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POLY-*p*-PHENYLENE OXIDE

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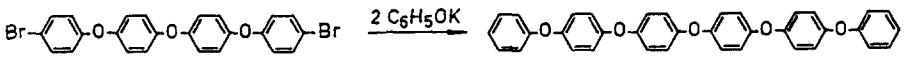
Abstract—Linear poly-*p*-phenylene oxide can be prepared by an Ullmann condensation of *p*-bromo sodium phenolate. The polymer is obtained as a crystalline ivory-coloured powder. Compression-moulding of this polymer at 320° and subsequent quenching in water result in amber transparent and tough amorphous films (T_g = 83°, $T_{crystall.}$ = 112° and T_m ~290°). The crystalline structure of the polymer has been elucidated to a large extent by means of X-ray analysis. The thermal and the oxidative stability of the polymer have been studied and the tensile strength of oriented films and of melt-spun fibres has been determined.

INTRODUCTION

ABOUT 30 years ago Staudinger and Staiger prepared oligomeric paraphenylene ethers by the Ullmann condensation of *p,p'*-dihalogeno-phenylene ethers and potassium phenolate,[1] e.g.:



In 1962 the polycondensation of *p*-bromo-sodium phenolate catalysed by cuprous compounds was described in a patent[2] but few properties of the polymer were mentioned. In connection with work in our laboratory on polyethers prepared by the oxidative coupling of phenols,[3–8] we were interested in the structure and properties of this polymer, and we therefore repeated and extended the work of Stamatow[2]. Some of the results obtained by another group are given in a recent patent.[9]

The Ullmann condensation has lately been the subject of renewed investigations.[10–25] The mechanistic aspects of the highly selective Ullmann condensation described here will not be discussed in this paper, however.

EXPERIMENTAL

Materials

Pyridine (Merck) was distilled over KOH.

CuCl was prepared by reduction of $CuSO_4$.[26]

1,4-dimethoxybenzene (Fluka) was distilled and recrystallized from methanol, m.p. 56° (lit. 56°).[27]

p-Bromo phenol was prepared according to the procedure of Podall and Foster.[29] The crude reaction mixture was fractionated in a ten-plate vigreux column. A fraction, b.p. 111–113°/10 mm, was collected and recrystallized three times from carbon tetrachloride. The *p*-bromo phenol* (m.p. 63–65°, overall yield 78%) was found to be pure in both gas chromatographic and thin-layer chromatographic analysis; no impurities were detected in the mother liquor.

* *p*-Bromo phenol prepared via *p*-bromo aniline showed a higher melting point (66°), lit. 66·4°.[27] After polymerization, however, no significant differences were found.

Polymerization

The reaction conditions are quite critical. The best results were obtained in the following way.

Apparatus. The apparatus used consisted of a 500 ml four-necked flask which could be heated by means of a vapour bath. The necks were provided with (a) a thermometer, (b) a stirrer, (c) a Claisen head equipped with two dropping funnels mounted one over the other for preparing and adding the catalyst, with a thermometer and with an inclined cooler, (d) a dropping funnel for adding the other reagents. All these parts were connected to each other for nitrogen supply and vacuum drawing.

Catalyst. The catalyst was prepared by bringing 0·1 g of CuCl in the upper of the two small dropping funnels and 2·5 ml of pyridine in the lower one. The funnels were deaerated and the CuCl was transferred to the lower funnel by means of vacuum. The turbid yellow solution could be added to the reaction mixture via a small filter plate in the stem of the lower funnel.

Procedure. Three times in succession, the apparatus was evacuated and filled with nitrogen. Methanol (40 ml) was put in the flask and a solution of 25·000 g of *p*-bromo-phenol in 40 ml of methanol in the funnel. Pure sodium (3·3222 g) was added to the methanol in the flask. In order to avoid reduction of the C–Br bond, the *p*-bromo phenol solution was transferred to the apparatus after the sodium had dissolved completely. Subsequently 84·5 g of 1,4-dimethoxy benzene was added. The flask was heated gradually and the methanol evaporated *in vacuo* until the temperature of the reaction mixture was 90° and the 1,4-dimethoxy benzene started to boil. The catalyst was added and the apparatus was filled with nitrogen. The temperature of the mixture was gradually increased by means of a vapour bath (acetophenone, b.p. 202·3°). At 140°, the solution suddenly became turbid as NaBr precipitated. After 1–2 hr at 200° the mixture became viscous. The reaction was stopped by adding 4 g of benzoic anhydride which caps the ONa end groups. Stirring was continued for 1 hr at 200°. The reaction mixture was cooled and at 100° acetone was slowly added. The solid polymer was filtered and washed with acetone until no further solids could be extracted. Next the polymer was washed with water until free from halogen. The tan-coloured powder was dried at 125° *in vacuo* for 24 hr.

Measurements of physical and mechanical properties

Solution viscosity. Clear 1 per cent solutions of the polymer samples were obtained in boiling nitrobenzene (N_2). The relative viscosities of the solutions were measured in an Ubbelohde viscometer at 140°.

Melt viscosity. The polymer was heated under nitrogen by means of a vapour bath (hexachlorobenzene, $t = 318°$) in a nitrogen atmosphere. The molten polymer was pressed upward into a capillary (21 mm dia.) by means of nitrogen pressure (5–20 cm Hg). The melt viscosity is calculated from the flow rate of the polymer in the capillary.[29]

The relationship between the solution viscosity and the melt viscosity is represented in Fig. 1.

Compression-moulding of film. Several grams of polymer powder were moulded between two sheets of aluminum foil at 320° and a pressure of 100 kg/cm^2. Immedi-

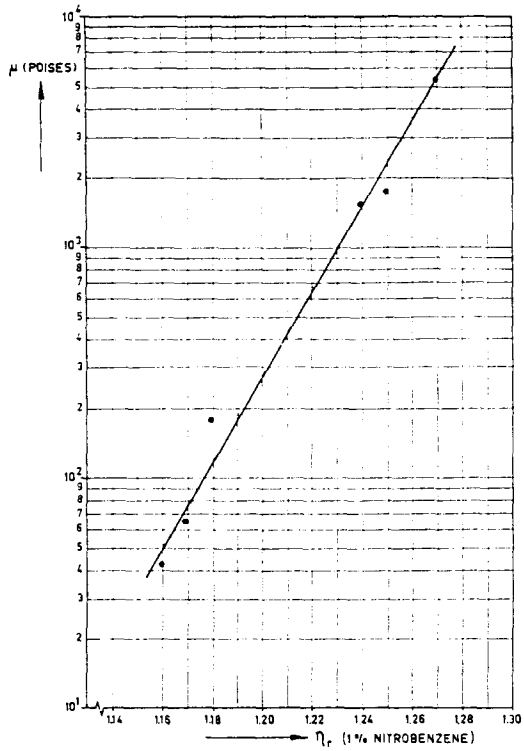


FIG. 1. Relation between solution viscosity and melt viscosity of poly-*p*-phenylene oxide.

ately after moulding the film was quenched by immersion in cold water to prevent crystallization.

Glass transition temperature (T_g), *crystallization temperature* (T_c), *melting point* (T_m). T_c and T_m were determined on films of 40 μ thickness with the aid of a Koffler polarization microscope equipped with a hot stage. The films were embedded in silicon oil between two cover glasses. T_g, T_c and T_m were also determined with the aid of a Dupont DTA apparatus (Fig. 6). In both cases the heating rate was 4°/min.

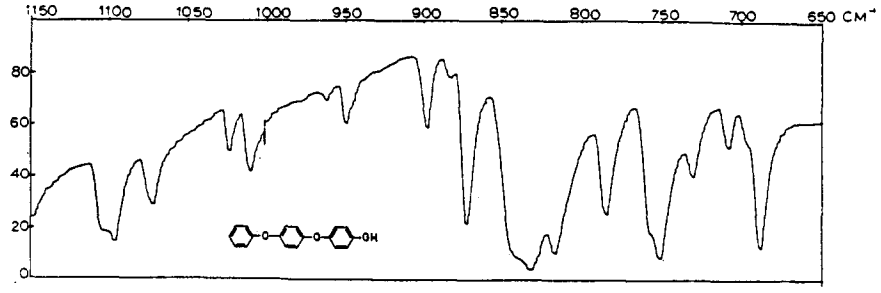
Tensile properties. These were determined with the aid of an Instron universal testing instrument (gauge length 5 cm; crosshead speed 5 cm/min).

X-ray photographs were made with Philips equipment. The Astbury diagrams were obtained with Ni filtered CuK α radiation. The distance to the screen was 3 cm.[30]

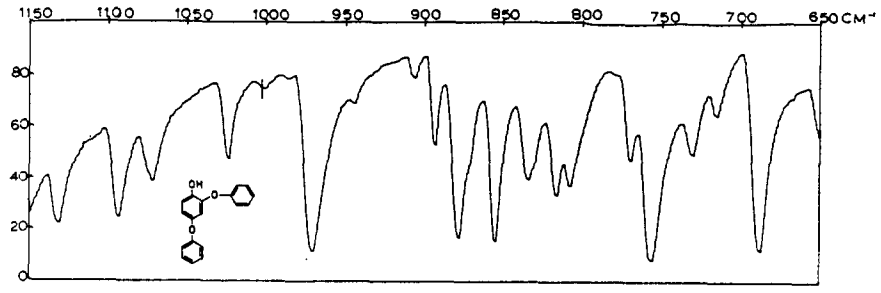
STRUCTURE AND PROPERTIES OF THE POLYMER

Infra-red spectra

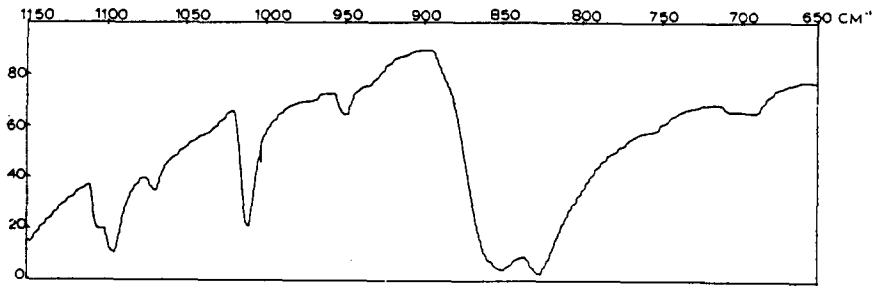
The i.r. spectra of the polymer samples obtained by the Ullmann condensation of bromo sodium phenolates were compared with those of a number of model compounds (cf. 3). In Fig. 2 examples are given of parts of the spectra of the linear *p*-trimer (I), the *ortho*-linked trimer (2,4-diphenoxy phenol) (II), and of two polymers (III and IV).



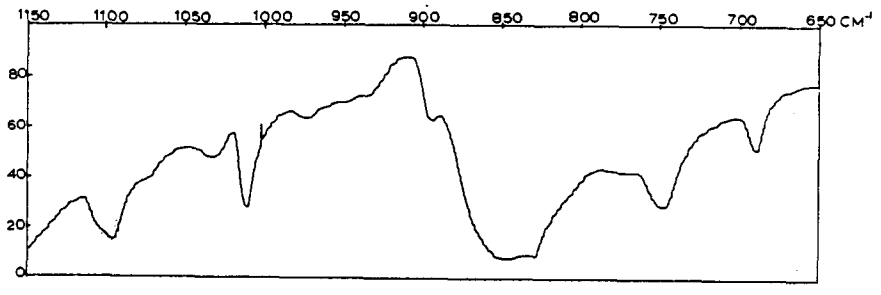
I



II



III



IV

FIG. 2. Infra-red spectra of some poly-phenylene ethers.

Polymer (III) was prepared by condensation of pure *p*-bromo sodium phenolate; the other (IV) is a copolymer of 80 per cent of *p*-bromo sodium phenolate and 20 per cent of the *ortho* isomer. In all model compounds an absorption of 1025 cm^{-1} is observed. In the "all *para*" oligomers (dimer → hexamer) the extinction of this peak gradually decreases, and in polymers prepared from *p*-bromo phenol such as (III) it is not observed at all. In *ortho*-linked phenylene ethers, on the other hand, the extinction of this 1025 cm^{-1} absorption remains virtually constant. In copolymers of *p*-bromo- and *o*-bromo phenol the 1025 cm^{-1} absorption is still visible in the i.r. spectra of polyphenylene ethers containing ~ 5 per cent of *ortho* links.

$$\left(\frac{p\text{-bromo phenol}}{o\text{-bromo phenol}} = \frac{95}{5}\right) \text{(cf. spectrum IV).}$$

An indication of branching is the absorption at 970 cm^{-1} (1,2,4-substitution); again in the homopolymer this band is hardly visible. From the spectra it may be concluded that (III) is essentially linear.

Elementary analysis

TABLE 1. ELEMENTARY ANALYSES OF POLYPHENYLENE OXIDE

Polymer	C (%)	H (%)	Br (%)	$\eta_{rel.}$
III	78·1	4·55	0·01	1·91
IV	77·6	4·57	0·54	1·19
Theor.	78·3	4·38	0·00	

Invariably, too high values for the hydrogen contents of the polymers are found, while the bromine numbers are too low. The i.r. spectra of nearly all the polymers show monosubstituted benzene nuclei (CH out of plane bending vibrations at 749 and 685 cm^{-1}). Apparently, reduction of the C–Br bond is one of the side reactions (cf. 14).

X-ray analysis[30]

X-ray examination of both untreated powder and compression-moulded quenched film which was annealed at 120–250°, yielded diffraction patterns with characteristic sharp rings, demonstrating that these samples were isotropically crystalline. An Astbury diffraction pattern of an oriented crystalline sample is shown in Fig. 3.
A comparable diagram, registered on a cylindrical instead of on a flat film, showed 25 different reflexion spots. All these reflexions could be indexed satisfactorily by assuming an orthorhombic unit cell with: $a = 5{\cdot}54 \pm 0{\cdot}05$ Å, $b = 8{\cdot}07 \pm 0{\cdot}05$ Å, $c = 9{\cdot}72 \pm 0{\cdot}05$ Å. Systematic extinctions reveal that the primitive cell has a glide-plane parallel to (001) with a translation 1/2 $(\overrightarrow{a} + \overrightarrow{b})$, and a twofold screw axis in the *c* direction (direction of orientation). Consequently the polymer chain has a zig-zag conformation in the crystal, the bond angle at the oxygen atom being approximately

121°. A reasonable value for the crystallographic density results only in the case of four oxyphenylene units per cell, viz. 1·407 ± 0·009 g/cm³.
The packing in the cell might approximately be as depicted in Fig. 4.

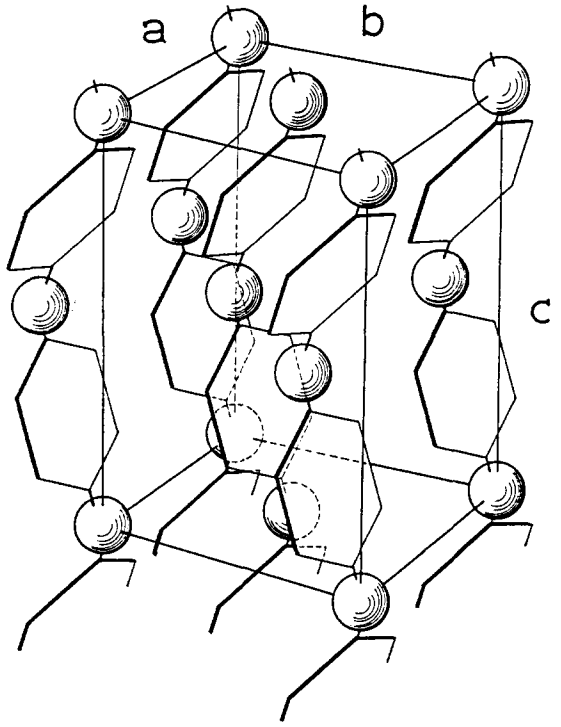


FIG. 4. A possible packing of the unit cell of poly-*p*-phenylene oxide.

The density of amorphous polyphenylene oxide amounts 1·27 ± 0·01 g/cm³ at 20°. Thus, the degree of crystallinity can be determined by simple density measurements; an originally amorphous film, annealed for 1 hr at 250° *in vacuo*, was found to be 28 per cent crystalline.

T_g, T_c and T_m

Poly-*p*-phenylene oxide is obtained as a crystalline powder. Compression-moulding and subsequent quenching in water at room temperature result in transparent, somewhat brownish amorphous films. The absorption spectrum of such a film is shown in Fig. 5.

The films are tough for polymers of $\eta_{rel.}$ above 1·25. The thermal behaviour of powder and film is illustrated by a DTA graph (Fig. 6).

The following values have been found for the best samples: $T_g = 82°$, $T_c = 112°$, $T_m = 298°$. The values of T_m are very sensitive to *ortho* linking of polymer chains, as illustrated for a number of polymers in Table 2.

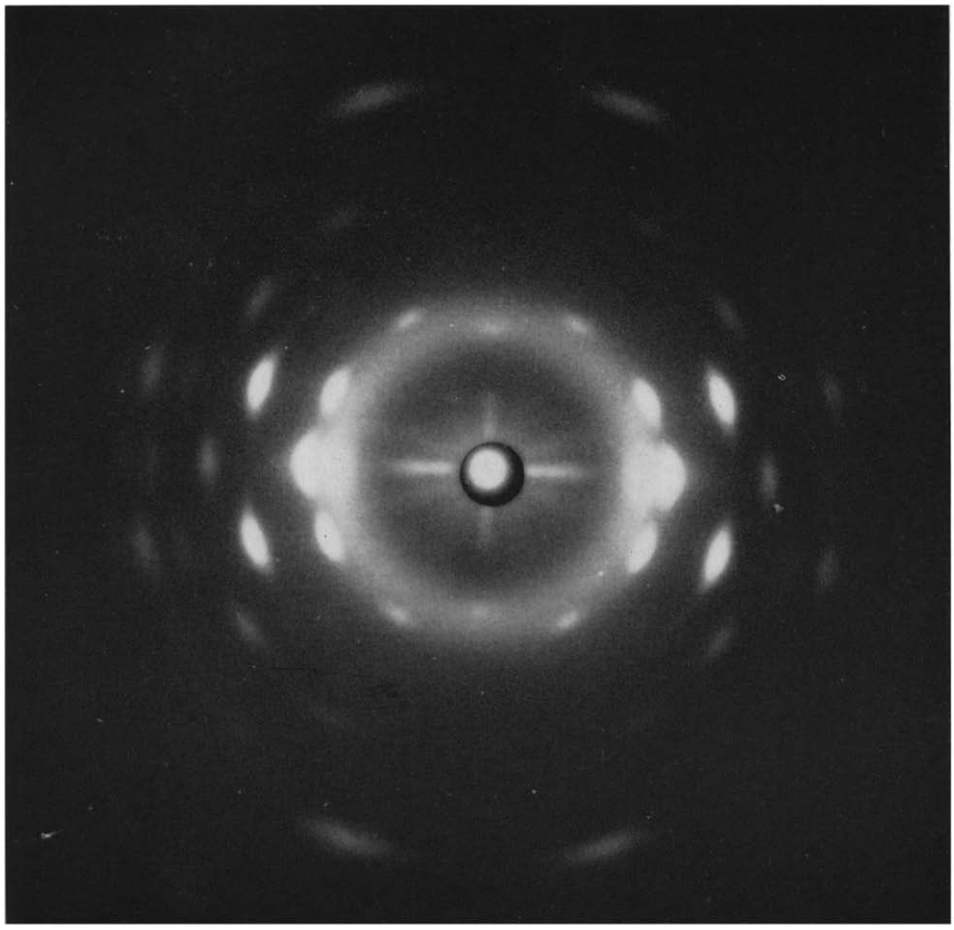


FIG. 3. Astbury diffraction pattern of oriented crystalline poly-*p*-phenylene oxide.

[*facing p. 280*]

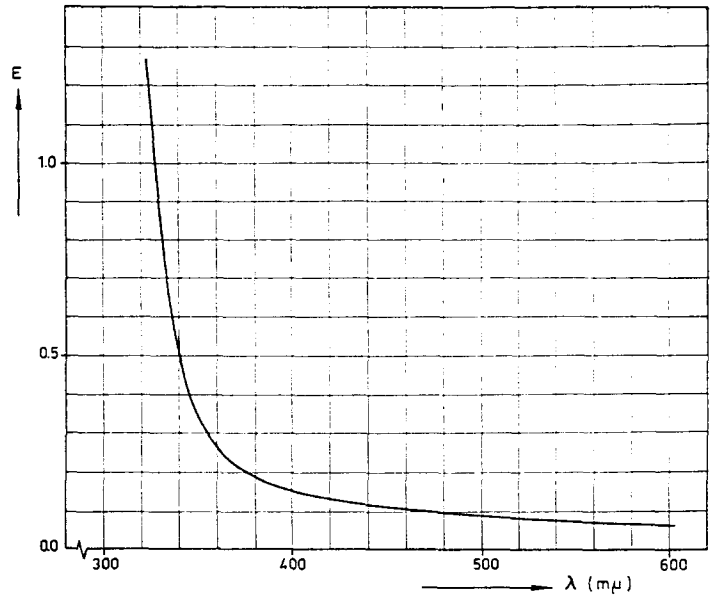


FIG. 5. The absorption spectrum of a poly-*p*-phenylene oxide film (Thickness: 17 μ).

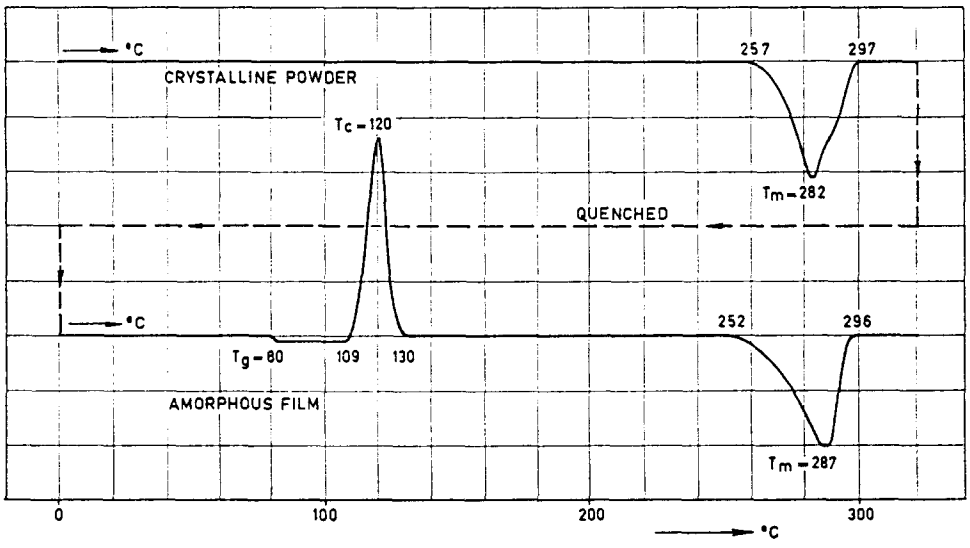


FIG. 6. Thermal behaviour of poly-*p*-phenylene oxide.

TABLE 2. MELTING POINTS (T_m) OF VARIOUS POLY-PHENYLENE OXIDE SAMPLES

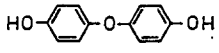
Polymer No.	Monomer composition: *p*-bromo-phenol (%)	Monomer composition: *o*-bromo-phenol (%)	$\eta_{rel.}$ nitrobenzene	T_m (°C)
5	100		1·10	297
6	100		1·83	298
7	90	10	1·09	247
8	90	10	1·19	247
9	80	20	1·09	200
10	70	30	1·09	143

Solubility

The polymer is soluble in boiling nitrobenzene, benzophenone, diphenyl oxide, N-methyl-pyrrolidone, tetralin, naphthalene and hexamethyl phosphoric triamide. Generally the solutions are stable at temperatures above 140–150°. Metastable, clear solutions of the polymer at room temperature can sometimes be obtained by pouring a hot (~10 per cent) solution of the polymer in hexamethyl-phosphoric-triamide into, e.g., a twofold amount of the same solvent of room temperature or lower.

Thermal stability in a N_2-atmosphere

The temperature (T_d) at which the weight of the sample in a nitrogen atmosphere had decreased by 2 per cent was determined with the aid of a thermo balance (heating rate 4°/min). Benzoylated polymers prepared with the aid of small amounts of phenol in order to remove the C–Br end groups were the most stable ones ($T_d \sim 450°$). An unesterified sample with a relatively high amount of C–Br end groups had a T_d of 390°. The T_d values of other polymers were between these two extremes. The decomposition products were isolated by means of thin layer chromatography and their structures were determined by i.r.-analysis. In the case of an unesterified sample, the following groups of compounds were found:

(1) phenol, *p*-phenoxyphenol, and higher oligomers
(2) compounds with two hydroxyl groups, such as: 
(3) one compound with a carbonyl group (cf. Ref. 31).

Heating of samples of both esterified and uncapped polymers for 30 min *in vacuo* at 320° does not lead to a change in relative solution viscosities, provided this viscosity is below ~ 1·25.

At higher values of $\eta_{rel.}$, an increase is observed and for values of $\eta_{rel.} > 1{\cdot}45$ the polymers become insoluble (Table 3).

TABLE 3. THE INFLUENCE OF HEATING ON THE SOLUTION VISCOSITY OF BENZOYLATED POLYMERS

Polymer No.	Solution viscosity		Remarks
	before heating	after heating	
11	1·10	1·09	Not benzoylated
12	1·17	1·18	Not benzoylated
13	1·28	1·29	Not benzoylated
14	1·35	1·38	—
15	1·36	1·42	—
16	1·44	1·72	Partly insoluble
17	1·44	—	Insoluble
18	1·77	—	Insoluble
19	2·11	—	Insoluble

The solution viscosity of sample 15 is plotted as a function of heating time in Fig. 7.

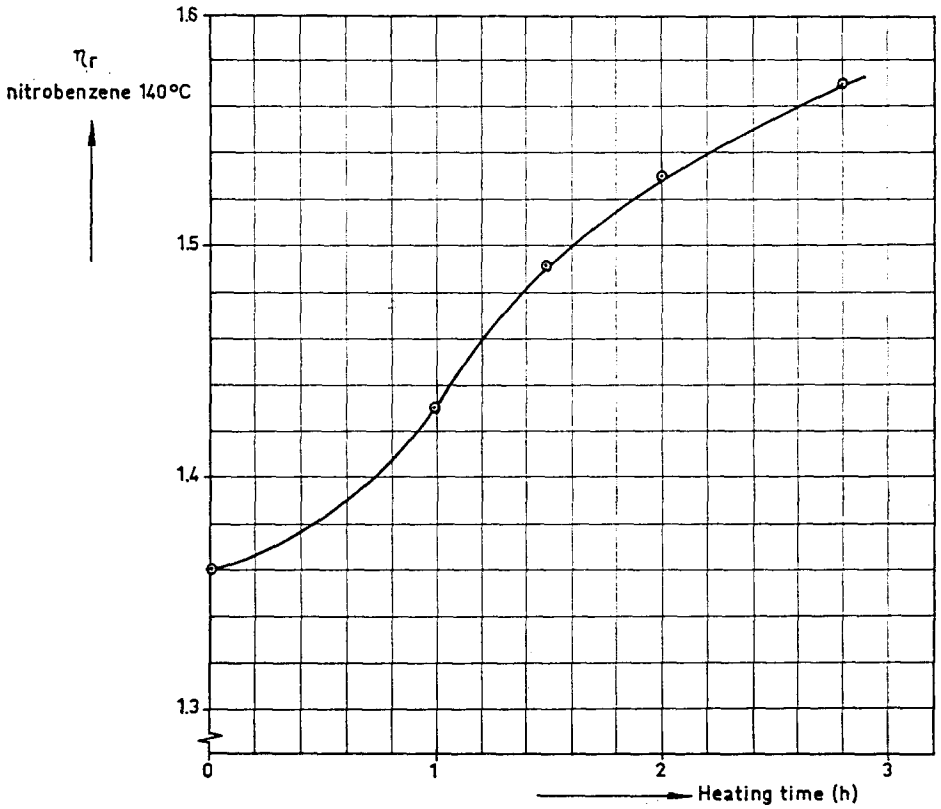


FIG. 7. The solution viscosity of poly-*p*-phenylene oxide as a function of heating time ($T = 320°$).

The melt viscosities of two polymers as a function of heating time are shown in Fig. 8.

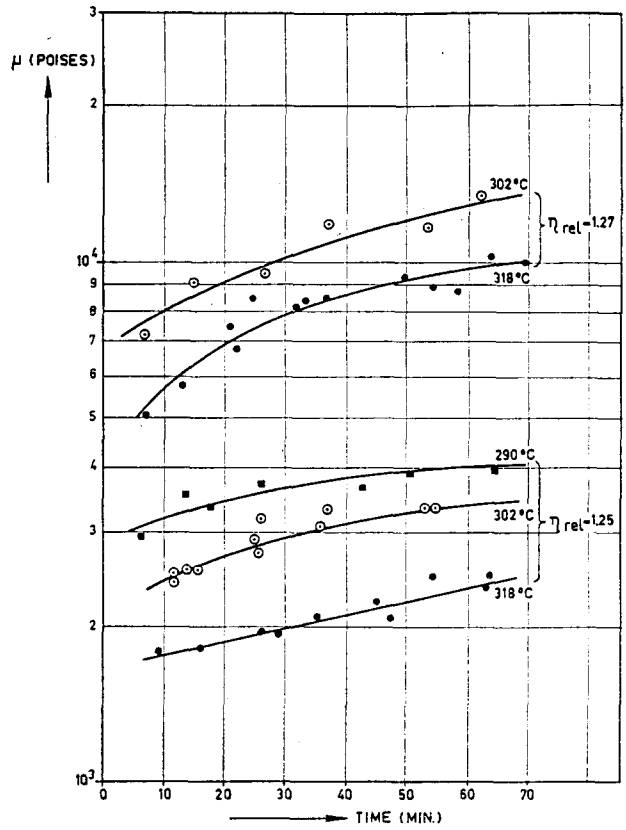


FIG. 8. Melt viscosity of poly-*p*-phenylene oxide as a function of time.

Within a short time (45 min) the values of the melt viscosity of polymers with an original value of $\eta_{rel.} > 1{\cdot}35$ become too high to be measured by our method.[29]

All data lead to the conclusion that, although the thermal stability in terms of weight loss of poly-*p*-phenylene oxide is high, the polymers are cross-linking at temperatures over 300°.

Oxidative stability

No oxygen was consumed when a sample of poly-*p*-phenylene oxide of which the end groups had been benzoylated was heated for 600 hr at 180° in an oxygen atmosphere; even on heating at 295° no increase in weight could be observed by TGA. After a heating period of 30 min at 320° in air, most of the polymer powders could no longer be compression-moulded into films. The oxidative stability of uncapped polymer is worse; completely cross-linked material (no flow at 320°) is obtained within 5 min at 320°.

Tensile strength of film and yarn

The elongation at break and the ultimate strength of compression-moulded films which were oriented near the T_g were determined at room temperature. In illustration, some of the results obtained with a relatively high molecular polymer ($\eta_{rel.} = 1{\cdot}90$) are given in Table 4.

TABLE 4. ELONGATION AT BREAK AND ULTIMATE STRENGTH OF POLY-*p*-PHENYLENE OXIDE FILMS

Drawing		Elongation at break (%)	Ultimate strength (kg/cm²)
temp. (°C)	ratio		
90	2	9·0	1070
90	2	7·0	900
93	2·7	14·0	1330
93	2·7	10·0	1230

As mentioned already, the stability of the melt is poor for polymers with a $\eta_{rel.} > 1{\cdot}30$ (p. 284). Tough films, on the other hand, are only obtained on compression-moulding of polymers with $\eta_{rel.} > 1{\cdot}25$. For melt-spinning experiments, polymer samples with $\eta_{rel.}$ between 1·25 and 1·30 were chosen. The desired polymers were prepared by using small amounts (0·5–1·0 mole per cent) of *p*-phenoxy phenol as a modifier in the polymerization. We succeeded in producing melt-spun yarn from a number of these polymers; the tensile strengths of these yarns are given in Table 5.

TABLE 5. STRENGTH OF MELT-SPUN YARN OF POLY-*p*-PHENYLENE OXIDE ($\eta_{rel.} = 1{\cdot}28$)

Draw ratio		Total draw ratio	Strength (g/tex)*
Pre-stretching at 93°	After stretching at 150°		
4·3	—	4·3	12·0
2·6	1·7	4·4	10·9
4·8	—	4·8	10·5
2·6	1·2	3·1	9·3
3·4	—	3·4	6·0
3·4	—	3·4	6·2
2·8	—	2·8	4·9

* The tex of a yarn is the weight in grams of 1000 metres of the yarn.

DISCUSSION

When starting this investigation, we expected poly-*p*-phenylene oxide to be a highly crystalline and thermostable polymer. In fact the polymer seems to cross-link, even in a nitrogen atmosphere, at temperatures over 290°. We are not sure whether this

thermal behaviour is an intrinsic property of the polymer or not. All samples contained small amounts of copper (1–3 ppm) and sodium (up to 100 ppm), which could not be removed completely by repeated precipitation of the polymer from hexamethyl phosphoric triamide solutions into acetone, methanol or water. These and other purifications did not improve the colour or the thermal stability of the polymer. Presumably, the thermal cross-linking is connected with the presence of some residual OH end groups* and/or copper and absorbed oxygen in the benzolyated polymers. The instability of the melt is a major obstacle in the further study of the properties of this polymer which is insoluble under normal conditions.

Another serious drawback in the study of this polymer is the irreproducibility of the Ullmann polycondensation. We studied the influence of the reaction temperature (170–300°) and the concentration of the monomer (10–40 per cent by weight) on both the course of the reaction in 1,4-dimethoxy benzene and the structure and properties of the polymers. At a reaction temperature of 170° the rate of the polycondensation is about seven times as low as at 200° (total reaction time $\sim$ 24 hr). At this low temperature the polymer has a tendency to crystallize during the reaction and to become insoluble; in most cases the polymers obtained were linear but of low molecular weight ($\eta_{rel.}$ 1·10–1·20). At temperature over 210° and also at high concentrations (>30 per cent) side reactions become more important [e.g. C–C coupling, branching, some cross-linking and reduction of the C–Br bond (cf. Ref. 14)]. We used a number of other solvents in the polycondensation, such as benzophenone, dimethyl formamide and hexamethyl phosphoric triamide, but the best results were obtained with 1, 4-dimethoxy benzene. In the experimental part the optimum reaction conditions are given. Although we obtained reproducible results in this way on a 20 g scale, we always had trouble with the reproducibility in experiments on a larger scale (>200 g).

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* We did not detect OH end groups by i.r. analysis in polymers of which the OH end groups had been benzoylated.

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(30) Full details will be given in a forthcoming paper by J. Boon and E. P. Magré on the X-ray analyses of a number of linear polyphenylene ethers.
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Résumé—Un poly(*p*-oxyde de phénylène) linéaire peut être préparé par condensation d'Ullman du *p*-bromophénolate de sodium. Le polymère se présente comme une poudre cristalline de couleur ivoire. En moulant ce polymère par compression à 320° et en trempant le produit obtenu dans l'eau on obtient des films transparents de couleur ambrée et présentant une bonne tenue mécanique. ($T_g = 83°$, $T_{cryst.} = 112°$ et $T_m \sim 290°$) La structure cristalline du polymère a pu être déterminée par analyse aux rayons X. On a étudié la stabilité du polymère à l'oxydation et à la chaleur et on a déterminé la tension de rupture de films orientés et de fibres filées par fusion.

Sommario—Il poli-*p*-fenilen ossido lineare può essere preparato con una condensazione di Ullman del *p*-bromo fenolato sodico. Il polimero è ottenuto come polvere cristallina di color avorio. Per fusione sotto pressione del polimero a 320° seguendo una tempra in acqua si ottengono film trasparenti color ambra amorfi ($T_g = 83°$, $T_{cryst} = 112°$ e $T_m \sim 290°C$). La struttura cristallina del polimero è stata quasi completamente determinata grazie ad analisi ai raggi X. Sono state studiate la stabilità alla temperatura e all'ossidazione del polimero e sono state determinate la resistenza alla tensione dei film orientati e delle fibre ottenute dal polimero fuso.

Zusammenfassung—Lineares Poly-*p*-phenylenoxid kann durch eine Ullman Kondensation von *p*-Brom-natriumphenolat hergestellt werden. Das Polymere wird als ein kristallines, elfenbeinfarbenes Pulver erhalten. Die Formung dieses Polymeren unter Druck bei 320° und nachfolgendes Abschrecken in Wasser liefert bernsteinfarbendurchsichtige und zähe amorphe Filme ($T_g = 83°$, $T_{krist.} = 112°$ und $T_m \sim 290°$). Die kristalline Struktur des Polymeren wurde weitgehend durch Röntgenanalyse aufgeklärt. Die thermische und oxidative Stabilität des Polymeren wurden untersucht und die Zerreissfestigkeit orientierter Filme und aus der Schmelze gesponnener Fasern wurde bestimmt.