

# Visible-Light-Induced Controlled/Living Radical Polymerization of Styrene with a Phenyl Seleno Group at One Terminal Chain End: 1-(Phenylseleno)ethyl Benzene as a Photoiniferter

K. Rathore,<sup>1</sup> K. Raghunatha Reddy,<sup>1</sup> N. S. Tomer,<sup>2</sup> S. M. Desai,<sup>1</sup> R. P. Singh<sup>1</sup>

<sup>1</sup>Polymer Chemistry Division, National Chemical Laboratory, Pune 411008, India

<sup>2</sup>Organic Chemistry (Synthesis) Division, National Chemical Laboratory, Pune 411008, India

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**ABSTRACT:** The photopolymerization of styrene with a well-defined molecular architecture and a low polydispersity index and with methyl and phenylseleno ( $-\text{SePh}$ ) groups at  $\alpha$ - and  $\omega$ -chain ends, respectively, was performed via a controlled/living radical polymerization with a new initiating system, 1-(phenylseleno)ethyl benzene/*tert*-butyl diphenyl (phenylseleno) silane, through the absorption of visible light at room temperature. A novel initiating living

radical polymerization was examined. The yield and number-average molecular weight ( $M_n$ ) of the resulting polymer increased with the reaction time. Furthermore, a linear relationship was found in a plot of  $M_n$  versus the polymer yield. These results indicated that this polymerization proceeded through a living radical mechanism. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 93: 348–355, 2004

## INTRODUCTION

Recently, several remarkable methodologies<sup>1–12</sup> have been developed to design macromolecules via controlled/living radical polymerization. Among them, the iniferter method, discovered by Otsu and coworkers,<sup>13–15</sup> is an effective way of synthesizing well-defined polymers<sup>16,17</sup> that do not polymerize via an ionic mechanism. An iniferter is an initiator that also functions as a chain-transfer radical terminator, a primary radical terminator, or both. The synthesis of end-functional polymers by living radical polymerization with an iniferter is requisite and convenient and hence is widely used.<sup>18,19</sup>

Organoselenium compounds are useful for synthetic chemistry<sup>20</sup> and photochemistry.<sup>21,22</sup> Kwon et al.<sup>23</sup> reported that photopolymerization in the presence of diphenyl diselenide (DPDS) afforded polystyrene carrying phenylseleno groups at both chain ends. The generation of  $[\text{PhSeSiR}_3] \cdot^-$  [where  $\text{PhSeSiR}_3$  is *tert*-butyl diphenyl (phenylseleno) silane] was envisioned to occur through one electron-reductive process involving 9,10-dimethoxyanthracene (DMA) as a

light-absorbing ( $>300$  nm) electron donor and ascorbic acid ( $\text{H}_2\text{A}$ ) as a coreductant.<sup>24</sup>

The objective of this invention is to provide a narrow-polydispersity polymer with photoinitiation at room temperature and controlled/living radical polymerization with visible light to avoid the use of a thermal free-radical initiator. Here we report a novel controlled/living radical polymerization by a photoelectron transfer (PET) process with 1-(phenylseleno)ethyl benzene (1-PESePh)/ $\text{PhSeSiR}_3$  as the initiating system, with methyl ( $-\text{CH}_3$ ) and phenylseleno ( $-\text{SePh}$ ) groups at the  $\alpha$ - and  $\omega$ -chain ends, respectively, of the resulting polystyrene.

## EXPERIMENTAL

### Materials

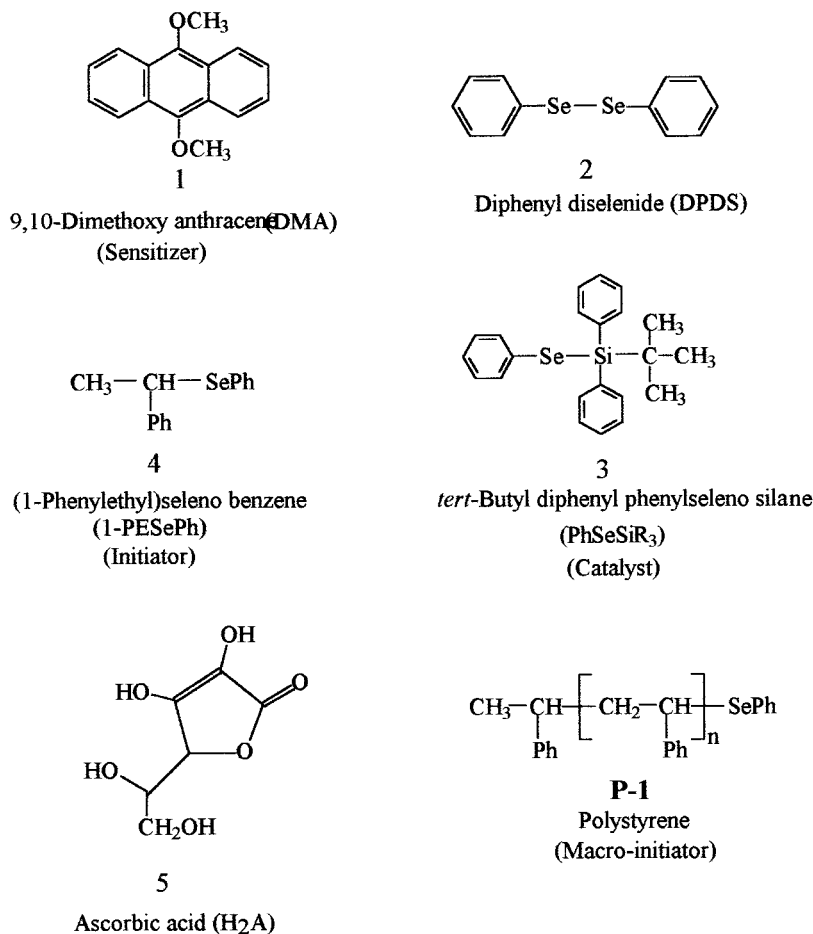
Styrene (Aldrich; analytical reagent) was purified by the usual method and was distilled in a stream of nitrogen before use. DMA,<sup>25</sup>  $\text{PhSeSiR}_3$ ,<sup>26</sup> DPDS,<sup>27</sup> and 1-PESePh<sup>28,29</sup> were synthesized according to literature procedures.<sup>30</sup> Tetrahydrofuran (THF; Merck) was distilled over sodium wire.  $\text{H}_2\text{A}$  (S.D. Fine Chemicals, India) and 1-(bromoethyl) benzene (Aldrich) were used as received. The structures of 1-PESePh, DMA,  $\text{PhSeSiR}_3$ , DPDS, and  $\text{H}_2\text{A}$  are shown in Scheme 1.

### Synthesis of 1-PESePh

A solution of DPDS (4.212 g, 0.0135 mol) in THF (20 mL; dried over sodium wire) was added to freshly

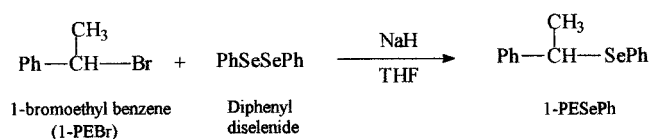
Correspondence to: R. P. Singh (singh@poly.ncl.res.in).

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Scheme 1 Structure of the initiator, sensitizer, and catalyst.

washed NaH [1.188 g (60%), 0.0297 mol] with dry petroleum ether, and the resulting mixture was refluxed for 5 h. The reaction mixture in the flask turned pale yellow or dirty white, and this indicated the generation of phenyl selenyl anions. 1-(Bromoethyl) benzene (5 g, 0.027 mol) was added to the solution, and the resulting mixture was allowed to reflux for 21 h (Scheme 2). The reaction mixture was cooled to room temperature, and ice-cooled water was added (10 mL) to it. The reaction mixture was extracted three times with petroleum ether, washed several times with cold water, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The distillation of the crude reaction mixture produced 5.51 g (78.1%) of 1-PESePh.



Scheme 2 Synthesis of 1-PESePh (initiator).

<sup>1</sup>H-NMR (CDCl<sub>3</sub>, δ): 7.3–7.57 (m, C<sub>6</sub>H<sub>5</sub>, 10H), 4.57 (q, CH, 1H), 1.87 (d, CH<sub>3</sub>, 3H; Fig. 1).

### Photopolymerization of styrene

The required amounts of the monomer (styrene), 1-PESePh, PhSeSiR<sub>3</sub>, DMA, H<sub>2</sub>A, and THF (Scheme 1) were charged into a Pyrex tube. The tube was degassed *in vacuo* by conventional freezing and thawing and was sealed off under an inert atmosphere. All polymerizations were carried out via irradiation with a 400-W, medium-pressure mercury vapor lamp at a distance of 8 cm at room temperature. The irradiation with the 400-W, medium-pressure mercury vapor lamp was performed in a specially designed double-walled photoreactor at room temperature. The first and outermost chamber contained a CuSO<sub>4</sub> · 5H<sub>2</sub>O/NH<sub>3</sub> filter solution. The filter solution allowed only 410-nm-wavelength light to pass through.<sup>31</sup> The light source was housed in a water-circulated, double-jacketed chamber; a 10-mm path length was maintained for the filter solution. The whole photoreactor was made of Pyrex glass (Fig. 2). After a given irradi-

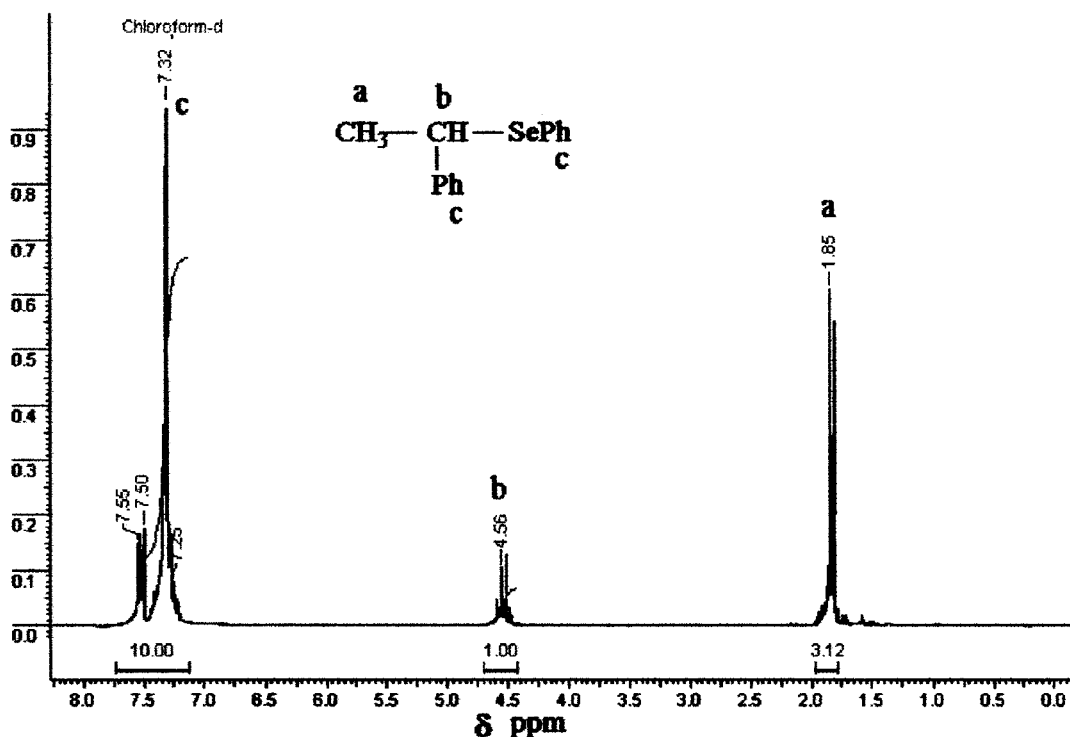


Figure 1  $^1\text{H-NMR}$  spectrum of 1-PSePh (3; 200 MHz in  $\text{CDCl}_3$ ).

ation time, the tube was opened, and the contents were poured into a large amount of methanol to precipitate the polymer. The resulting polymer was then purified by reprecipitation and dried *in vacuo* at approximately  $45^\circ\text{C}$  for 12 h; then, gel permeation chromatography (GPC) was performed.

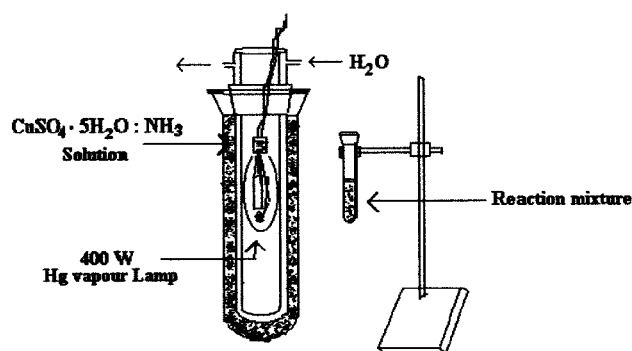


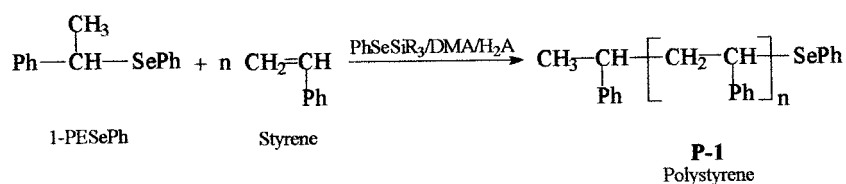
Figure 2 Irradiation setup for the photopolymerization of styrene.

### Photopolymerization of styrene with P-1 as a polymeric photoinverter

A solution of P-1 (0.2 g, 0.0356 mmol), styrene (1.5 mL, 13.635 g, 13.0916 mmol),  $\text{PhSeSiR}_3$  (0.0082 mmol), DMA (0.01022 mmol), and  $\text{H}_2\text{A}$  (0.0511 mmol) in THF (3 mL; Scheme 3) was irradiated with the 400-W, medium-pressure mercury vapor lamp in the aforementioned photoreactor at room temperature. At appropriate intervals, the reaction mixture was poured into methanol. The resulting polymer was dried *in vacuo* at  $45^\circ\text{C}$  for 12 h.

### Measurements

$^1\text{H-NMR}$  spectra were recorded on Bruker AC 200 and MSL 300 spectrometers in  $\text{CDCl}_3$  at  $25^\circ\text{C}$  with tetramethylsilane as an internal standard. The molecular weights and molecular weight distributions of the polymer samples were measured with a Waters Spectra AS 300 GPC instrument with  $\mu$ -Styragel columns



Scheme 3 Polymerization of styrene.

**TABLE I**  
Photopolymerization of Styrene at Various Monomer/  
Initiator/Catalyst Molar Ratios

Monomer/Initiator/ Catalyst Molar Ratio	Time (h)	Conversion (%)	$M_n$	$M_w/M_n$
200/1/1	20	29	5905	1.65
200/2/1	20	25	2550	1.32
450/2/1	20	30	6880	1.69
400/4/1	20	26	2610	1.29
600/4/1	20	29	4210	1.5
960/4/1	20	31	7475	1.71
800/10/1	20	28	2190	1.3
1200/10/1	20	33	4050	1.43
1600/10/1	20	35	5500	1.4

[DMA]/[H<sub>2</sub>A] = 1:5; [catalyst]/[DMA]/[H<sub>2</sub>A] = 1:1.25:6.25; [catalyst] = 0.0252 mmol in 25 mL of THF (i.e. [catalyst] = 0.001 mol/L).

(500, 10<sup>3</sup>, and 10<sup>4</sup> A°) with THF as an eluent. The calibration was performed with monodispersed polystyrene as the standard. The monomer conversion was determined with a PerkinElmer Auto System XL gas chromatograph with a 30-m DB-Wax column with toluene as an internal standard.

## RESULTS AND DISCUSSION

### Photopolymerization of styrene

The polymerization of styrene was carried out through the irradiation, with a 400-W, medium-pres-

**TABLE II**  
Photopolymerization of Styrene at a Monomer/Initiator/  
Catalyst Molar Ratio of 1600/10/1 for Different  
Irradiation Times

Time (h)	Conversion (%)	$M_n$ (th) $\times 10^{-3}$	$M_n$ (GPC) $\times 10^{-3}$	$M_w/M_n$
10	17	2830	2700	1.23
14	23	3827	3623	1.3
22.5	34.5	5740	5604	1.35
28.5	41.5	6905	6880	1.42
34	47	7820	7455	1.45
38.5	51.5	8569	8156	1.5
42	55	9152	8899	1.52
48.5	59.5	9900	9483	1.55
51.5	62	10316	10050	1.6

[Monomer]/[initiator]/[catalyst]/[DMA]/[H<sub>2</sub>A] = 1600:10:1:1.25:6.25; [Monomer] = 40.23 mmol; [Initiator] = 0.252 mmol; [DMA] = 0.0315 mmol; [H<sub>2</sub>A] = 0.1575 mmol. [catalyst] = 0.0252 mmol in 25 mL THF (i.e., [catalyst] = 0.001 mol/L).

sure mercury lamp, of a solution of styrene, 1-PeSePh, and PhSeSiR<sub>3</sub> (maximum wavelength = 410 nm) in a Pyrex glass photoreactor at room temperature. First, to obtain information on the initiation ability of the 1-PeSePh/PhSeSiR<sub>3</sub> system, we examined the polymerization at various monomer/initiator ([styrene]/[1-PeSePh]) concentrations, but the DMA/H<sub>2</sub>A molar ratios were kept constant (Table I). The polymerization proceeded smoothly at all concentrations. A higher molecular weight and a higher polydispersity

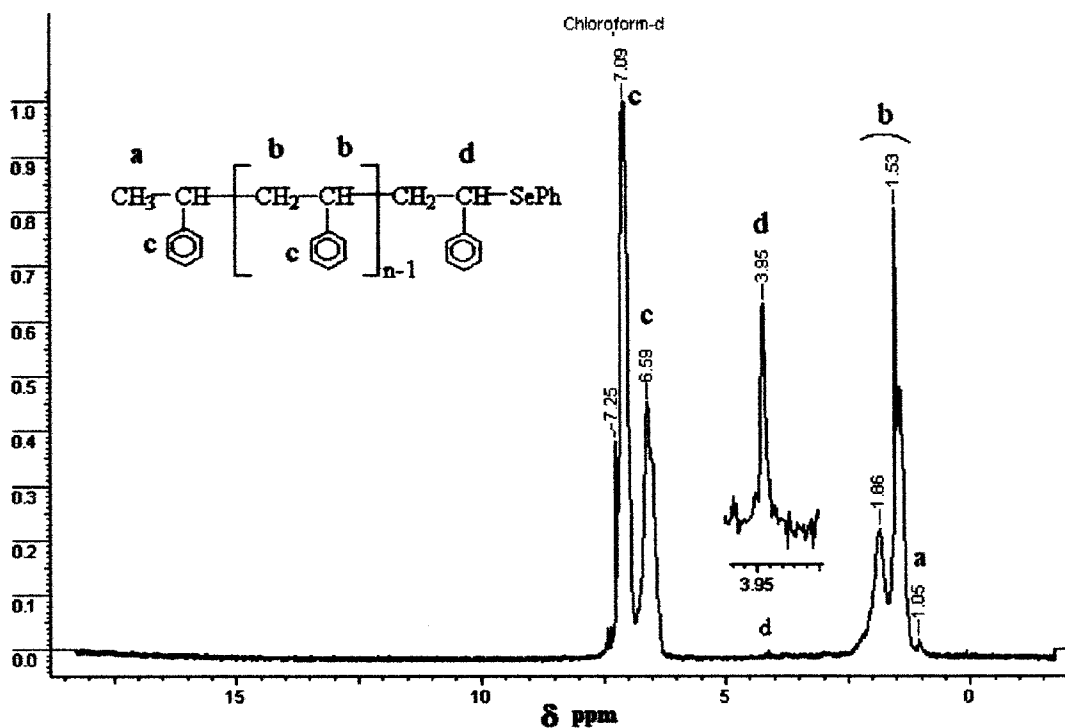


Figure 3 <sup>1</sup>H-NMR spectrum of polystyrene (200 MHz in CDCl<sub>3</sub>).

TABLE III  
Photopolymerization of Styrene with P-1  
as a Polymeric Photoiniferter

Conversion (%)	$M_n \times 10^{-3}$	$M_w/M_n$
0	5,605	1.35
8	8,350	1.4
13	10,150	1.42
21	13,050	1.47
31	17,100	1.6

[Monomer]/[P-1]/[catalyst]/[DMA]/[H<sub>2</sub>A] = 1600:4:1:1.25:6.25; [monomer] = 13.0916 mmol; [P-1] = 0.0356 mmol; [catalyst] = 0.0082 mmol; [DMA] = 0.0102 mmol; [H<sub>2</sub>A] = 0.0511 mmol.

index were observed in runs 1, 3, and 5 (Table I), whereas a moderate molecular weight and a narrow polydispersity index were observed in runs 5, 8, and 9 (Table I). A higher initiator/catalyst (1-PeSePh/PhSeSiR<sub>3</sub>) ratio produced a higher number-average molecular weight ( $M_n$ ) and a polydispersity, whereas a lower initiator/catalyst ratio produced polystyrene with a moderate  $M_n$  value and a lower polydispersity index. Furthermore, the polymer yield increased when the ratio of styrene to the initiating system increased. A similar finding was reported for tetraethylthiuram disulfide with a large transfer constant.<sup>32</sup> These results suggest that the catalyst not only worked as a photoinitiator but also functioned as a chain-transfer agent.<sup>33</sup>

The <sup>1</sup>H-NMR spectrum for run 9 is shown in Figure 3. The <sup>1</sup>H-NMR signals (CDCl<sub>3</sub>) at  $\delta = 7.2\text{--}6.4$  ppm ( $-\text{Ph}$ ),  $\delta = 3.95$  ppm [ $-\text{CH}(\text{Ph})-\text{SePh}$ ],  $\delta = 2.20\text{--}1.20$  ppm ( $-\text{CH}_2-\text{CH}-$ ), and  $\delta = 1.02$  ppm ( $-\text{CH}_3$ ) ex-

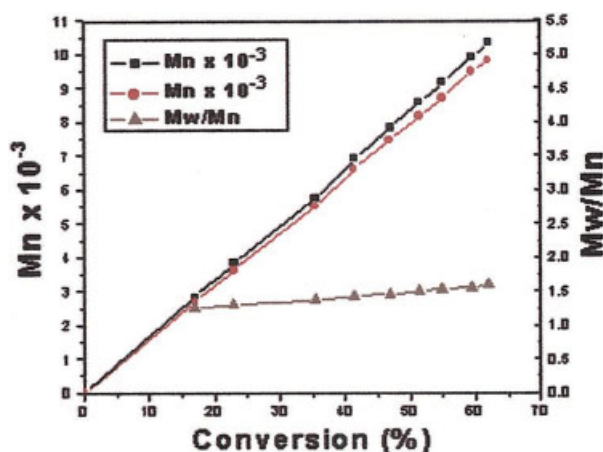


Figure 4  $M_n$ -conversion and  $M_w/M_n$ -conversion relationships for the photopolymerization of styrene ([monomer]/[initiator]/[catalyst]/[DMA]/[H<sub>2</sub>A] = 1600:10:1:1.25:6.25; [monomer] = 40.32 mmol; [initiator] = 0.252 mmol; [catalyst] = 0.0252 mmol; [DMA] = 0.0315 mmol; [H<sub>2</sub>A] = 0.1575 mmol).

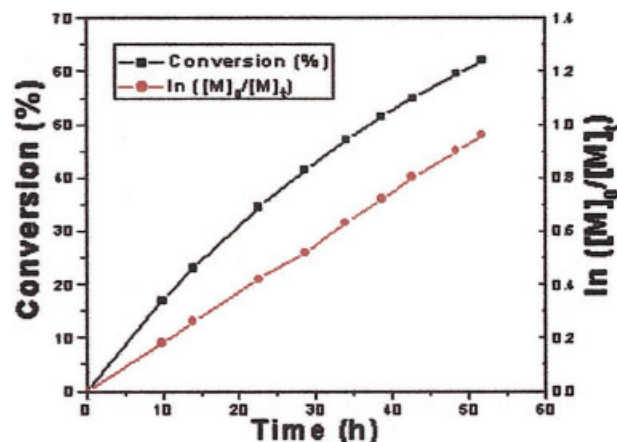


Figure 5 Time-conversion and time- $\ln([M]_0/[M]_t)$  relationships for the photopolymerization of styrene ([monomer]/[initiator]/[catalyst]/[DMA]/[H<sub>2</sub>A] = 1600:10:1:1.25:6.25 [monomer] = 40.32 mmol; [initiator] = 0.252 mmol; [catalyst] = 0.0252 mmol; [DMA] = 0.0315 mmol; [H<sub>2</sub>A] = 0.1575 mmol).

hibit the characteristic shifts. There is no signal corresponding to the protons of vinyl unsaturation ( $-\text{CH}_2=\text{CH}-$ ). These results indicate that the polystyrene chain has a methyl group at one end and a  $-\text{SePh}$  group at the other chain end. The signal of the  $-\text{CH}$  proton attaches to the  $-\text{SePh}$  group at 3.95 ppm,<sup>34</sup> but it is detectable only. This indicates that polystyrene synthesized by this method contains methyl and phenylseleno groups at the  $\alpha$ - and  $\omega$ -chain ends, respectively.

The results for the photopolymerization of styrene at a monomer/initiator/catalyst (styrene/1-PeSePh/PhSeSiR<sub>3</sub>) ratio of 1600/10/1, for different irradiation

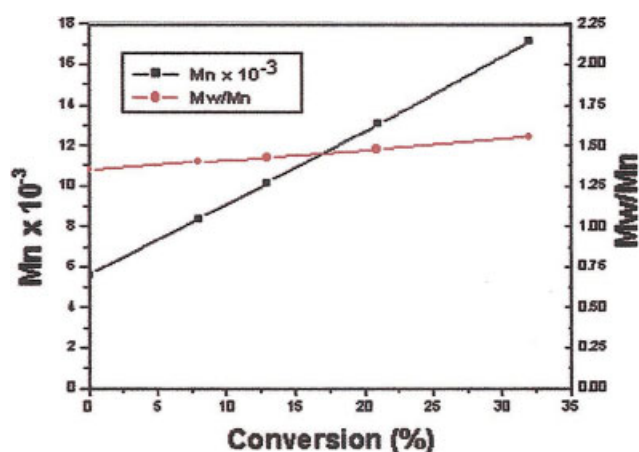


Figure 6  $M_n$ -conversion and  $M_w/M_n$ -conversion relationships for P-1 ( $M_n = 5605$ ,  $M_w/M_n = 1.35$ ) as a polymeric photoiniferter ([monomer]/[P-1]/[catalyst]/[DMA]/[H<sub>2</sub>A] = 1600:4:1:1.25:6.25; [monomer] = 13.0916 mmol; [P-1] = 0.0356 mmol; [catalyst] = 0.0082 mmol; [DMA] = 0.0102 mmol; [H<sub>2</sub>A] = 0.0511 mmol).

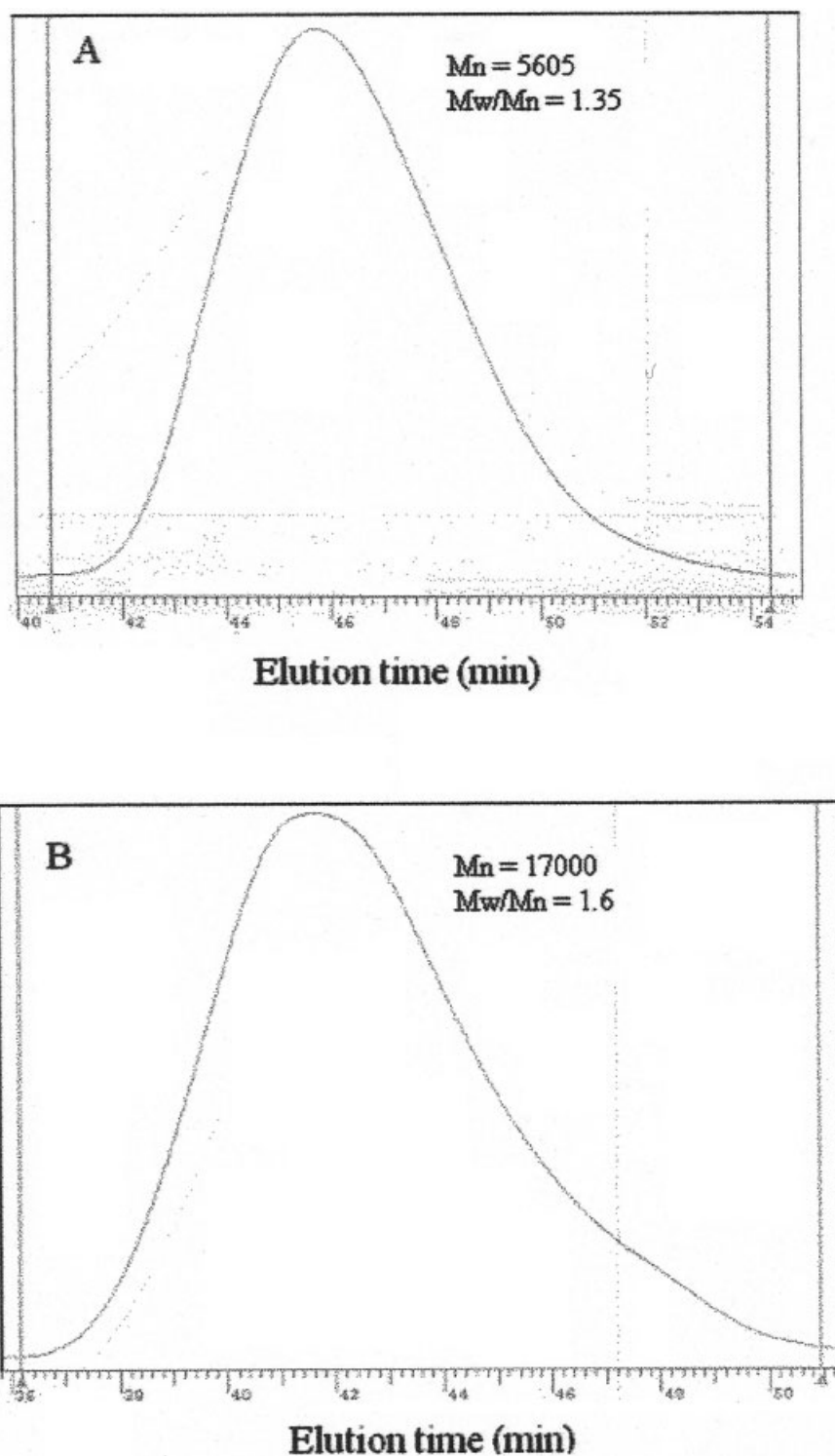
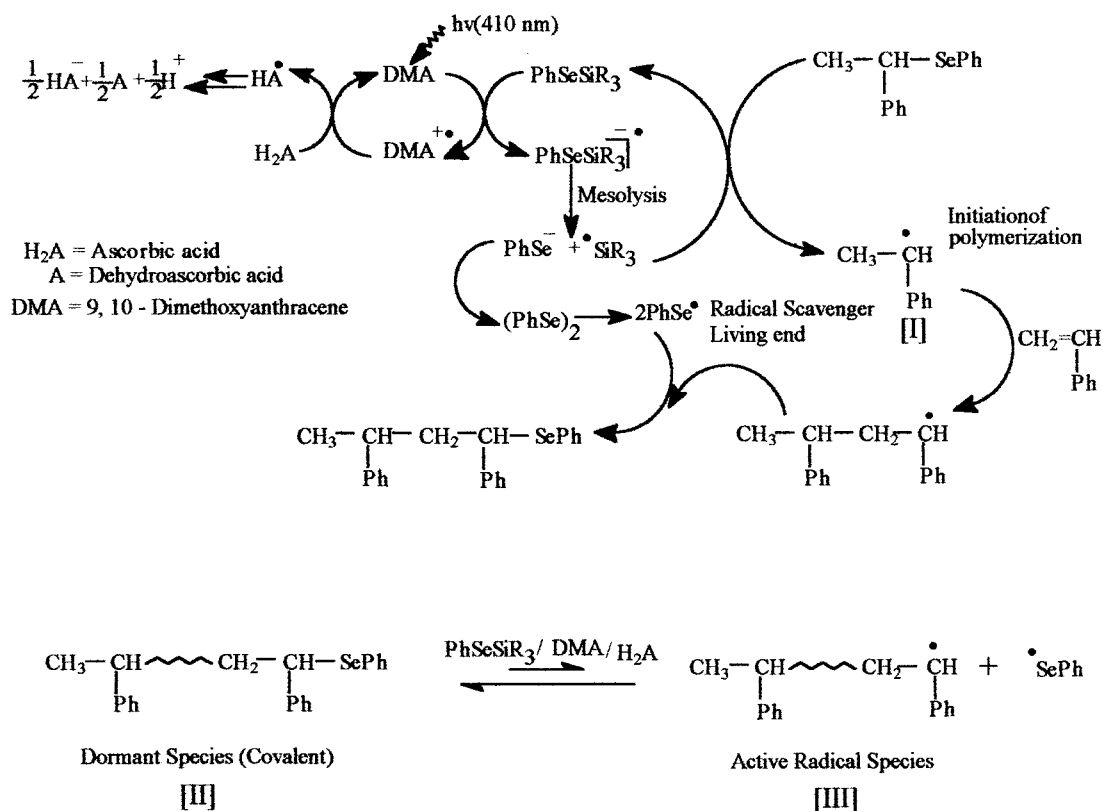


Figure 7 GPC curves for the photopolymerization of styrene (a) before and (b) after chain extension.

times, are compiled in Table II. Here we took samples at different intervals and poured them into an excess of methanol. Figure 4 shows that  $M_n$  increased linearly with the conversion from 2700 to 10,050 and that the polydispersity index rose from 1.23 to 1.60. The GPC-determined number-average molecular weight

( $M_{n(GPC)}$ ) was close to a theoretical number-average molecular weight ( $M_{n(th)}$ ) computed as follows:  $M_{n(th)} = ([St]_0/[1-PESePh]_0) \times M_{w(St)} \times \text{conversion}$  (where  $M_{w(St)}$  is the weight-average molecular weight of styrene). The efficiency of the initiator ( $f = M_{n(th)}/M_{n(GPC)}$ ) was around 1.0. A plot of  $\ln([M]_0/[M]_t)$  ver-



**Scheme 4** Mechanism of the photopolymerization of styrene with 1-PESePh/PhSeSiR<sub>3</sub> as an initiating system.

sus time  $t$  is shown in Figure 5. A straight line can be observed, indicating that the kinetics were first-order in the monomer conversion. From this figure, the  $M_n$  values of the resulting polymers were also found to increase with the reaction time. This means that the concentration of the propagating radical was constant during the polymerization. From these results, it is clear that the styrene polymerization with the 1-PESePh/PhSeSiR<sub>3</sub> initiating system at room temperature proceeded in a controlled/living manner as expected. However, the polydispersities [number-average molecular weight/number-average molecular weight ( $M_w/M_n$ )] were 1.23–1.60, similar to the values obtained from living radical polymerization with sulfur compounds by Otsu and Yoshida.<sup>13</sup> Therefore, the polymers produced with the initiation system were well defined, having not only a narrow polydispersity index but also precise end groups, that is,  $\alpha$ -methyl and  $\omega$ -phenylseleno groups. The presence of an  $\omega$ -phenylseleno end group in the obtained polystyrene suggested that the polymerization proceeded via a controlled/living radical polymerization process.

#### Chain extension of polystyrene

To confirm the living nature of the system, we performed chain-extension experiments under fixed conditions. Styrene (1.5 mL, 1.3635 g, 13.0916 mmol), P-1

(0.2 g, 0.0356 mmol;  $M_n = 5605$ ,  $M_w/M_n = 1.35$ ; run 3, Table II), PhSeSiR<sub>3</sub> (0.0082 mmol), DMA (0.01022 mmol), and H<sub>2</sub>A (0.0511 mmol) in THF (3 mL) were mixed together at room temperature. After 50 h of irradiation of this solution, a conversion of the styrene monomer of 32% was achieved. As shown in Figure 6 (at 0% conversion and  $M_n = 5605$ , i.e., P-1, the polymer obtained in run 3, Table II), the polymer yield and  $M_n$  increased with the irradiation time. These results show that polystyrene with a seleno group at the  $\omega$ -chain end effectively worked as a polymeric photoiniferter. The molecular weight ( $M_n$ ) increased linearly from 5605 to 17,100, and the polydispersity index slightly increased from 1.35 to 1.60. The increase in  $M_n$  is clearly demonstrated in the GPC curves shown in Figure 7. This suggests that the active species were possibly radicals. Furthermore, from the weight of the resulting polymer and  $M_n$ , we found that the number of polymer molecules through the polymerization reaction was almost unchanged.

#### Polymerization mechanism

On the basis of the aforementioned results, we propose a mechanism for the controlled/living radical polymerization (Scheme 4). The potential of PhSeSiR<sub>3</sub> to initiate radical chain sequences has been considered in light of the expected dissociation of the radical

anion ( $[\text{PhSeSiR}_3] \cdot^-$ ) into the silyl radical ( $\cdot \text{SiR}_3$ ), which is useful for chain initiation, and  $\text{PhSeSePh}$ , which is formed after the oxidative dimerization of the corresponding  $\text{PhSe}^-$  anion and also participates in the termination step.<sup>35</sup>

The efficient generation of the silyl radical ( $\cdot \text{SiR}_3$ ) and phenylselenide anion ( $\text{PhSe}^-$ ) by the mesolysis<sup>36-38</sup> of  $[\text{PhSeSiR}_3] \cdot^-$  by the visible-light (410 nm)-initiated PET activation of  $\text{PhSeSiR}_3$  provided us a unique opportunity for radical chain initiation and group-transfer reactions.  $\cdot \text{SiR}_3$  abstracts  $\cdot \text{SePh}$  from 1-PESePh and forms a new free radical (**I**; Scheme 4). Thus, 1-PESePh acts as an initiator. The styrene monomer is inserted into this new radical and thus produces a polymer, as shown in Scheme 4. The propagation takes place through the addition of the resulting carbon radical to styrene. The coupling of the polymer radical with the stable seleno radical terminates this polymerization. There is dynamic equilibrium between the active radical species (**III**) and dormant species (**II**; Scheme 4).

## CONCLUSIONS

Well-defined polystyrene with  $\alpha$ -methyl and  $\omega$ -phenylseleno ( $-\text{SePh}$ ) end groups, a medium  $M_n$  value, and a narrow polydispersity was synthesized with a new initiating system consisting of 1-PESePh and  $\text{PhSeSiR}_3$ .  $\text{PhSeSiR}_3$  functioned as a photoiniferter for the living polymerization of styrene. Because the polymer chain was end-functionalized by the seleno group, it could be used as a polymeric photoiniferter, which could undergo chain extension via a conventional controlled/living radical polymerization process.

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