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NOTE
SYNTHESIS AND CHARACTERIZATION OF SOME NEW POLYESTERS FROM 4,4'-DICARBOXYACETYLDIPHENYL ETHER AND ITS DIMETHYL ESTER

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INTRODUCTION

In continuation of the work in connection with the synthesis and characterization of polyketoetheresters [1], the present paper describes the synthesis of structurally related polyketoetheresters in which the ether linkage is in the acid moiety of the repeat unit. The polyesters listed in Table 1 were prepared by polycondensation of equimolar amounts of 4,4'-dicarboxyacetyldiphenyl ether (DCADPE) and the required diacetate of dihydroxyarenes and/or of the dimethyl ester of DCADPE and the required aliphatic diol or arene diol in the presence of zinc acetate.

EXPERIMENTAL

Polycondensation of 4,4'-Dicarboxyacetyldiphenyl Ether with Bisphenol A Diacetate

A mixture of DCADPE [2, 3] (3.42 g, 0.01 mol), Bisphenol A diacetate [4] (3.12 g, 0.01 mol), and zinc acetate (0.028 g) was heated at 200°C for

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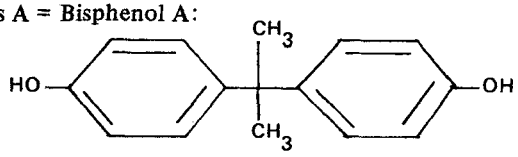
BLE 1. Polycondensation of DCADPE with Diacetate of Dihydroxyarenes[b] and Dimethyl Ester of DCADPI
n Dihydroxyarenes and Characterization[a]

Diol[b]	Yield, %	$\bar{M}_n \pm 100$ by EGA	TGA Data				
			$E_a \pm 0.5$ kcal/mol	IDT, K	T_{max}, K	T_s, K	IPD K
Bis A diAc	77	1650	28	565	699	685	753
Bis C diAc	62	1350	26	561	672	689	769
Bis F diAc	63	1300	26	563	689	683	760
Hydroquinone diAc	72	1400	29	560	663	680	741
Resorcinol diAc	68	1600	28	562	689	690	735
Catechol diAc	67	1450	30	564	627	680	721
1:5 diAc naphthalene	64	1800	24	563	654	678	715
2:7 diAc naphthalene	64	1600	27	565	681	673	741
Phenolphthalein diAc	64	1700	28	566	703	675	743
Anthraquinone diAc	70	1600	25	555	689	687	726
1,2-Ethanediol	54	1200	25	560	690	688	725
1,3-Propanediol	60	1100	24	553	681	687	722
1,4-Butanediol	58	1300	25	559	680	692	725
Digol	60	1300	23	562	698	689	739
Trigol	59	1250	23	563	699	681	749

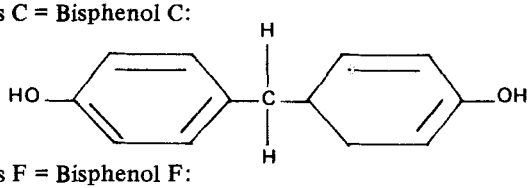
16	Bis A	72	1600	26	560	676	680	7
17	Hydroquinone	70	1350	27	558	663	679	7
18	1:5-Dihydroxynapthalene	63	1550	22	561	653	676	7

[a]Numbers 1-10, reaction with DCADPE with diacetates; Nos. 11-18, by ester exchange with dimethyl este
DCADPE.

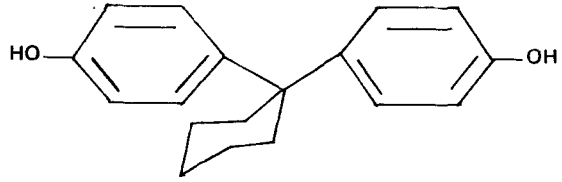
[b]Bis A = Bisphenol A:



Bis C = Bisphenol C:



Bis F = Bisphenol F:



digol = Diethylene glycol:

$HOCH_2CH_2OCH_2CH_2OH$

trigol = Triethylene glycol:

$HOCH_2CH_2OCH_2CH_2OCH_2CH_2OH$

2 h and then at 220°C for 8 h while the volatiles were allowed to escape. This solid reaction mixture was powdered and extracted in a Soxhlet extractor with methanol to remove unreacted monomers. The DCADPE-bis A diAc sample was a brown-colored solid. It was insoluble in all organic solvents and did not fuse up to 300°C.

Polycondensation of the dimethylester of DCADPE with ethylene glycol was effected in a similar manner. The designation and the yields of all the polyester samples prepared by either of these methods are shown in Table 1. The C and H contents of all the samples agreed reasonably well with the expected values. They are formed in 54 to 77% yield depending upon the nature of the monomer.

RESULTS AND DISCUSSION

The polyketoetherester samples reported in Table 1 are brown to dark-brown solids and are insoluble in all organic solvents like the polyketoetherester reported in the earlier communication [1]. The values of $\bar{M}_n$ of the insoluble and infusible polyester samples, estimated by end –COOH group titration of the swelled samples with 0.1 *M* alkali, was found to lie between 1100 to 1800, depending upon the nature of the monomers involved and the method of synthesis. The IR spectra of the polyketoetherester samples show all the expected characteristics, particularly the ester carbonyl bond either around 1740 or 1730 cm^{-1}, depending upon the structure, and a keto carbonyl band at 1692 cm^{-1}.

Examination by TGA and analysis by the Broido method [5] revealed that all the polyester samples degrade in a single step. The energy of activation, E_a, of the degradation reaction of the samples were estimated by the Broido method (Table 1). The E_a of polyesters prepared from DCADPE and diacetate of dihydroxyarenes was 22-30 kcal/mol and that of polyesters from the dimethyl ester of DCADPE and aliphatic diols was 23-25 kcal/mol. The temperature characteristics, such as initial decomposition temperature (IDT), maximum rate of decomposition temperature (T_{max}), half volatilization point temperature (T_s), and integral procedural decomposition temperature (IPDT) for the degradation reaction of the polyesters [6], were found to be very similar.

The infusibility and insolubility of these polyketoetheresters of low molecular weights seem to suggest a strong intermolecular interaction between the vicinal flat and planar biphenylene moieties of the repeat units of polymer

chains [1]. The intermolecular attractive dipolar interaction cannot account for such properties.

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