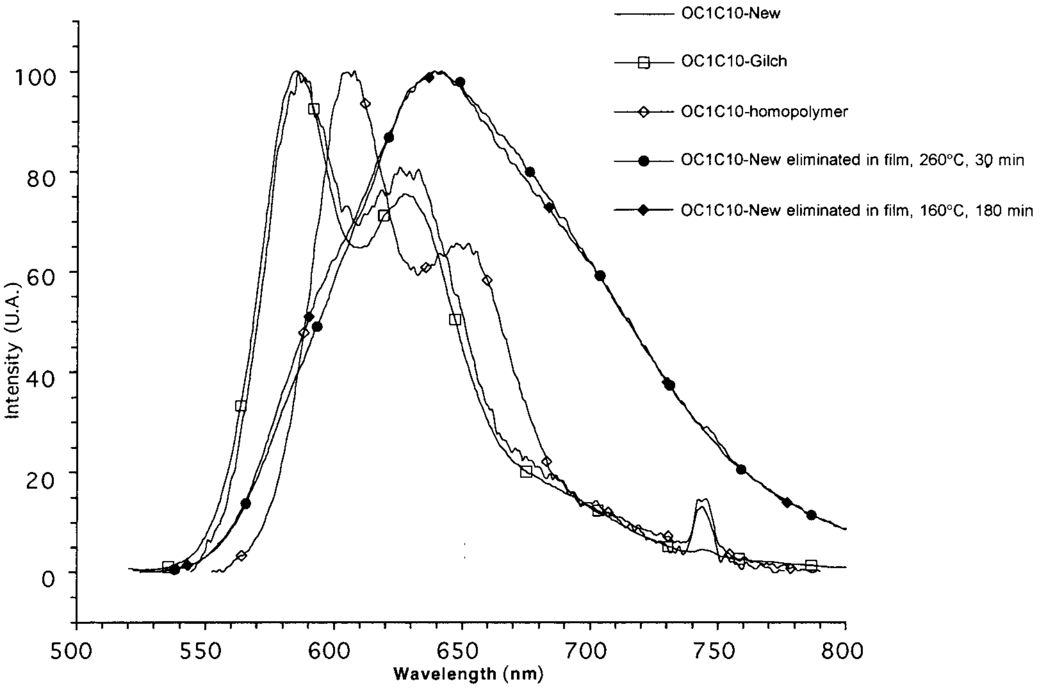


Figure 5. PL spectra of OC1C10 polymers.

Table 5. Temperature of the Initial Loss of Mass Observed on TGA Thermogram, T_i, Char Yield Calculated at 600 °C from TGA Thermogram, T_g Temperature, UV λ_{max} and PL λ_{max} Values of OC1C10-Gilch and OC1C10-New

				λ_{max} (nm)	
polymers	T_i (°C)	yield (%)	T_g (°C)	UV	PL
OC1C10-Gilch	315	28	48	481	586−628
OC1C10-New	320	25	50	507	584−628

described by two T_{2H} relaxation components at room temperature and three at higher temperature. No main difference is noticed in fraction repartition nor in spin−spin relaxation time for each component.

Physicochemical Properties. The thermal behavior properties, UV λ_{max} and PL λ_{max} of the OC1C10 polymers are presented in Table 5 and shown in Figure 3. A study by DIP−MS−EI (50 °C/mn, N_2 flow, figure not shown) shows, for both polymers, a side chain OC_{10} degradation occurring between 330 and 450 °C. This gas elimination shows only one maximum at 380 °C for OC1C10-New and two maxima at 380 and 430 °C for OC1C10-Gilch.

Gelation behavior of OC1C10 polymers in solution in toluene/THF or pure toluene was studied by means of viscosity measurements against temperature, Figure 4. Starting from solutions at 50 °C $< T <$ 60 °C, the solutions were cooled with a rate of 1 °C min^{-1}. The viscosity was measured every $^1/_2$ min at a shear rate of 20 s^{-1}. When the sample is cooled, the viscosity increases until a gelation point is reached. Gelation temperatures and viscosities of OC1C10 polymers in a 70:30 solution of toluene/THF are summarized in Table 6. The aggregation phenomena of polymer chains seems to be easier for the OC1C10-New polymer than for the OC1C10-Gilch polymer. This might be a consequence of the structure of OC1C10-New which, by means of the synthesis method, is expected to have a more regular structure and/or a lower molecular weight distribution.

Table 6. Gelation Temperature and Viscosity Characterizatics of OC1C10 Polymers

polymers	gelation temp (°C)	viscosity 5 mg/ml, 30°C (cps)
OC1C10-Gilch	3	46
OC1C10-New	23	8.5

Table 7. Comparison PL_{eff} of Different OC1C10 Devices: Precursor Eliminated in Solution

polymers	PL_{eff}	av PL_{eff}
OC1C10-Gilch	20.12, 16.84	17.85
OC1C10-New	18.37, 17.34	17.85

Table 8. PL_{eff} of OC1C10-New: Precursor Eliminated in Films

polymer	temp (°C)	time (min)	atm	PL_{eff}	av PL_{eff}
OC1C10-New	260	30	vac	8.89	8.77
	260	30	vac	8.65	
	160	180	vac	11.37	11.38
	160	180	vac	11.40	

Optical and Photoluminescent Properties. The photoluminescence (PL) of OC1C10 polymer thin films coated on glass is shown in Figure 5. A laser with a wavelength of 488 nm was used. PL emissive peaks with maxima approximatively at 590 and 630 nm can be observed in the red region for both OC1C10-Gilch and OC1C10-New. Although the method of synthesis of OC1C10 is expected not to have an influence on the photoluminescence properties, the method of processing the thin film, or on the other hand a slight difference in structure of the polymer, can have some influence as shown below.

(1) We can observe only one broad PL emissive peak with a maximum at 640 nm in films coated from precursor polymers and converted to conjugated polymers afterward by heating at 160 °C and 180 min or 260 °C and 30 min under vacuum.

(2) The PL spectrum of the homopolymer of one pure isomer OC1C10-homopolymer, shows a red shift of about 20 nm due to a higher regularity of structure.

In Tables 7 and 8 a comparison is shown of PL_{eff} for different OC1C10 polymers. No strong differences are

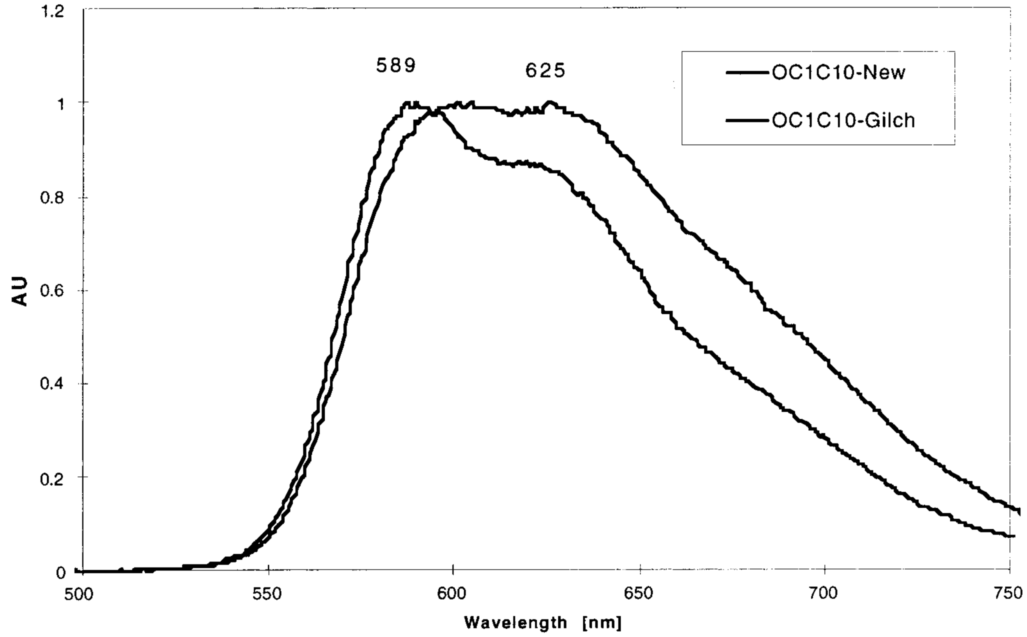
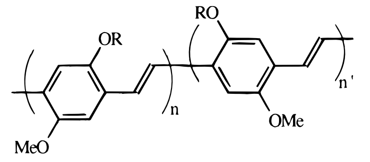
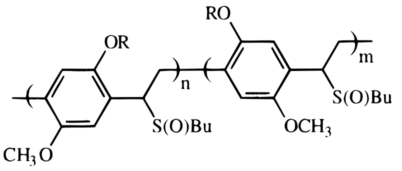


Figure 6. EL spectra of OC1C10 polymers.

detected between OC1C10-Gilch and OC1C10-New for spin-coated conjugated polymers obtained from boiling toluene eliminated precursor polymer. Low PL efficiency can be detected for films spin-coated with soluble precursor polymers and converted in films by heating under vacuum. Temperature and time for conversion reaction seems to have an influence as shown in Table 8. A lower conversion temperature but a longer time gives a better PL efficency.



OC1C10-New



Precursor polymer of OC1C10-New

Electroluminescent Properties and Current–Voltage Characteristic. The spectra of OC1C10 polymers, Figure 6, give EL emissive bands at 590 and 625 nm, at which the maxima in the PL spectra also appeared.

Figure 7 shows the current–voltage (*I*–*V*) characteristics of a single layer light-emitting diode of a glass/ITO/OC1C10/Ca. The forward bias current is obtained when the ITO electrode is positively biased and the Ca electrode is negatively biased. The current increases with increasing forward bias voltage. The turn on voltage is in the range 2–3 V. The luminance against current density is also shown in Figure 8. The luminance obtained for OC1C10-New is substantially lower compared with that of OC1C10-Gilch. The luminescence efficiency of OC1C10-Gilch is three times higher than of OC1C10-New, Table 9. Light emission becomes visible at a bias of about 3.5 V for OC1C10-New against 2.5 V for OC1C10-Gilch, Figure 9.

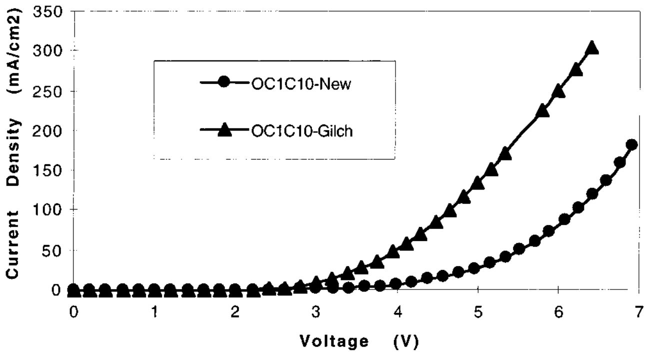


Figure 7. Current–voltage spectra of OC1C10 polymers.

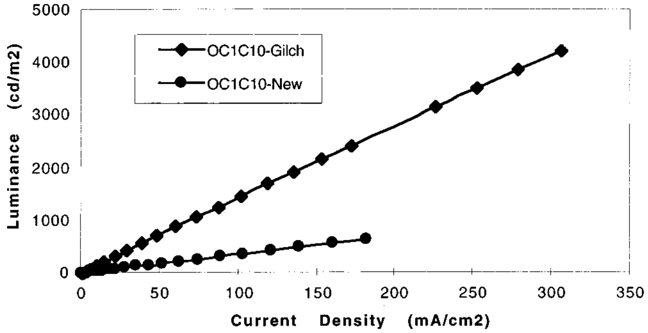


Figure 8. Current density–luminance spectra of OC1C10 polymers.

Table 9. Luminescence Efficiency for OC1C10 Polymers

polymer	luminescence efficiency (cd/A)
OC1C10-Gilch	1.2
OC1C10-New	0.4

IV. Experimental Section

Precursor Polymer 9. A 7 g sample of a 50:50 mixture of isomers α-chloro-α′–*n*-butylsulfinyl-2-(3,7-dimethyloctyloxy)-5-methoxy-*p*-xylene and α-chloro-α′–*n*-butylsulfinyl-2-meth-

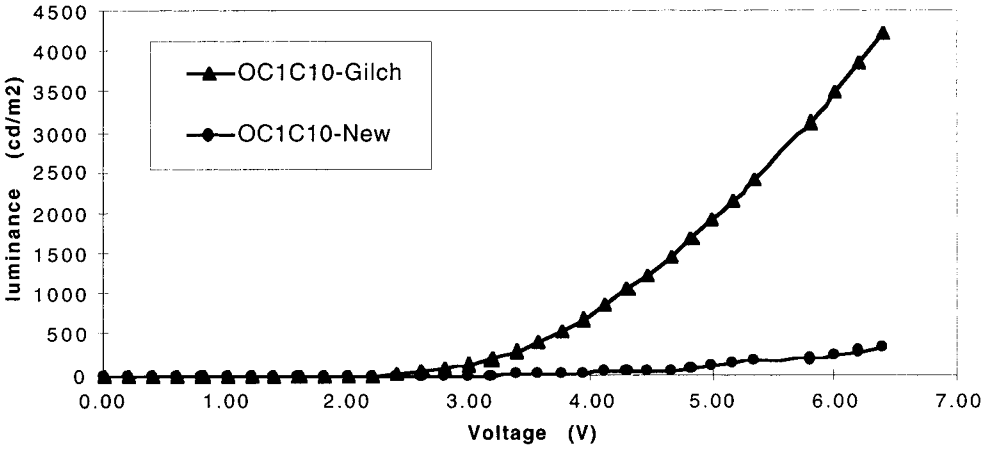


Figure 9. Voltage−luminance characteristics of OC1C10 polymers.

oxy-5-(3,7-dimethyloctyloxy)-*p*-xylene (0.0163 mol) was dissolved in 60 mL of *s*-BuOH, and the solution was flushed with dry nitrogen under stirring for 1 h. A 2.04 g sample of NaO-*t*-Bu (0.0214 mol) was dissolved in 60 mL *s*-BuOH, and the solution was flushed with nitrogen for 1 h. NaO-*t*-Bu solution was added in one go and the polymerization was allowed to proceed at 23 °C for 1 h (still under a constant stream of nitrogen). Then the reaction mixture was poured into 300 mL of H_2O and neutralized with 0.1 M HCl solution. The water layer was extracted with 200 mL of chloroform. The organic layer was dried over $MgSO_4$ and concentrated under vacuum. A 6 g yield of raw precursor polymer was obtained. Molecular weights according to SEC (polystyrene standards): $M_p = 2.18 \times 10^5$; $M_w = 3.07 \times 10^5$; $M_n = 1.47 \times 10^5$; $D = 2.08$. IR (NaCl): ν 2954, 2919, 2862, 1508, 1459, 1409, 1325, 1219, 1035, 866, 753, 683 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ 0.6−0.9 (3H, br), 1.1−1.4 (2H, br), 1.4−1.7 (2H, br), 1.8−2.1 + 2.1−2.5 (2H, br), 2.9−3.3 (1H, br), 3.3−3.9 (2H, br), 6.7−7.5 (4H, br) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.5 (1H, C13), 19.1 (1H, CI), 21.7 (1H, C12), 22.4 (1H, CH), 24.5 (2H, CE + C11), 27.7 (1H, CG), 28.2 + 29.0 (1H, C8), 29.7 (1H, CC), 36.3 (1H, CB), 37.2 + 37.4 (1H, CD), 39.1 (1H, CF), 49.1 (1H, C10), 54.0−59.0 (1H, C7), 55.6 (1H, C9), 66.9 (1H, CA), 110.7 (2H, C3 + C6), 114.6 (2H, C6 + C3), 122.0 (2H, C5 + C2), 126.5 + 127.0 (2H, C2 + C5), 150.1 (1H, C1), 151.1 (1H, C4) ppm.

Conversion of Precursor Polymer 9 to OC_1C_{10} (10). The conversion of the precursor polymer to conjugated polymer is carried out in refluxing toluene (110 °C) for 3 h under nitrogen atmosphere. A yield of 3.6 g of conjugated polymer was obtained after precipitation in methanol. Molecular weights according to SEC (polystyrene standards): $M_p = 4.42 \times 10^5$; $M_w = 5.60 \times 10^5$; $M_n = 1.47 \times 10^5$; $D = 3.80$. IR (NaCl): ν 3060, 2947, 2919, 2862, 1501, 1459, 1417, 1381, 1353, 1261, 1191, 1043, 965, 859, 761, 690 cm^{-1}. ^1H and ^{13}C NMR data were as described in Table 2.

V. Summary

Advantages and drawbacks of both OC1C10 obtained by two different methods of synthesis are summarized in Table 10.

The new nonionic polymerization seems to give better results than the classical dehydrohalogenation method in terms of polymerization yield (98% against 52%), monomer concentration used in the polymerization reaction (0.15 vs 0.015 M), and solvent (butanol vs toluene, dioxane, or THF). On the other hand, the nonionic polymerization leads to polymers having better solubility and viscosity characteristics due to a better control of the molecular structure, so that no side reaction occurs. Nevertheless, this control in structural regularity introduces some drawbacks for LED application. Gelation temperature increases with structural regularity, and the processability is decreased with a more difficult spin-coating of the polymer. Second, the electroluminescent efficiency of the more regular polymer can be reduced. As for OC1C10-Gilch, variations at the molecular level (for instance structure defects) can act to trap excitons, hindering their migration to quenching sites, and so these materials give strongly enhanced quantum yields for electroluminescence.

Table 10. Qualitative Comparison between OC1C10 Polymers Synthesized by Two Methods[a]

	OC1C10-Gilch	OC1C10-New
monomer concentration in polymerization		***
polymerization yield		***
structure control		***
solubility		***
PL_{eff}	***	***
EL_{eff}	***	
viscosity		***
gelation temperature	***	

[a] Asterisks indicate an advantage for this polymer.

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Supporting Information Available: Figures showing the spectral characterization of OC1C10-Gilch and OC1C10-New (IR−FT and ^1H and ^{13}C NMR spectra). This material is available free of charge via the Internet at http://pubs.acs.org.

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