

# Synthesis and Characterization of New Aliphatic Polyesters Containing Methanodinaphtho[1,3]dioxocin Part

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**ABSTRACT:** A novel dinaphthodioxocin-containing diol monomer, 8,16-methano-16H-dinaphtho[2,1-d:1',2'-g][1,3]dioxocin-2,14-diol (MDDD) was synthesized in high yield and purity through the condensation of 2,7-dihydroxynaphthalene with malonaldehydetetramethyl acetal at room temperature. It is used as a new monomer for the synthesis of a series of novel dinaphthodioxocin-containing aliphatic polyesters by interfacial polycondensation method. The new diol monomer was fully characterized by IR, NMR, mass spectroscopies, and elemental analysis. Optimal conditions for polyesterification were obtained via study of the model compounds. Polyesters derived

from MDDD and four aliphatic diacid chlorides had inherent viscosities ranging from 0.26 to 0.38 dL/g. The chemical structure of the polymers was fully characterized by IR, NMR spectroscopies, and elemental analysis. The physical properties of the polymers were studied. All the new polymers show good thermal stability and very good solubility in most organic solvents. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 3357–3362, 2011

**Key words:** polyesters; aliphatic diacid chlorides; interfacial polycondensation; solution polycondensation; adipoyl chloride; step-growth polycondensation

## INTRODUCTION

Polyesters are well known as high-performance engineering thermoplastics because of their good thermal stability and excellent mechanical properties.<sup>1,2</sup> The ordering and varying of backbone functions have profound effects on the final properties such as solubility and thermal characteristics of the resulting heterocyclic macromolecules.<sup>3</sup> The investigation of thermally stable linear polymer systems has received great emphasis due to requests for heat resistant materials such as laminates, films, and fibers. The introduction of heterocyclic and bulky (such as tetraphenylethylene, pyrrole, thiophene, and benzopinacolone) units into the polymer backbone has been explored as a convenient way to obtain polymers with good solubility behavior and thermal stability.<sup>4–11</sup> Polyesters containing heterocyclic and bulky units in the main chain possess excellent thermal stabilities. In our previous work, we demonstrated that dinaphthodioxocin units containing polymers exhibited moderately high- $T_g$ s and enhanced thermal stability and solubility.<sup>12</sup> The work presented here was designed to obtain the synthesis of a new diol monomer-containing dinaphthodioxocin heterocyclic group and its polycondensation with a set of

aliphatic diacid chlorides. Synthesis of the dimeric products containing methano-dibenzo[1,3]dioxocin and methano-16H-dinaphtho[1,3]dioxocin derivatives nuclei has been reported.<sup>13</sup>

Aromatic and aliphatic polyesters have been widely prepared and used. The purpose of this investigation was to synthesis a novel dinaphthodioxocin containing diol monomer, 8,16-methano-16H-dinaphtho[2,1-d:1',2'-g][1,3] dioxocin-2,14-diol (MDDD) and to examine the step-growth polymerization reactions of MDDD as a heterocyclic monomer with aliphatic diacid chlorides. In the present article, we report on the successful polycondensation reaction where MDDD as a novel monomer is used for the synthesis of new aliphatic polyesters containing dinaphthodioxocin heterocyclic parts. The physical properties of polymers including inherent viscosity, solubility behavior, thermal properties, and thermal stabilities were studied.

## EXPERIMENTAL

### Materials

All chemicals were purchased from Fluka AG and Merck Chemical companies. 2,7-Dihydroxynaphthalene (1) and tetrabutylammonium bromide were recrystallized before use. Malonaldehydetetramethyl acetal (2), acetyl chloride (3), and four commercial aliphatic diacid chlorides including succinyl chloride (4), adipoyl chloride (5), suberoyl chloride (6), and sebacoyl chloride (7) were used without any

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**TABLE I**  
**Characterization Data of Monomer and the Model Compound**

Code	Yield (%)	mp (°C)	IR (cm <sup>-1</sup> )	<sup>1</sup> H-NMR (ppm)	Mol. Ion (m/z)
MDDD	81	306	3400–3250, 2990–2910, 1240, 1150, 1075	2.2 (2H, CH <sub>2</sub> ), 5.2(1H, t, Ar–CH–Ar) 6.4 (1H, t, O–CH–O), 6.8–7.9 (10H, m, CH arom), 9.2 (2H, s, OH)	356
MDE	89	296–297	3045, 2950, 2840, 1755, 1625, 1515, 1450, 1390, 1315, 1250, 1175, 1065, 1025, 915, 830	2.23–2.65 (8H, CH <sub>2</sub> , CH <sub>3</sub> ), 5.45 (1H, Ar–CH–Ar), 6.42 (1H, O–CH–O), 6.9–8.16 (10H, m, CH arom)	440

purification. All the solvents used were purified by distillation before use.

### Instruments

Melting points were determined in open capillaries with a Buchi 535 instrument. <sup>1</sup>H-NMR spectra were recorded on a 250 MHz Bruker Advance DPX-250 spectrometer in dimethyl sulfoxide (DMSO)-*d*<sub>6</sub> as solvent at 25°C with frequencies of 250.13 and 62.89 MHz for the <sup>1</sup>H and <sup>13</sup>C-NMR spectra, respectively. Tetramethylsilane was used as shift reference and internal standard. FTIR spectra were recorded on a Unicam Matteson 1000 Instrument using KBr as a background reference. Vibrational transition frequencies were reported in wave number (cm<sup>-1</sup>). Mass spectra were recorded on Shimadzu GC-MS QP 1000 EX. Number-average molecular weights (*M<sub>n</sub>*) of polyesters were measured with a Waters GPC instrument (Gel Permeation Chromatography) at room temperature. Polystyrene was used as the reference standard. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis were performed on a TGA 1500 (version 5.32) system with a heating rate of 20°C/min under nitrogen atmosphere. Elemental analysis was performed on a Heraeus CHN-O-Rapid system by Research Institute of Petroleum Industry. The inherent viscosities of the polymers were determined for a solution of 0.5 g/dL in *N,N*-dimethylacetamide (DMAc) solvent at 30°C using an Ostwald viscometer.

### Monomer synthesis

8,16-Methano-16H-dinaphtho[2,1-d:1',2'-g][1,3]dioxocin-2,14-diol

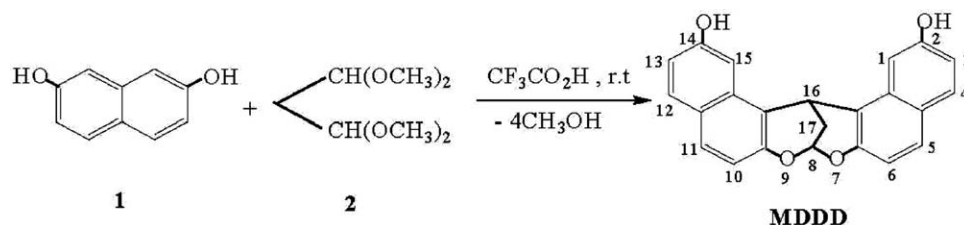
Into a 25-mL round-bottomed flask, 2,7-dihydroxynaphthalene (1.60 g, 10 mmol), malon aldehyde-

tetramethyl acetal (1.40 g, 10 mmol), and trifluoroacetic acid (TFA; 10 mL) were mixed at room temperature. The product began to precipitate from the solution gradually. The reaction mixture was then stirred at room temperature for 8 h and then poured into methanol. The product precipitated was filtered, washed with water several times, and boiled with water to remove unreacted dihydroxynaphthalene. The crude product was then recrystallized from ethanol–water (5/1, v/v) mixture to give MDDD. Yield and characterization data of monomer are shown in Table I. [Elemental analysis, Found: C, 77.15; H, 4.61, C<sub>23</sub>H<sub>16</sub>O<sub>4</sub> requires C, 77.53; H, 4.50%].

### Model compound synthesis

Preparation of 2,14-bis-acetyl-8,16-methano-16H-dinaphtho [2,1-d:1',2'-g][1,3]dioxocin

*Method A (interfacial condensation).* In a 50-mL two-necked, round-bottomed flask equipped with a reflux condenser, and a nitrogen gas inlet tube, MDDD (0.356 g, 1 mmol) and a small catalytic amount of tetrabutyl ammonium bromide (40 mg) were dissolved in 2.5 mL of 1M aqueous sodium hydroxide under nitrogen atmosphere. To this stirred solution, acetyl chloride (0.157 g, 2 mmol) in dichloromethane (10 mL) was added rapidly, and the mixture was stirred vigorously at room temperature for 4 h. The supernatant aqueous layer was decanted, and the mixture was then poured into hot water (150 mL) containing a small amount of concentrated HCl. The crude product was collected by filtration, washed with refluxing methanol, and dried at 60°C under vacuum overnight. This solid was recrystallized from acetone and water. mp =



**Scheme 1** Preparation of diol (MDDD).

TABLE II  
Conditions for the Preparation of Model Compound via Two Methods (A,B)

Code	Method A <sup>a</sup>				Method B <sup>b</sup>			
	Cat (40 mg)	Solvent	Temp. (°C)	Yield (%)	Cat.	Solvent	Temp. (°C)	Yield (%)
1	TBAB	Toluene	25	70	Py	Nitrobenzene	25	51
2	TBAB	Nitrobenzene	25	73	Py	TCE	25	55
3	TBAB	1,2-Dichloroethane	25	69	Py	Toluene	25	56
4	TBAB	Methylenechloride	25	89				

<sup>a</sup> Two-phase polycondensation method.

<sup>b</sup> Solution polycondensation method. Py, Pyridine; TCE, *sym* tetrachloroethane; TBAB, tetrabutylammonium bromide.

297–298°C; [Elemental analysis; Found: C, 72.74; H, 4.63, C<sub>27</sub>H<sub>20</sub>O<sub>6</sub> requires C, 73.63; H, 4.54%].

*Method B (solution condensation).* In a 50-mL two-necked, round-bottomed flask equipped with a reflux condenser, and a nitrogen gas inlet tube, acetyl chloride **3** (2 mmol, 0.157 g) in *sym*-tetrachloroethane (5 mL) was added dropwise to a solution of MDDD (1 mmol, 0.356 g), pyridine (1 mL) and *sym*-tetrachloroethane (5 mL). The solution was stirred for 8 h at room temperature under nitrogen atmosphere. The resulting mixture was poured into methanol (30 mL). The resulting solid was filtered off, washed thoroughly with refluxing methanol, and dried at 60°C to give model diester (MDE).

## Polymer synthesis

### Interfacial polycondensation

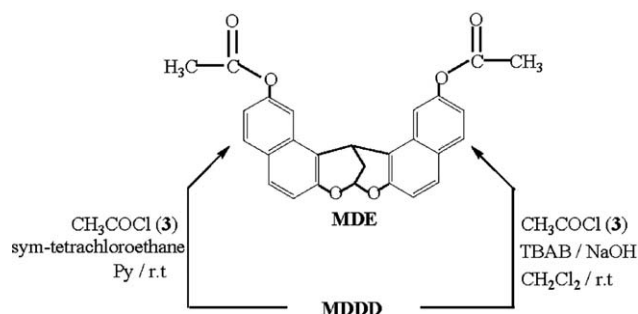
A typical procedure for the synthesis of polyester PE<sub>b</sub> is given as follows. In a 100-mL two-necked round-bottomed flask equipped with a reflux condenser and a nitrogen inlet tube, MMD (0.712 g, 2 mmol), aqueous sodium hydroxide (4.5 mL, 1M) were placed. To this stirred solution, a solution of adipoyl chloride **5** (2 mmol, 0.366g) in dichloromethane (10 mL) was added all at once. The resultant reaction mixture was stirred vigorously at room temperature for 2 h under nitrogen atmosphere. The polymer mixture was then poured into boiling water (300 mL) containing a small amount of concentrated HCl. The precipitated polymer was collected by fil-

tration and washed with hot water and refluxing methanol thoroughly. It was then vacuum-dried at 60°C overnight.

Polyesters, PE<sub>a</sub>, PE<sub>c</sub>, and PE<sub>d</sub>, were prepared from diol (MDDD) with the corresponding aliphatic diacid chlorides by a similar procedure described as earlier.

## RESULTS AND DISCUSSION

The polyester family is extremely large and, depending on the nature of monomers, exhibits an enormous variety of structures, architectures, properties, and, therefore, applications. Thus, synthesis of suitable monomers for esterification is required to tailor polyester properties. In our work, we selected methano-dinaphtho[1,3]dioxocin containing diol to introduce heterocyclic and bulky units in the main chain of final polyester. Hence, diol monomer (MDDD) containing dinaphthodioxocin was synthesized according to a well-established method<sup>14</sup> by the acid-catalyzed condensation of 2,7-dihydroxynaphthalene (**1**) and malonaldehydetetramethyl acetal (**2**) in TFA as both solvent and catalyst (Scheme 1). This method offers advantages such as the efficiency and simplicity, high yield, readily available starting materials, and potential for introducing an acetal or dinaphthodioxocin structure and flexible linkages between the rigid aromatic moieties. Therefore, this single-step reaction was utilized for the preparation of new diol monomer. The structure of diol was confirmed by elemental analysis and IR, NMR, and mass spectroscopies. FTIR spectra of MDDD showed characteristic broad absorption band of hydroxyl groups (O–H stretching) at 3550–3250 cm<sup>-1</sup>. In addition, acetalic C–O and phenolic C–O absorption bands at 1215, 1235, 1020, 1075, 1150, and 1130 cm<sup>-1</sup> were present, respectively. <sup>1</sup>H-NMR spectra of diol showed proton of hydroxyl groups at δ 9.2 ppm, proton on the diarylmethyl carbon (Ar–CH–Ar) at δ 5.2 ppm, proton on the acetal carbon (O–CH–O) at δ 6.4 ppm, and proton on the bridge carbon at δ 2.2 ppm, which confirmed the formation of acetalic structure and the overall mirror symmetry. Also, mass spectra of diol showed the appropriate molecular ion peak.



Scheme 2 Preparation of model compound (MDE).

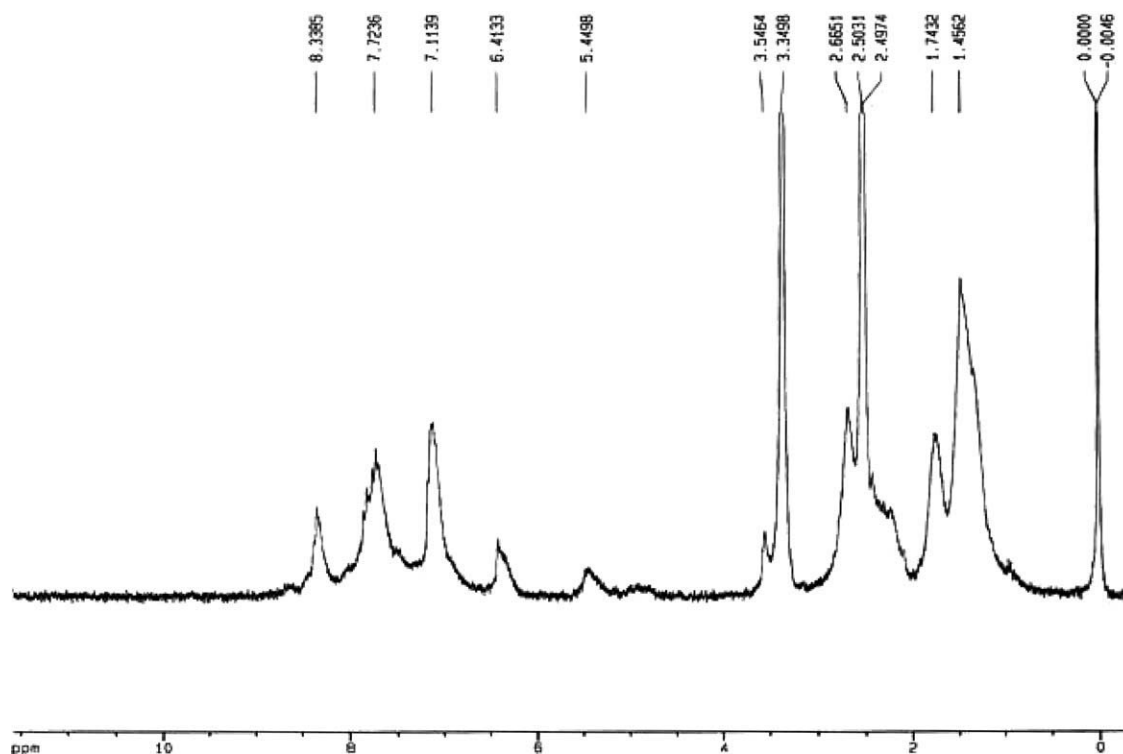


Figure 1  $^1\text{H-NMR}$  spectrum of polyester  $\text{PE}_d$  in  $\text{DMSO-}d_6$ .

TABLE III  
Yield, Inherent Viscosity,  $M_n$ , and Spectroscopic Data of Polyesters

Code	Yield (%)	$\eta_{\text{inh}}$ (dL/g) <sup>a</sup>	$M_n$ <sup>b</sup>	IR ( $\text{cm}^{-1}$ )	$^1\text{H-NMR}$ (ppm)
$\text{PE}_a$	91	0.38	22,100	2945, 2835, 1740, 1625, 1515, 1260, 1170, 1065, 1015, 720	2.45–2.61(6H, $\text{CH}_2$ ), 5.51(1H, Ar–CH–Ar), 6.45(1H, O–CH–O), 6.9–8.45(10H, Ar)
$\text{PE}_b$	90	0.31	19,100	2940, 2840, 1740, 1620, 1515, 1265, 1160, 1055, 1015, 715	2.40–2.60(6H, $\text{CH}_2$ ), 1.35–1.65(4H, $\text{CH}_2$ ), 5.55(1H, Ar–CH–Ar), 6.43(1H, O–CH–O), 6.9–8.4 (10H, Ar)
$\text{PE}_c$	86	0.29	18,720	2940, 2835, 1740, 1630, 1515, 1260, 1165, 1090, 1020, 740	2.39–2.60(6H, $\text{CH}_2$ ), 1.30–1.60(8H, $\text{CH}_2$ ), 5.45(1H, Ar–CH–Ar), 6.50(1H, O–CH–O), 6.9–8.6(14H, Ar)
$\text{PE}_d$	84	0.26	17,600	2940, 2835, 1745, 1630, 1510, 1235, 1152, 1060, 1015, 745	2.35–2.66(6H, $\text{CH}_2$ ), 1.27–1.80(12H, $\text{CH}_2$ ), 5.44(1H, Ar–CH–Ar), 6.41(1H, O–CH–O), 6.95–8.40(18H, Ar)

<sup>a</sup> About 0.5 g/dL in DMAc as solvent at 30°C.

<sup>b</sup> Measured by GPC in chloroform.

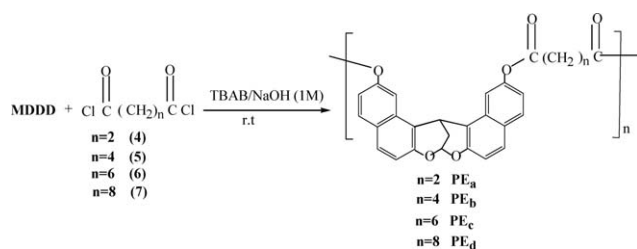
TABLE IV  
Solubility Behavior of Polyesters

Polymer	DMSO	DMAc	DMF	NMP	HMPTA	$\text{H}_2\text{SO}_4$	<i>m</i> -Cresol	$\text{CH}_2\text{Cl}_2$	THF	TCE	Acetone
$\text{PE}_a$	+	+	+	+	+	+	+	±	±	±	±
$\text{PE}_b$	+	+	+	+	+	+	+	±	±	±	±
$\text{PE}_c$	+	+	+	+	+	+	+	+	+	+	±
$\text{PE}_d$	+	+	+	+	+	+	+	+	+	+	±

+, soluble at room temperature; ±, soluble with heating; DMSO, dimethyl sulfoxide; DMAc, *N,N*-dimethylacetamide; DMF, *N,N*-dimethyl formamide; NMP, *N*-methyl pyrrolidone; HMPTA, hexamethyl phosphoric triamide; THF, tetrahydrofuran; TCE, 1,1,2,2-tetrachloroethane.

<sup>a</sup>Qualitative solubility was performed with 5 mg sample in 1 mL solvent.



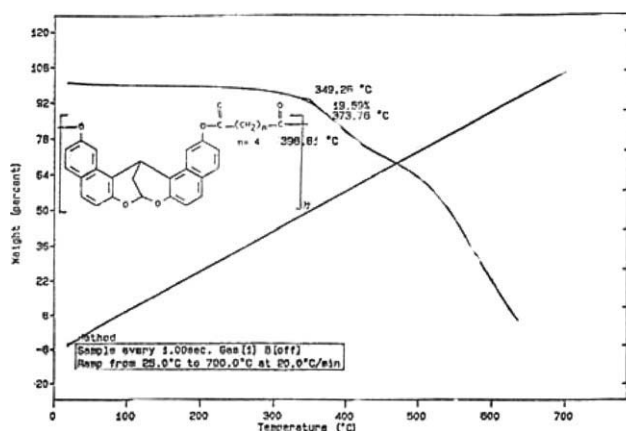


**Scheme 3** Preparation of polyesters (PE<sub>a-d</sub>).

The elemental analysis of diol was in good agreement with the expected value.

To find information about the structures and most suitable preparation procedure for the final polyesters, a MDE was synthesized according to well-known techniques (two methods A,B)<sup>14–18</sup> with different catalyst/solvent systems (Table II). As seen, both of these methods were successful for preparation of MDE, and the best results were obtained via interfacial condensation method (Scheme 2). On the other hand, the reaction of MDDD with acetyl chloride by two-phase condensation method was optimized by the use of different solvents as organic media. The results showed that the efficiency and the yield of the reaction in dichloromethane were higher than those obtained in other solvents, such as, toluene, nitrobenzene, and 1,2-dichloroethane. Thus, dichloromethane was chosen as the best organic media. FTIR spectrum of MDE showed ester bands at 1735 cm<sup>-1</sup> (C=O stretching) and 1055 cm<sup>-1</sup> (C–O stretching). <sup>1</sup>H-NMR of this compound showed protons on the diarylmethyl carbon, acetal carbon, bridge carbon, and methyl carbon at  $\delta$  5.47, 6.44, 2.23, and 2.65 ppm and a broad peak at  $\delta$ (6.9–8.16) ppm for aromatic protons, respectively. In the mass spectra, the molecular ion peak at 440 was observed. Also, the results of elemental analysis of MDE confirmed the formation of proposed structure.

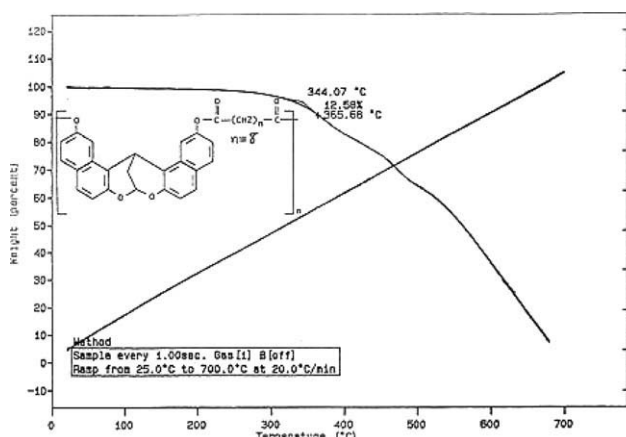
Polyesters (PE<sub>a-d</sub>) were prepared using optimized conditions obtained from the study of the model compound (Scheme 2). The two-phase polycondensation method offered polymers with higher molecular weight and yield than the solution method. So, this method was chosen for the preparation of polyesters. The polyesters (PE<sub>a-d</sub>) were characterized by IR and NMR spectroscopies. FTIR spectra of the polyesters showed characteristic absorption bands of ester linkage around 1750 cm<sup>-1</sup> (C=O stretching) and 1025–1035 cm<sup>-1</sup> (C–O stretching). The IR spectra also showed two medium absorption peaks of aliphatic C–H at around 2925 and 2845 cm<sup>-1</sup> (asymmetric and symmetric stretching vibrations). <sup>1</sup>H-NMR spectra of the polyesters showed a peak in the region  $\delta$  5.44–5.51 ppm assigned to the hydrogen on the diarylmethyl carbon (Ar–CH–Ar), a peak in the region  $\delta$  6.39–6.48 ppm assigned to the hydrogen on the acetal carbon (O–CH–O), a peak in the region



**Figure 2** TG curve of polyester PE<sub>b</sub>.

1.15–2.35 ppm assigned to the hydrogen on the bridge carbon and aliphatic segments, and broad peaks in the region  $\delta$  6.9–8.32 ppm due to the aromatic protons. A representative <sup>1</sup>H-NMR spectrum of polyester PE<sub>d</sub> is shown in Figure 1. Spectroscopic data of all polyesters are collected in Table III. Also, as a representative example, the elemental analysis of PE<sub>b</sub> was as follows: [Elemental analysis; Found: C, 73.93; H, 4.76. C<sub>29</sub>H<sub>22</sub>O<sub>6</sub> requires C, 74.67; H, 4.72%].

The inherent viscosity of the polymers as a criterion for evaluation of molecular weights was measured in DMAc at 30°C, and they were in the range of 0.26–0.38 dL/g, which revealed that this method of polymerization was suitable, and the polymers were of moderately high-molecular weights (Table III). The results of GPC measurements for the polyesters are presented in Table III. Number-average molecular weights ( $M_n$ ) of polyesters were in the range of 17600–22100. A decrease of inherent viscosity was observed by the introduction of aliphatic diacid chlorides with a higher number of methylene groups. This could be due to the formation of cyclic structure from long alkyl chain. Only polyester PE<sub>a</sub>



**Figure 3** TG curve of polyester PE<sub>d</sub>.

TABLE V  
Thermal Properties of Polymers (PE<sub>a-d</sub>)

Polymer	IDT <sup>a</sup> (°C)	T <sup>b</sup> <sub>10%</sub> (°C)	T <sub>max1</sub> <sup>c</sup> (°C)	T <sub>max2</sub> <sup>d</sup> (°C)	T <sub>g</sub> <sup>f</sup> (°C)	Ch. Y <sup>e</sup> (%)
PE <sub>a</sub>	361	379	419	566	126	24
PE <sub>b</sub>	356	378	411	561	114	26
PE <sub>c</sub>	349	368	393	558	101	23
PE <sub>d</sub>	344	367	384	566	93	27

<sup>a</sup> Initial decomposition temperature = the temperature of 5% weight loss.

<sup>b</sup> Temperature of 10% weight loss, obtained from TGA.

<sup>c</sup> First maximum polymer decomposition temperature.

<sup>d</sup> Second maximum polymer decomposition temperature.

<sup>e</sup> Char yield percent at 600°C, obtained from TGA.

<sup>f</sup> Measured on DSC at a heating rate of 20°C/min under nitrogen.

could be cast readily from chloroform solution to form transparent and flexible film.

The organosolubility of the polyesters was examined qualitatively in different solvents. As shown in Table IV, these polymers were readily soluble at room temperature in strongly polar aprotic solvents such as DMSO, DMAc, dimethylacetamide (DMF), *N*-methyl pyrrolidone (NMP), hexamethyl phosphoric triamide (HMPTA), and sulfuric acid without need for heating. Also, by heating, they were soluble in less-efficient polar solvents such as *m*-cresol and THF and chlorinated solvents, but insoluble in acetone and methanol. In contrast, polyesters based on bisphenol-A with aliphatic diacid chlorides (4–7 in Scheme 3) showed less solubility in common organic solvents.<sup>19</sup> This result indicates that the incorporation of bulky methano-dinaphtho[1,3]dioxocin moieties into the polyester backbone enhanced the solubility of polyesters. The incorporation of aliphatic ester linkage into the polymer backbone enhances the solubility in common organic solvents to a high degree.

The thermal behavior of the resulting polyesters was studied by TGA at a rate of 20°C/min under nitrogen atmosphere. Figures 2 and 3 show the TGA results generated on the PE<sub>b</sub>, PE<sub>d</sub> samples under study. The most important TGA data (the initial decomposition temperature, temperature of 10% weight loss, and the yield of char residue at 600°C) are also summarized in Table V. The polymers did not show significant weight loss below 296°C. They began to decompose in the range of 344–361°C showing a 10% weight loss in the range of 367–379°C. As it can be seen from TGA curves, the dinaphthodioxocin-containing aliphatic polyesters decompose in a two-stage weight loss process. The first maximum of decomposition ( $T_{max1}$ ) was in the range of 384–419°C and was due to the destruction

of aliphatic segments. The second maximum of the decomposition ( $T_{max2}$ ) was in the range of 549–566°C and was due to the degradation of aromatic residue (Table V). Glass transition temperature ( $T_g$ ) of polyesters was determined by DSC at a heating rate of 20°C/min under nitrogen. The  $T_g$  values are given in Table V.  $T_g$  values for polyesters containing dinaphthodioxocin moieties were in the range of 93–126°C. The weights of remaining at 600°C (char yields) were in the range of 23–27% (Table V).

The preparation of polyurethanes, polyarylates, and other polymers from MDDD is under investigation in our laboratory.

## CONCLUSIONS

A new MDDD was synthesized through the condensation of 2,7-dihydroxynaphthalene with malonaldehydetetramethyl acetal. The monomer and model compound were fully characterized by IR, NMR spectroscopies, and elemental analysis. Step-growth polycondensation reactions of MDDD with different aliphatic diacid chlorides by interfacial technique yielded different polyesters. All polymers were characterized, and their physical and thermal properties were studied.

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