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## Liquid Crystals

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# New liquid crystal polymers with 1,3-dithiane or 1,3-dioxane rings

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New polymerizable liquid crystal materials: 10-{4-[5-alkyl-1,3-dithian-2-yl]phenoxy}decylmethacrylates (**10**) and 10-{4-[5-alkyl-1,3-dioxan-2-yl]phenoxy}decylmethacrylates (**11**) were synthesized and converted into polymers. The mesomorphic behaviour of these compounds was measured. Though the 1,3-dithiane-type monomer did not exhibit any liquid crystal phases, the corresponding polymer exhibited the schlieren texture of a nematic liquid crystal phase at around room temperature (for example **11-1**: G 30 N 76 I). To exhibit a nematic liquid crystal phase at around room temperature is a principal feature of these 1,3-dithiane type polymer. The corresponding 1,3-dioxane-type polymer exhibited the focal conic fan textures of a smectic A phase (for example **10-1**: Cr80 S<sub>A</sub> 101 I).

## 1. Introduction

For the last fifteen years, 2,5-disubstituted-1,3-dithianes, 1,3-oxathianes and 1,3-dioxanes have been reported as new types of liquid crystal materials [1–16]. From many years new liquid crystal polymers have been developed as functional materials and various liquid crystal polymers have been synthesized. However, dithiane-type liquid crystal polymers have not been reported. In a previous letter [16] we reported a new liquid crystal polymer with a 1,3-dithiane ring. This paper deals with six monomers and the corresponding polymers of 1,3-dithiane or 1,3-dioxane compounds. Some of the 1,3-dithiane type polymers exhibited a nematic phase at around room temperature. This is a special property, because most of liquid crystal polymers are nematic only at higher temperature [17].

In this paper, we wish to report the syntheses and the mesomorphic behaviour of these compounds in detail.

## 2. Results and discussion

10-{4-[5-Alkyl-1, 3-dithian-2-yl]phenoxy}decylmethacrylates (**10**) and 10-{4-[5-alkyl-1,3-dioxan-2-yl]phenoxy}decylmethacrylates (**11**) were synthesized by the route shown in figure 1.

In the bromination step of diol (**3**) both the mono and di-bromides were produced, however, separation of them was not carried out in this step. Thus, in the syntheses of mercaptane, a mixture of mono and

di-bromide was used. Compound (**5**) could be purified by column chromatography, that is, (**5**) and (**5'**) eluted in a hexane fraction and ether fraction, respectively.

Compound (**9**) was synthesized from 1,10-dibromodecane and methacrylic acid.

In the syntheses of compounds (**7**) and (**8**), both *trans* and *cis*-isomers were produced, respectively. Therefore, (**7**) and (**8**), were purified by column chromatography (Wakogel C-300, benzene:ether=5:1) and repeated recrystallizations (ether:hexane=2:1), respectively.

In the <sup>1</sup>H NMR data for the compounds (**7**), C-2 proton signals of the 1,3-dioxane ring for the *trans* and *cis*-isomers appeared at  $\delta=5.45$  and 5.50 ppm, respectively. Therefore, the removal of *cis*-isomer could be checked by the disappearance of this *cis*-isomer peak. In the same way, in the <sup>1</sup>H NMR data for compound (**8**), C-2 proton signals of the 1,3-dithiane ring for the *trans* and *cis*-isomers appeared at  $\delta=5.20$  and 5.15 ppm, respectively. Therefore, the removal of *cis*-isomer could be checked in the same manner as for monomers (**7**).

Side chain polymers were synthesized by the free radical polymerization of monomers (**10**) and (**11**). The number and weight average molecular weights ( $M_n$  and  $M_w$ ) were determined using a GPC system, indicating the production of polymers.

Measurement of transition temperatures and assignment of the mesophases were carried out by means of a micro-melting point apparatus equipped with polarizers, a differential scanning calorimeter and X-ray diffraction. Phase identification was made by comparing the

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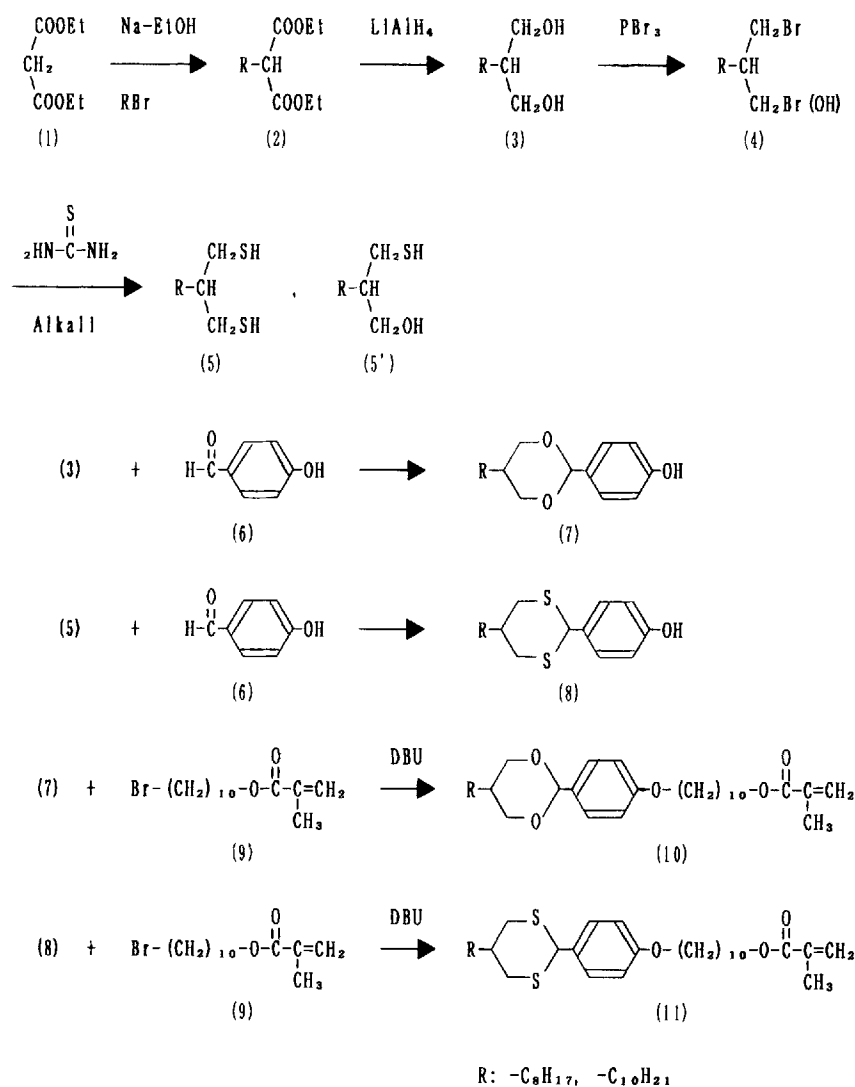


Figure 1. Synthetic pathway for compound (10) and (11).

observed textures with those in the literature [18, 19], and via X-ray diffraction. Phase transition temperatures for monomers (10) and (11), the corresponding polymers (10) and (11), and related compounds are given in tables 1 and 2.

Though polymers 12 and 13 exhibit the nematic phase, the corresponding 1,3-dithiane polymer 14 did not exhibit any liquid crystal phases. These facts may indicate that the 1,3-dithiane-type polymer is less suitable in enhancing liquid crystallinity than 1,3-dioxane or 1,3-oxathiane-type polymers. Therefore, polymers 10 having alkyl chain spacers were synthesized and these polymers exhibited the schlieren texture of the nematic liquid crystal phase. The result of X-ray diffraction also supports this result, that is, there is a very broad peak at around 20°, but no peaks are seen at the small angle as shown in figure 2.

Though polymers 10 and 11 have long alkyl spacers which connect the principal chain and mesogenic side groups, respectively, the corresponding polymers 12 and 14 do not. This long alkyl spacer seems to affect the mesomorphic behaviour, that is, in the 1,3-dioxane-type polymer, polymer 10-1 having a long alkyl spacer exhibited the smectic A phase, but the corresponding polymer 12 exhibited the nematic phase. In the 1,3-dithiane-type polymer, polymer 11-1 having a long alkyl spacer exhibits the nematic phase, but the corresponding polymer 14 did not exhibit any liquid crystal phases. These results imply that long alkyl spacers order mesogenic side groups by their dispersion force.

Though monomers 10 exhibited the nematic phase, the corresponding monomers 11 did not exhibit any liquid crystal phases. Polymers 10 exhibit the  $S_A$  phase, but the corresponding polymers 11 exhibit the nematic phase.

Table 1. Transition temperatures for monomers **10**, **11**, **13**, **14** and **15**.

$$\text{R}-\text{C}_4\text{H}_6\text{X}_2-\text{C}_6\text{H}_4-\text{O}-(\text{CH}_2)_{10}-\text{O}-\text{C}(=\text{O})-\text{C}(\text{CH}_3)=\text{CH}_2 \quad (10, 11)$$

Compound	R	X	Y	Phase transition temperatures/ $^{\circ}\text{C}^a$
<b>10-1</b>	$\text{C}_8\text{H}_{17}$	O	O	
<b>10-2</b>	$\text{C}_{10}\text{H}_{21}$	O	O	
<b>11-1</b>	$\text{C}_8\text{H}_{17}$	S	S	
<b>11-2</b>	$\text{C}_{10}\text{H}_{21}$	S	S	

$$\text{C}_8\text{H}_{17}-\text{C}_4\text{H}_6\text{X}_2-\text{C}_6\text{H}_4-\text{O}-\text{C}(=\text{O})-\text{C}(\text{CH}_3)=\text{CH}_2 \quad (13, 14, 15)$$

Compound	X	Y	Phase transition temperature/ $^{\circ}\text{C}$
<b>13</b>	O	O	
<b>14</b>	S	O	
<b>15</b>	S	S	

<sup>a</sup> Cr: Crystal, N: Nematic, S: Smectic, I: Isotropic.

Monomers **10** and polymers **10** are 1,3-dithiane-type compounds and have two sulphur atoms instead of two oxygen atoms in monomer **11** and polymer **11**. Sulphur atoms are bigger than oxygen atoms, therefore, the molecular width of monomer **11** and the width of mesogenic side group of polymer **11** are greater than those of monomer **10** and polymer **10**, respectively. This may cause the results given above.

The principal features of the mesomorphic behaviour of 1,3-dithiane-type liquid crystal polymers are to exhibit the nematic liquid crystal phase at around room temperature, and lower the nematic-isotropic transition

temperatures (**11-1**:  $76^{\circ}\text{C}$ , **11-2**:  $81^{\circ}\text{C}$ ) than those of ordinary methacrylate type liquid crystal polymers having alkoxy-type side chains [17].

### 3. Experimental

#### 3.1. Analysis

IR,  $^1\text{H}$  NMR and mass spectra were obtained with a Hitachi 215 spectrometer, a JNM-PMX 60 spectrometer and a Hitachi M-80B spectrometer, respectively. Elemental analyses were carried out with a Perkin-Elmer 250 instrument. The transition temperatures and mesomorphic phases were determined by means of a

Table 2. Transition temperatures for polymers **10**, **11**, **13**, **14** and **15**.

Compound	X	Y	Phase transition temperatures/°C <sup>a</sup>	$M_n^b$	$M_w^b$
<b>10-1</b>	O	O	Cr $\xrightarrow{80}$ S <sub>A</sub> $\xrightarrow{101}$ I	25 000	71 000
<b>10-2</b>	O	O	Cr $\xrightarrow{85}$ S <sub>A</sub> $\xrightarrow{105}$ I	31 000	81 000
<b>11-1</b>	S	S	Cr $\xrightarrow{30}$ N $\xrightarrow{76}$ I	18 000	39 000
<b>11-2</b>	S	S	Cr $\xrightarrow{21}$ N $\xrightarrow{81}$ I	27 000	63 000

<b>12</b>	O	O	G $\xrightarrow{79}$ N $\xrightarrow{164}$ I	33 000	85 000
<b>13</b>	S	O	G $\xrightarrow{81}$ N $\xrightarrow{140}$ I	21 900	96 000
<b>14</b>	S	S	Cr $\xrightarrow{250}$ (dec.)	21 600	60 700

<sup>a</sup> C: Crystal, N: Nematic, S: Smectic, I: Isotropic, G: Glass, dec.: decomposed. <sup>b</sup>  $M_n$ : number average molecular weight.  $M_w$ : weight average molecular weight.  $M_n$  and  $M_w$  were determined using a Toso high speed GPC system (solvent: THF, standard: polystyrene).

Mitamura Riken micro-melting point apparatus equipped with polarizers and a Mettler DSC 20 system, respectively. The number and weight average molecular weight ( $M_n$  and  $M_w$ ) were determined using a Toso high speed GPC system HLC 8020 (solvent: THF, standard: polystyrene). X-ray diffraction was performed with a Rigaku X-ray Rad 2B system using Ni-filtered  $\text{CuK}\alpha$  radiation.

### 3.2. Synthesis

#### 3.2.1. 2-Alkyl-1,3-propanedithiol (**5**)

To a solution of thiourea (0.2 mol) in triethyleneglycol (20 ml) kept at 75°C was added compound **4** (about 0.05 mol) in a nitrogen atmosphere, followed by stirring at 75°C for 18 h. Tetraethylenepentamine (0.05 mol) was then added, and the mixture was stirred at 75°C for 2 h

under a nitrogen atmosphere. The reaction mixture was then poured into ice water (300 g), and extracted twice with ether (each 300 ml). The extract was dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated on an evaporator at 30°C. The separation of compounds **5** and **5'** was carried out by column chromatography (Wakogel C-300). Compound **5** effused in a hexane fraction. Compound **5** was a yellow transparent liquid. IR ( $\text{CHCl}_3$ ), 2800–3000 (alkyl).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.6–2.0 (m, R-CH, SH), 2.5–2.9 (m, 4 H,  $-\text{CH}_2\text{S}$ ).

#### 3.2.2. 5-Alkyl-2-(*p*-hydroxyphenyl)-1,3-dioxane (**7**)

To a solution of compound **3** (0.01 mol) and *p*-hydroxybenzaldehyde (0.01 mol) in anhydrous  $\text{CHCl}_3$  (200 ml) cooled in an ice bath were added  $\text{BF}_3 \cdot (\text{C}_2\text{H}_5)_2\text{O}$  (0.5 g) and molecular sieves (3 A, 1/15; 3 g). The mixture

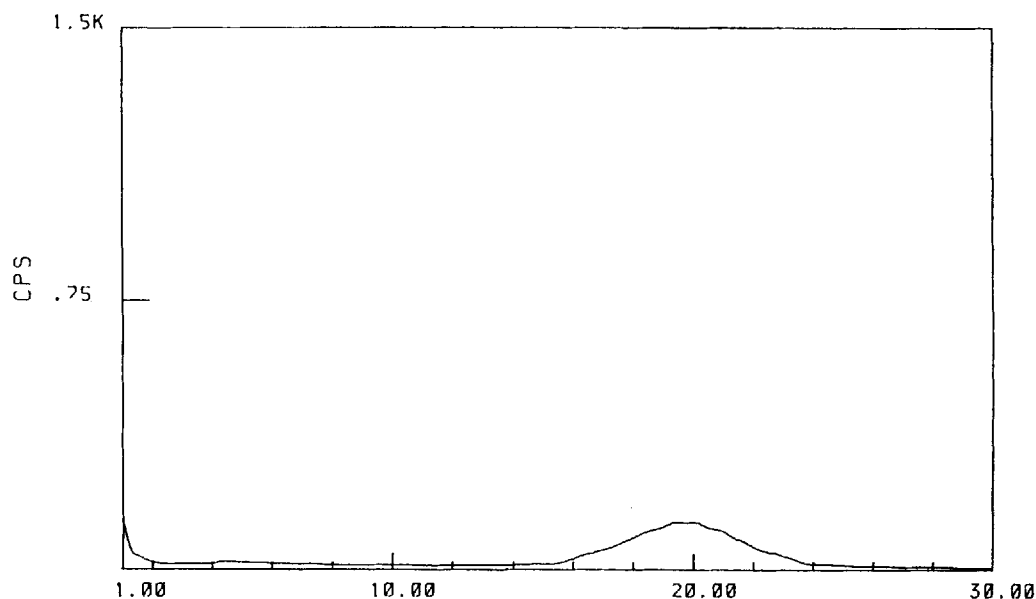


Figure 2. X-ray diffraction pattern of liquid crystal polymer 11-2.

was stirred at 0–5°C for 8 h and then at 20°C for 15 h. The solution was first washed with 10 per cent aq.  $\text{NaHCO}_3$  (400 ml), dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and evaporated *in vacuo* at 40°C. The crude product was purified by column chromatography (Wakogel C-300, benzene:ether=5:1), and repeatedly recrystallized (ether:hexane=2:1), respectively. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 3600 (OH), 2800–3000 (alkyl), 1605 (Ar), 1170 (ether).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.5–2.0 (m, R-CH), 3.5–4.5 (m, 4 H,  $\text{CH}_2\text{O}$ ), 5.45 (s, 1 H, HcOO), 6.7–7.6 (q, 4 H, ArH), 9.1 (s, 1 H, OH).

### 3.2.3. 5-Alkyl-2-(*p*-hydroxyphenyl)-1,3-dithiane (8)

The same procedure as that for compound 7 was used for the reaction of compounds 5 and 6. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 3600 (OH), 2800–3000 (alkyl), 1605 (Ar), 1170 (ether).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.7–2.0 (m, R-CH), 2.7–3.0 (m, 4 H,  $\text{CH}_2\text{S}$ ), 5.20 (s, 1 H, HCSS), 6.8–7.6 (q, 4 H, ArH), 9.2 (s, 1 H, OH).

### 3.2.4. 10-[4-(5-Alkyl-1,3-dioxan-2-yl)phenoxy]decyl methacrylate (10)

A solution of compound 7 (0.005 mol) and compound 9 (0.005 mol) and 1,8-diazabicyclo [5. 4. 0]undec-7-ene (0.03 mol) and phenothiazine (0.1 g) in anhydrous DMF (50 ml) was stirred at 30°C for 18 h under a nitrogen atmosphere. The solution was poured into ice water and extracted twice with ether (200 ml). The extract was washed, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and evaporated *in vacuo* at 30°C. The crude product was purified by column chromatography and recrystallized from hexane. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 2800–3000

(alkyl), 1720 (C=O), 1605 (Ar), 1170 (ether), 895 (C=CH<sub>2</sub>).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.5–2.0 (m, R-CH, O- $\text{CH}_2\text{C}_8\text{H}_{16}\text{CH}_2\text{-O}$ ), 3.3–4.5 (m, 8 H,  $\text{CH}_2\text{O}$ ), 5.45 (s, 1 H, HcOO), 5.6, 6.2 (d, 2 H, - $\text{CH}_2$ ), 6.7–7.6 (q, 4 H, ArH).

10-1: Yield, 35 per cent. Found: C, 73.60; H, 10.25 per cent. Calcd for  $\text{C}_{32}\text{H}_{52}\text{O}_5$ : C, 74.37; H, 10.14 per cent. Mass 516 ( $\text{M}^+$ ).

10-2: Yield, 30 per cent. Found: C, 73.57; H, 10.86 per cent. Calcd for  $\text{C}_{34}\text{H}_{56}\text{O}_5$ : C, 74.95; H, 10.36 per cent. Mass 544 ( $\text{M}^+$ ).

### 3.2.5. 10-[4-(5-Alkyl-1,3-dithian-2-yl)phenoxy]decyl methacrylate (11)

The same procedure as that for compound 10 was used for the reaction of compounds 8 and 9. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 2800–3000 (alkyl), 1720 (C=O), 1605 (Ar), 1170 (ether), 895 (C=CH<sub>2</sub>).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.5–2.0 (m, R-CH, O- $\text{CH}_2\text{C}_8\text{H}_{16}\text{CH}_2\text{-O}$ ), 2.5–3.0 (m, 4 H,  $\text{CH}_2\text{S}$ ), 3.9–4.4 (m, 4 H,  $\text{CH}_2\text{O}$ ), 5.20 (s, 1 H, HcSS), 5.6, 6.2 (d, 2 H, = $\text{CH}_2$ ), 6.8–7.6 (q, 4 H, ArH).

11-1: Yield, 26 per cent. Found: C, 69.54; H, 9.68 per cent. Calcd for  $\text{C}_{32}\text{H}_{52}\text{O}_3\text{S}_2$ : C, 70.02; H, 9.55 per cent. Mass 548 ( $\text{M}^+$ ).

11-2: Yield, 31 per cent. Found: C, 70.96; H, 9.75 per cent. Calcd for  $\text{C}_{34}\text{H}_{56}\text{O}_3\text{S}_2$ : C, 70.78; H, 9.78 per cent. Mass 576 ( $\text{M}^+$ ).

### 3.2.6. 4-(5-Alkyl-1,3-dioxan-2-yl) phenyl methacrylate (12)

Compound 12 was synthesized by the acetalization of compound 3 and *p*-formylphenyl methacrylate. The

crude product was purified by column chromatography and recrystallizations from hexane. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 2950–2800 (alkyl), 1750 (C=O), 1610 (Ar), 1170 (ether), 895 (C=CH<sub>2</sub>). <sup>1</sup>H NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.7–2.5 (m, R-CH, CH<sub>3</sub>), 3.3–4.4 (m, 4H, CH<sub>2</sub>O), 5.45 (s, 1H, HcOO), 5.8, 6.4 (d, 2H, =CH<sub>2</sub>), 7.0–7.7 (q, 4H, ArH). Yield, 42 per cent. Found: C, 73.76; H, 9.81 per cent. Calcd for C<sub>22</sub>H<sub>32</sub>O<sub>4</sub>: C, 73.30; H, 8.95 per cent. Mass 360 (M<sup>+</sup>).

### 3.2.7. 4-(5-Alkyl-1,3-oxathian-2-yl)phenyl methacrylate (13)

The same procedure as that for compound **12** was used for the reaction of compound 5' and p-formylphenyl methacrylate. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 2950–2800 (alkyl), 1750 (C=O), 1610 (Ar), 1170 (ether), 895 (C=CH<sub>2</sub>). <sup>1</sup>H NMR ( $\text{CDCl}_3$ ,  $\delta$ ), 0.7–2.2 (m, R-CH, CH<sub>3</sub>), 2.7–2.9 (d, 2H, CH<sub>2</sub>S), 3.2–4.4 (m, 2H, CH<sub>2</sub>O), 5.75 (s, 1H, HcOS), 5.6, 6.2 (d, 2H, =CH<sub>2</sub>), 7.0–7.7 (q, 4H, ArH). Yield, 48 per cent. Found: C, 70.16; H, 8.57 per cent. Calcd for C<sub>22</sub>H<sub>32</sub>O<sub>3</sub>S: C, 70.17; H, 8.57 per cent. Mass 376 (M<sup>+</sup>).

### 3.2.8. 4-(5-Alkyl-1,3-dithian-2-yl)phenyl methacrylate (14)

The same procedure as that for compound **12** was used for the reaction of compound **5** and p-formylphenyl methacrylate. A white powder was obtained. IR ( $\text{CHCl}_3$ ), 2950–2800 (alkyl), 1750 (C=O), 1610 (Ar), 895 (C=CH<sub>2</sub>). <sup>1</sup>H NMR ( $\text{CDCl}_3$ ,  $\delta$ ) 0.7–2.3 (m, R-CH, CH<sub>3</sub>), 2.7–3.0 (m, 4H, CH<sub>2</sub>S), 5.2 (s, 1H, HcSS), 5.8, 6.4 (d, 2H, =CH<sub>2</sub>), 7.0–7.7 (q, 4H, ArH). Yield, 15 per cent. Found: C, 67.05; H, 8.25 per cent. Calcd for C<sub>22</sub>H<sub>32</sub>O<sub>2</sub>S<sub>2</sub>: C, 67.30; H, 8.22 per cent. Mass 392 (M<sup>+</sup>).

## 3.3. Syntheses of polymers

Polymers of compounds **10**, **11**, **12**, **13** and **14** were synthesized by the free radical polymerization of the corresponding monomers (0.5 mol l<sup>-1</sup>) and AIBN (0.02 mol l<sup>-1</sup>) as initiator in dioxane (2 ml) at 70°C

for 72 h, deaerated ampules being employed. As the monomers were soluble in hexane, the polymers were obtained by reprecipitation with hexane. A reprecipitation from tetrahydrofuran solution was also carried out.

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