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Experimental. PPO, ($\bar{M}_v = 40\ 000$), a commercial product from General Electric Co., was dissolved in α-pinene[1] and PPOO, ($\bar{M}_v = 70\ 000$), synthesized as described in Ref. 2, in cyclohexanone. After dissolution of the PPO sample, the solution was held at 353 K for 5 days. The solution of the PPOO sample was stored at room temperature for the same period of time. After the crystals of PPO had been separated from α-pinene, they were washed several times with ether and dried under vacuum. Cyclohexanone was removed from the crystalline PPOO in a Hösli HT 125 vacuum desiccator. The samples (about 5 mg) were coated with a layer of gold about 200 Å thick and scanned with a Jeolco JSM-U3 scanning electron microscope. The tilting angle of the specimen was 45°. Calorimetric studies were carried out with a Perkin-Elmer 1B differential scanning calorimeter. The weight of the PPOO sample was 15 mg and the scan speed 16°/min.
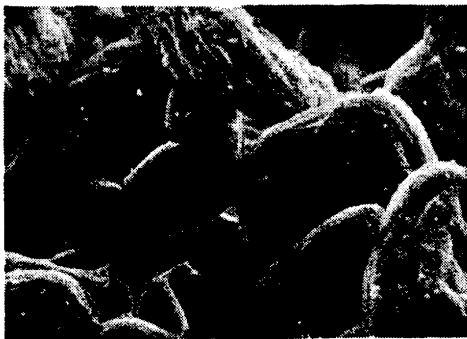
Studies of Crystals of 2,6-Disubstituted Polyphenylene Ethers with a Scanning Electron Microscope

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Barrales-Rienda crystallized poly(2,6-dimethyl-1,4-phenylene ether) (PPO)* from α-pinene and obtained a melting point of 507 K for the polymer crystals.[1] He also investigated the crystals with an optical microscope, but as far as we know, no high-resolution studies of the crystals have been conducted with a scanning electron microscope (SEM). In order to elucidate the crystalline structure of PPO and also that of the analogous poly(2,6-dimethoxy-1,4-phenylene ether) (PPOO), we have studied crystals of the mentioned polymers by the SEM technique and compared details of the micrographs with the results of DSC measurements.

* PPO is a trademark of the General Electric Co., U.S.A.
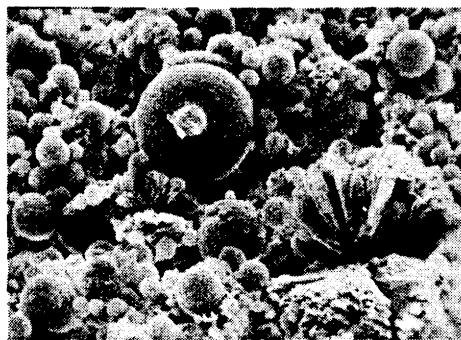

Fig. 1a. Crystals of PPO (6000×).

Fig. 1b. Crystals of PPOO (4500×).

Discussion and results. Fig. 1a shows that all the crystals of PPO are hedritic. The micrograph of PPOO in Fig. 1b shows that two different types of crystals, partly dendrites and partly spheres, are formed during the crystallization. In DSC analysis PPO gives only one endothermic peak at 507 K[1] whereas PPOO gives two peaks at
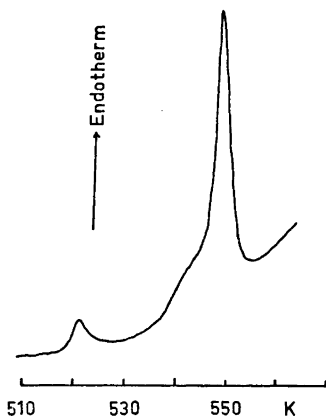

Fig. 2. DSC thermogram of PPOO. Endothermic peaks occur at 523 K and 549 K.

523 K and 549 K (Fig. 2). It can be assumed that these endothermic peaks of PPOO correspond to the two different crystalline forms.

The work is being continued.

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Dissociation Constants of Thiomalic Acid in Aqueous Potassium Chloride Solutions

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In connection with studies on metal complex formation by thiomalic acid, $HOOC \cdot CHSH \cdot CH_2 \cdot COOH$ (mercaptosuccinic acid), the dependence of the dissociation constants of the acid on ionic strength with potassium chloride as the neutral salt was studied potentiometrically.

Table 1. Potentiometrically determined dissociation constants of thiomalic acid at 25°C. The calculated values were obtained by means of eqns. 4 – 6.

Observed		Calculated	
I	pK_1	pK_1	$pK_1{}^\circ$
0.005	3.375	3.372	3.435
0.203	3.231	3.235	3.429
0.504	3.214	3.220	3.428
1.003	3.249	3.238	3.445
2.004	3.306	3.310	3.430
	pK_2	pK_2	$pK_2{}^\circ$
0.006	4.880	4.872	5.020
0.011	4.828	4.830	5.010
0.020	4.751	4.779	4.984
0.031	4.750	4.735	5.026
0.211	4.502	4.504	5.010
0.511	4.457	4.418	5.050
1.011	4.366	4.408	4.970
2.011	4.516	4.506	5.024
	pK_3	pK_3	$pK_3{}^\circ$
0.012	10.872	10.848	11.167
0.018	10.763	10.791	11.115
0.030	10.678	10.707	11.114
0.038	10.718	10.664	11.198
0.219	10.212	10.254	11.102
0.519	10.051	10.036	11.159
1.020	9.918	9.906	11.155
2.019	9.870	9.876	11.138