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SYNTHESIS AND PROPERTIES OF FERROELECTRIC 4-[4-(S-1-METHYLHEPTYLOXY)BENZOYLOXY]-4'-n-ALKYLOXYCARBONYLBIPHENYLS

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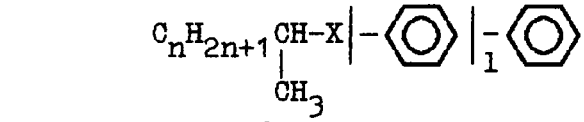
Abstract Synthesis, mesomorphic properties and spontaneous polarization values of 4-[4-(S-1-methyl-heptyloxy)benzoyloxy]--4'-alkyloxycarbonylbiphenyls are given.

INTRODUCTION

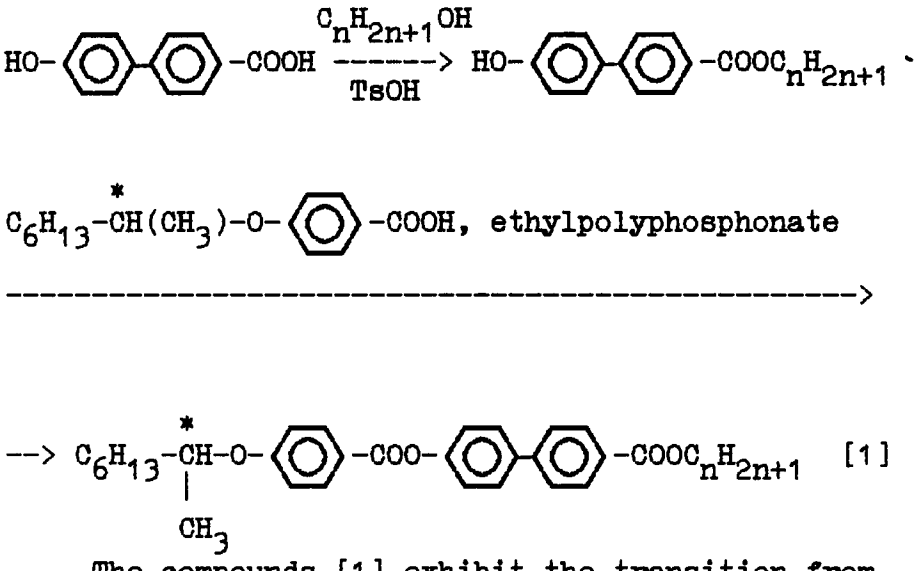
The patent[1] of Chisso company is known, which claims the compounds



where n=2÷16, l=0, 1 or 2, X=$-CH_2-$ or -O-, Y=-R, -OR, -COR, -OCOR, -OCOOR or -COOR. The authors indicate the high stability and small response times, but no examples of numerous classes of claimed compounds are presented. We have synthesized the homologous 4-[4-(S-1-methyl-heptyloxy) benzoyloxy]-4'-n-alkyloxy- carbonyl-biphenyls. The phase transition temperatures, the values of spontaneous polarization and the response times of electrooptical cells are presented.

RESULTS

The 4-[4-(S-1-methylheptyloxy)benzoyloxy] 4'-n-alkyloxycarbonylbiphenyls were synthesized according following scheme:



The compounds [1] exhibit the transition from crystal form to ferroelectric smectic C phase at moderate temperatures. Most of the compounds [1] form different crystal modifications when crystallized from different solvents (higher melting points are obtained from ethanol, the lower ones - from hexane). The phase transition temperatures are given in Table 1.

The values of spontaneous polarization have been determined by Sawyer-Tower method in the cells with an interelectrode gap 10 μm, they are given in Table 1. These values correspond to the temperatures, by 10 °C lower than the phase transition points (S_C-S_A or S_C-N). The tilt angle is $\theta=30^\circ$ (n=6, 46°C), 32° (n=8, 60°C).

The same cells have been used both for spontaneous polarization and electrooptical measurements.The cells have been placed between the crossed polarizers, the electrodes being connected with the source of the rectangular voltage pulses with an alternation of polarity at 1 Hz frequency. The response less than 1 ms is observed when voltage is 10V (see Table 1).

TABLE 1 The phase transition temperatures (^{o}C), the values of spontaneous polarization (P_s,nC/cm^2) and electrooptical cell response times (τ,ms) of 4-[4-(S-1-methylheptyloxy)benzoyloxy]-4'-n-alkyloxycarbonylbiphenyls.

n	Cryst solvent	K	S_C	S_A	N	I	P_s	τ_{on}	τ_{off}
5	Hexane	.40.5 .		- 66	.78	. 58		9.7	1,5
6	Hexane	.45 .		- 63	.72.5.	84		0.8	0.5
	EtOH	.52 .		- 63	.72,5.				
7	EtOH	.53 .		- 64	.76	. 77		1.0	0,5
8	EtOH	.48,5 .	66 .	67	.74.5.	65		0,8	0.3
9	Hexane	.46	. 64 .	67	.76.5.	58		0,9	0,6
	EtOH	.50	. 64 .	67	.76,5.				
10	Hexane	.38	. 63 .	66	.73	.			
	EtOH	.53	. 63 .	66	.73	.			
11	Hexane	.52	. 64 .	69	.72	.			
12	Hexane	.49	. 62 .	64	.69	. 51		1,1	0.9

EXPERIMENTAL

4-Alkyloxycarbonyl-4'-hydroxybiphenyls

A mixture of 0.02 mol (4.3 g) 4-hydroxybiphenyl-4'-carboxylic acid, 0.1 mol n-alkanol, 150 ml toluene and 0,5g p-toluenesulfonic acid was refluxed with azeotropical elimination of water untill dissolving all the 4-hydroxy biphenyl-4'-carboxylic acid (6-10 h). Toluene (200 ml) was added, the solution was washed with water and dried. The solvent and the excess of n-alkanol were evaporated at a reduced pressure, the residue was recrystallized from mixture of toluene and hexane (4:1). The yield (%), melting point (^{o}C) and n are given: 56, 105-106, 5; 67, 99-101, 6; 53, 97-98, 7; 55, 101-103, 8; 60, 98-100, 9; 54, 109-110, 10; 53, 101-103, 11; 52, 104-105, 12.

The structure of the compounds was confirmed by PMR spectra and elementary analysis data.

4-[4-(1-S-Methylheptyloxy)benzoyloxy]-4'-n-alkyloxycarbonylbiphenyls

A mixture of 0.01 mol 4-alkyloxycarbonyl-4'-hydroxybiphenyl, 0.01 mol (2.50 g) 4-(S-1-methylhepthyloxy)-benzoic acid, was refluxed for 30h. The solvent was evaporated, the residue was diluted by 50 ml water and extracted with diethyl ether. The extract was washed by water, dried and the solvent was evaporated. The residue was chromatographed on silicagel, an eluant - a mixture of hexane with diethyl ether (1:1). The solvent was evaporated, the residue was crystallized, by turns, from hexane and ethanol

till constant phase transition temperatures. The structure of all compounds [1] was confirmed by PMR spectra and elementary analysis data.

REFERENCES

1. H.Inoue, S.Saito, K.Miyazawa, T.Inukai and K.Terashima, Eur. Pat. Appl. 0164814 A2(1985).