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# Syntheses of new poly(amide–imide)s: 13. Preparation and properties of poly(amide–imide)s based on the diimide–diacid condensed from 3,3-bis[4-(4-aminophenoxy)phenyl]phthalide and trimellitic anhydride

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An imide ring-containing dicarboxylic acid, 3,3-bis[4-trimellitimidophenoxy)phenyl]phthalide, was prepared by the condensation of 3,3-bis[4-(4-aminophenoxy)phenyl]phthalide and trimellitic anhydride. A series of new aromatic bis(phenoxy)phthalide-containing poly(amide–imide)s having inherent viscosities of 0.84–1.10 dl g$^{-1}$ were prepared by the direct polycondensation of this diimide–diacid with various aromatic diamines using triphenyl phosphite and pyridine as condensing agents in *N*-methyl-2-pyrrolidone (NMP) in the presence of calcium chloride. Most of the resulting polymers showed an amorphous nature and were readily soluble in polar solvents such as NMP and *N*,*N*-dimethylacetamide. All the soluble poly(amide–imide)s afforded transparent, flexible and tough films. The glass transition temperature of these polymers were in the range of 250–318°C and showed almost no weight loss up to 450°C under air or nitrogen atmosphere.

**(Keywords: poly(amide–imide)s; polycondensation; triphenyl phosphite)**

## INTRODUCTION

The major limitations of aromatic polyimides for versatile applications are their high melting points and insolubility in organic solvents[1,2]. To overcome these drawbacks, some copolymers have been proposed, in particular for poly(amide–imide)s. This class of polymers seems to offer a good compromise between thermostability and processability[3–6]. Conventionally, aromatic poly(amide–imide)s can be prepared in several ways starting from trimellitic anhydride (TMA): first, two-step polycondensation from the acid chloride of TMA with aromatic diamines involving polyaddition and subsequent cyclodehydration[7], second, low-temperature-solution polycondensation of TMA-derived imide ring-preformed diacid chlorides and aromatic diamines[8–13], and third, polycondensation of TMA or TMA-derived imide ring-containing dicarboxylic acids with diisocyanates[14–16]. Recently, we have successfully applied phosphorylation reaction[17] to the synthesis of high-molecular-weight poly(amide–imide)s by the direct polycondensation of imide-containing dicarboxylic acids with aromatic diamines using triphenyl phosphite and pyridine as condensing agents[18–30]. The direct polycondensation route avoids using moisture-sensitive acid chlorides or isocyanates and provides significant advantages in manufacturing operations compared with conventional methods.

It was reported that the polymer chain having a cyclic side cardo group (for example, a fluorene, a phthalide or a phthalimidine group) can cause a significant increase in both glass transition temperature and thermal oxidative stability while providing good solubility[31,32]. On the other hand, incorporation of an aryl ether linkage into a polymer macrochain is known to impart processability to the polymer with little reduction in thermal stability[33,34]. Very recently, we have successfully synthesized soluble, high-temperature aromatic cardo polyamides and polyimides[35–37].

In a continuation of this study, the present article describes the synthesis of a series of novel poly(amide–imide)s from TMA, 3,3-bis[4-(4-aminophenoxy)phenyl] phthalide and other aromatic diamines. The solubility, tensile properties, crystallinity and thermal properties of the obtained polymers will also be investigated.

## EXPERIMENTAL

### *Materials*

3,3-Bis[4-(4-aminophenoxy)phenyl]phthalide (**3l**) was synthesized according to the method reported[36]. TMA (from Wako) and triphenyl phosphite (from TCI) were

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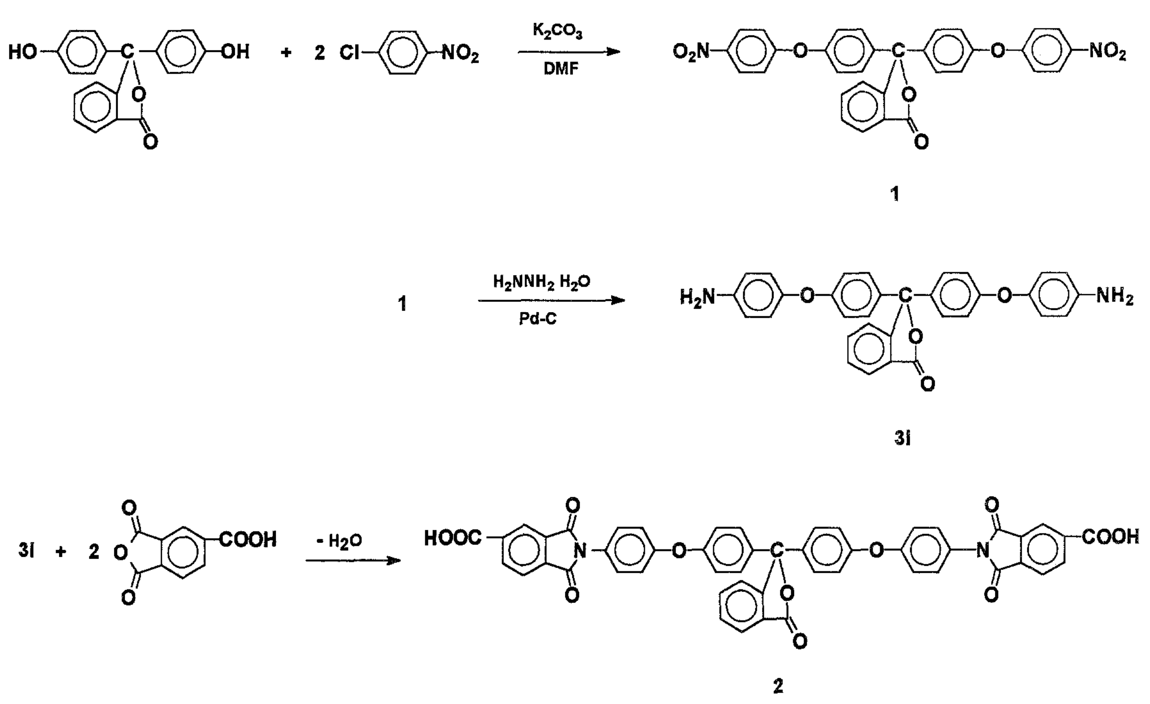


Scheme 1

used without previous purification. Commercially obtained anhydrous calcium chloride was dried under vacuum at 150°C for 6 h. *N*-Methyl-2-pyrrolidone (NMP), *N*,*N*-dimethylformamide (DMF) and pyridine were purified by distillation under reduced pressure over calcium hydride and stored over 4 Å molecular sieves. *p*-Phenylenediamine (**3a**; from Wako) and *m*-phenylenediamine (**3b**; from TCI) were vacuum-distilled before use. All other diamines, such as 2,4-diaminotoluene (**3c**; from TCI), 4,4′-methylenedianiline (**3d**; from TCI), 4,4′-oxydianiline (**3e**; from TCI); 3,4′-oxydianiline (**3f**; from Teijin Ltd, Tokyo), 4,4′-thioldianiline (**3g**; from TCI), 1,4-bis(4-aminophenoxy)benzene (**3h**; from TCI), 1,3-bis(4-aminophenoxy)benzene (**3i**; from Chriskev), 2,2-bis[4-(4-aminophenoxy)phenyl]isopropane (**3j**; from Chriskev) and bis[4-(4-aminophenoxy)phenyl]sulfone (**3k**; from Chriskev), were used with further purification.

### *Synthesis of 3,3-bis[4-(4-trimellitimidophenoxy)phenyl] phthalide (2)*

A mixture of 7.68 g (0.04 mol) of TMA and 10 g (0.02 mol) of 3,3-bis[4-(4-aminophenoxy)phenyl]phthalide was dissolved in 40 ml of dry dimethylformamide (DMF) at 60°C and stirred for 1 h. About 30 ml of toluene was then added, and the mixture was heated with reflux for 3 h until about 0.7 ml of water was distilled off azeotropically under a Dean-Stark trap. Heating was continued to distil off the residual toluene. After cooling, 30 ml of methanol was added and the yellow precipitate of diimide–diacid **2** was isolated by filtration and washed with methanol. It was purified by recrystallization from DMF–water. The purified product was dried in vacuum, and the mass of the solid thus obtained was 16.13 g (95%, m.p. = 279°C). I.r. (KBr) (cm$^{-1}$) 3462 (acid–OH), 1773 (imide I, symmetric C=O stretching), 1725 (acid C=O stretching and asymmetric imide C=O stretching), 1383 (imide II, imide ring vibration, axial), 1118 (imide III, imide ring vibration, transverse), 729 (imide IV, imide ring vibration, out-of-plane). Analysis calculated for $C_{50}H_{28}N_2O_{12}$: C, 70.75; H, 3.33; N, 3.30. Found: C, 69.86; H, 3.81; N, 3.62.

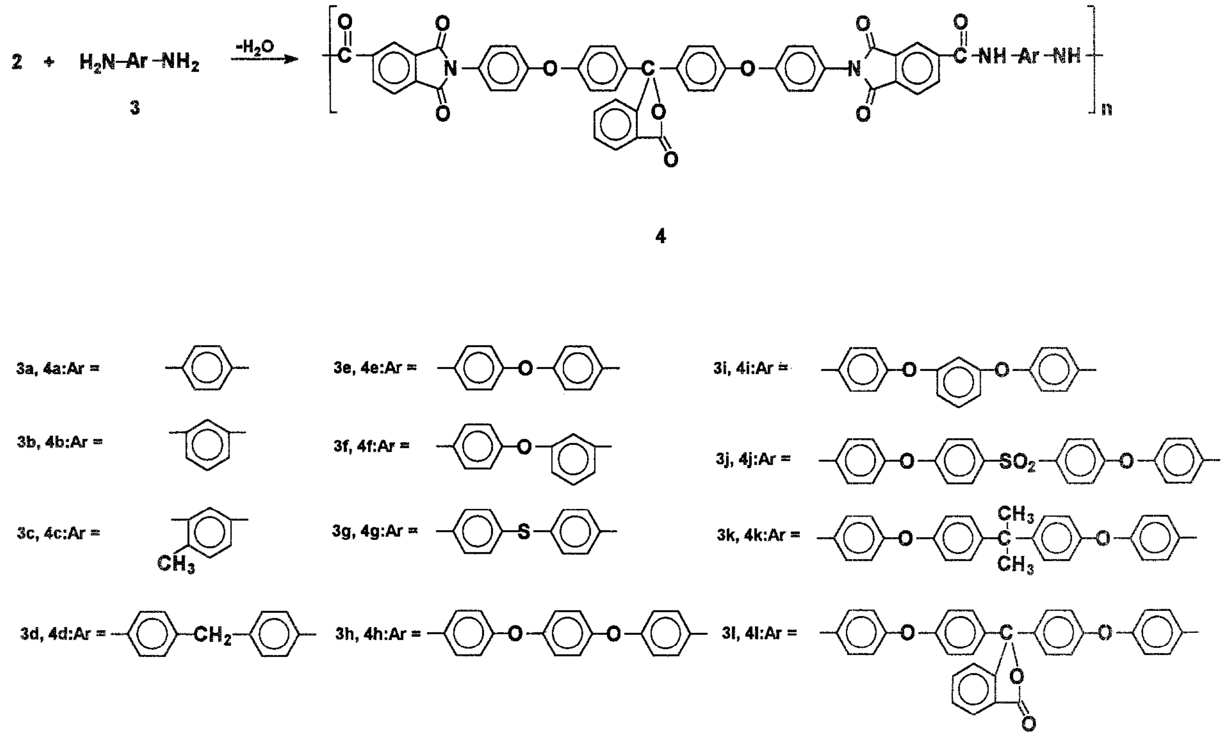
### *Polymerization*

A typical example of polycondensation is as follows. A mixture of 1.061 g (1.25 mmol) of diimide–diacid **2**, 0.25 g (1.25 mmol) of 4,4′-oxydianiline (**3e**), 0.5 g of calcium chloride, 5.5 ml of NMP, 0.8 ml of pyridine and 1.2 ml of triphenyl phosphite was heated with stirring at 100°C under nitrogen for 3 h. The polymer solution was too viscous to stir after 1 h and was diluted gradually with an additional 4 ml of NMP to maintain smooth stirring. The obtained polymer solution was trickled into 500 ml of stirring methanol. The stringy polymer was washed thoroughly with methanol and hot water, collected by filtration and dried at 100°C under vacuum. The yield was quantitative. The inherent viscosity of the polymer was 0.97 dl g$^{-1}$, measured at a concentration of 0.5 g dl$^{-1}$ in *N*,*N*-dimethylacetamide (DMAc) at 30°C. I.r. (film) (cm$^{-1}$) 3342 (N–H), 1775 (imide I, symmetric C=O stretching), 1725 (acid C=O stretching and asymmetric imide C=O stretching), 1671 (amide, C=O stretching), 1106 (imide III, ring vibration, transverse), 727 (imide IV, imide ring vibration, out-of-plane). Analysis calculated for $C_{62}H_{36}N_4O_{11}$: C, 73.51; H, 3.58; N, 5.53. Found: C, 70.44; H, 3.75; N, 5.30.

Other poly(amide–imide)s were synthesized in an analogous manner.

### *Measurements*

I.r. spectra were recorded on a Jasco FT/IR-7000 Fourier transform i.r. spectrometer. Elemental analyses were run in a Perkin-Elmer model 240 C, N, N analyser. Inherent viscosities of all polymers were determined at 0.5 g dl$^{-1}$ concentration using a Cannon-Fenske viscosimeter. Thermogravimetric data were obtained on a Dupont 951 thermogravimetric analyser coupled to a Dupont 2000 thermal analyser. Measurements were performed with 10 ± 2 mg samples heated in flowing



Scheme 2

nitrogen (50 cm$^3$ min$^{-1}$) at a heating rate of 20°C min$^{-1}$. Differential scanning calorimetry (d.s.c) analyses were performed on a Sinku Riko DSC-7000 differential scanning calorimeter coupled to a Sinku Riko TA-7000 thermal analysis controller in following nitrogen (30 cm$^3$ min$^{-1}$) at a heating rate of 20°C min$^{-1}$. Wide-angle X-ray diffractograms were obtained on a Rigaku Geiger Flex D-Max IIIa, using Ni-filtered Cu-K$_\alpha$ radiation (40 kV, 15 mA), and the scanning rate was 4° min$^{-1}$. Measurements were performed with film specimens 0.5 cm wide, 6 cm long and about 0.1 mm in thickness. An Instron Universal Tester model 1130 with a load cell of 5 kg was used to study the stress–strain behaviour of the samples. A gauge of 2 cm and a strain rate of 5 cm min$^{-1}$ were used for this study. Measurements were performed at room temperature (~ 20°C) and an average of at least five individual determinations was used.

**Table 1** Preparation of poly(amide–imide)s from diimide–diacid **2** and various aromatic diamines[a]

| Polymer | Initial amount of NMP (ml) | Additional amount of NMP (ml) | Amount of $CaCl_2$ (g) | $\eta_{inh}$[b] (dl g$^{-1}$) |
|---|---|---|---|---|
| **4a** | 5.5 | 6.0 | 0.40 | 1.04[c] |
| **4b** | 5.0 | 3.5 | 0.32 | 1.00 |
| **4c** | 3.7 | 2.0 | 0.30 | 0.84 |
| **4d** | 5.0 | 3.0 | 0.33 | 0.95 |
| **4e** | 5.5 | 4.0 | 0.50 | 0.97 |
| **4f** | 5.5 | 4.0 | 0.50 | 0.98 |
| **4g** | 5.0 | 4.0 | 0.50 | 1.04 |
| **4h** | 5.5 | 4.0 | 0.50 | 0.92 |
| **4i** | 5.5 | 1.5 | 0.50 | 1.10 |
| **4j** | 5.5 | 3.0 | 0.50 | 0.93 |
| **4k** | 5.0 | 4.5 | 0.32 | 0.97 |
| **4l** | 5.5 | 4.0 | 0.55 | 0.97 |

[a] Polymerization was carried out with 1.25 mmol of each monomer, 1.2 ml of triphenyl phosphite, 0.8 ml of pyridine, and calcium chloride in NMP at 100°C for 3 h

[b] Measured at a concentration of 0.5 g dl$^{-1}$ in DMAc at 30°C

[c] Measured at a concentration of 0.5 g dl$^{-1}$ in DMAc–5% LiCl at 30°C

## RESULTS AND DISCUSSION

### *Syntheses*

The diamine, 3,3-bis[4-(4-aminophenoxy)phenyl] phthalide (**3l**), was synthesized in two steps, first from the nucleophilic displacement of the activated chlorine atoms of *p*-chloronitrobenzene with bisphenoxide ions of phenolphthalein, giving rise to 3,3-bis[4-(4-nitrophenoxy) phenyl]phthalide, followed by hydrazine/Pd–C reduction[36] (*Scheme 1*). 3,3-Bis[4-(4-trimellitimidophenoxy) phenyl]phthalide (**2**), the monomer with preformed imide rings, was prepared by condensation of the diamine **3l** with two equivalent amounts of TMA, followed by cyclodehydration to the imidodicarboxylic acid by means of toluene–water azeotropic distillation.

A series of novel poly(amide–imide)s (**4a–l**) containing phthalide and arylene ether units were prepared from diimidodicarboxylic acid **2** and various aromatic diamines **3a–l** by the direct polycondensation reaction using triphenyl phosphite and pyridine as condensing agents (*Scheme 2*). Synthesis conditions and inherent viscosities of the produced polymers are shown in *Table 1*. Under these conditions the reaction solutions were homogeneously transparent, and the inherent viscosities of obtained poly(amide–imide)s ranged from 0.84 to 1.10 dl g$^{-1}$. In each case, 3.7–5.5 ml of the initial dosage of NMP was used, and an additional volume of 1.5–6.0 ml of NMP was gradually added to the reaction mixture after 1 h before gelation took place. The solubility of the polymer solution and the state of stirring affected the inherent viscosity of the resulting poly(amide–imide) significantly. Generally, the molecular weight of the polymer obtained from the

**Table 2** Solubility of poly(amide–imide)s[a]

| | Solvent[b] | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Polymer | DMAc | DMF | DMSO | NMP | *m*-Cresol | *o*-Chlop | Py | DMAc–5% LiCl |
| **4a** | − | − | − | + | − | − | − | − |
| **4b** | + | + | + | + | + | + | + | + |
| **4c** | + | + | + | + | + | + | + | + |
| **4d** | + | + | + | + | + | + | + | + |
| **4e** | + | + | + | + | + | + | + | + |
| **4f** | + | + | + | + | + | + | + | + |
| **4g** | + | + | + | + | + | + | + | + |
| **4h** | + | + | + | + | + | + | + | + |
| **4i** | + | + | + | + | + | + | + | + |
| **4j** | + | + | + | + | + | + | + | + |
| **4k** | + | + | + | + | + | + | + | + |
| **4l** | + | + | + | + | + | + | + | + |

[a] Solubility: +, soluble at room temperature; −, insoluble

[b] DMAc, *N,N*-dimethylacetamide; DMF, *N,N*-dimethylformamide; DMSO, dimethylsulfoxide; NMP, *N*-methyl-2-pyrrolidone; *o*-Chlop, *o*-chlorophenol; Py, pyridine

phosphorylation reaction is highly dependent on the reactant concentration. Higher molecular weights of these polymers could be obtained by using a higher initial reactant concentration and adding a proper amount of supplemental NMP into the viscous reaction medium before the formation of swollen gel. The molecular weights of these polymers are sufficiently high to permit the casting of tough films.

### *Properties of polymers*

The qualitative solubility of the poly(amide–imide)s in various solvents is shown in *Table 2*. Most polymers were soluble in aprotic polar solvents such as DMAc, DMF and NMP, and even in less polar solvents such as *m*-cresol and *o*-chlorophenol; however, polymer **4a** with *p*-phenylene structure had somewhat limited solubility and was insoluble in most of the tested solvents, possibly because of the semicrystalline nature of this polymer. All the polymers are insoluble in common organic solvents such as tetrahydrofuran, chloroform, acetone and benzene. The superior solubility can apparently be attributed to the combined favourable effects of aromatic ether and phthalide linkages in the trimellitimide-based poly(amide–imide) backbone.

Most of the X-ray diffractograms of poly(amide–imide)s derived from imide-containing dicarboxylic acid **2**, except for polymer **4a**, exhibit amorphous patterns. The semicrystalline behaviour of polymer **4a** caused the insoluble nature in polar solvents, as mentioned above, which is in agreement with the general rule that the solubility decreases with increasing crystallinity. Transparent, flexible and tough films could be cast from the 10% (w/v) DMAC solutions of most of the obtained poly(amide–imide)s. The tensile properties of some typical poly(amide–imide)s are summarized in *Table 3*. The films had a tensile strength of 63–80 MPa, elongation to break of 6–26%, and initial modulus of 1.39–1.67 GPa. Except for the brittle behaviour of polymer **4a**, all the polymers listed in *Table 3* behave as ductile materials with good tensile strengths and moderate elongations-to-break.

The thermal stability of the poly(amide–imide)s was studied by thermogravimetric analysis (t.g.a.). The

**Table 3** Tensile and thermal properties of poly(amide–imide)s

| | | | | D.s.c. | | T.g.a. | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | $T_d$ (°C)[e] | | |
| Polymer[a] | Tensile strength (MPa) | Elongation at break (%) | Tensile modulus (GPa) | $T_g$[b] (°C) | $T_m$[c] (°C) | in air | in $N_2$ | Char yield[f] (wt%) |
| **4a** | – | – | – | 318[d] | 333 | 525 | 530 | 65 |
| **4b** | 75 | 10 | 1.67 | 290 | – | 530 | 543 | 65 |
| **4c** | 80 | 9 | 1.54 | 284 | – | 532 | 529 | 64 |
| **4d** | 74 | 26 | 1.44 | 284 | – | 504 | 530 | 61 |
| **4e** | 80 | 9 | 1.50 | 288 | – | 519 | 537 | 59 |
| **4f** | 73 | 9 | 1.49 | 269 | – | 527 | 544 | 61 |
| **4g** | 75 | 6 | 1.60 | 279 | – | 530 | 537 | 65 |
| **4h** | 74 | 8 | 1.61 | 268 | – | 533 | 552 | 64 |
| **4i** | 75 | 9 | 1.53 | 256 | – | 522 | 545 | 60 |
| **4j** | 63 | 9 | 1.39 | 250 | – | 514 | 527 | 61 |
| **4k** | 74 | 9 | 1.50 | 268 | – | 514 | 536 | 62 |
| **4l** | 78 | 9 | 1.53 | 276 | – | 521 | 530 | 63 |

[a] Films were cast from polymer solutions in DMAc

[b] From the first heating traces of d.s.c measurements conducted with a heating rate of 20°C min$^{-1}$ in nitrogen

[c] Temperature of the endotherm peak in the first heating trace of d.s.c.

[d] From the second heating traces of d.s.c measurements conducted with a heating rate of 20°C min$^{-1}$ in nitrogen

[e] Temperature at which a 10% weight loss was recorded by t.g.a at a hearing rate of 20°C min$^{-1}$ in nitrogen

[f] Char yield at 800°C in nitrogen

temperatures of 10% weight loss ($T_d$) in nitrogen and air atmospheres were determined from original thermograms and are tabulated in *Table 3*. In general, all the polymers exhibited good thermal stability with no significant weight loss up to temperatures of approximately 450°C in nitrogen; and their $T_d$ values were recorded in the range 527–552°C in nitrogen and 503–533°C in air. The amount of carbonized residue of the aromatic poly(amide–imide)s at 800°C in nitrogen atmosphere was higher than 59%. This is probably due to the fact that the phenolphthalein-derived polymers may produce a higher degree of crosslinking during pyrolysis, leading to higher char yield[38].

*Table 3* also summarizes the thermal transition data of the poly(amide–imide)s. Except for **4a**, a glass transition temperature ($T_g$) was observed for all polymers in the first heating curve in a temperature range of 250–290°C. The $T_g$ values of the polymers can be controlled by varying the bridge group of diamine residues. Polymers **4k** and **4l**, containing sulfone and lactone units, respectively, had higher $T_g$ values than polymer **4j**, which was derived from the diamine having isopropylidene linkages. This order is probably caused by the polar character of the sulfone group and bulky lactone unit. A melting ($T_m$) endotherm peak of polymer **4a** could be observed on its first d.s.c. trace. After quenching, a clear glass transition at around 318°C was detectable in the second heating trace.

## CONCLUSIONS

The diimide–diacid 3,3-bis[4-(4-trimellitimidophenoxy) phenyl]phthalide was used as a poly(amide–imide) building block. Poly(amide–imide)s having moderate to high molecular weight were successfully prepared by direct polycondensation of the diimide–diacide with various aromatic diamines by means of triphenyl phosphite and pyridine. Polymers, in general, showed good thermal stability and excellent solubility, and the cast films exhibited excellent tensile properties. Thus, the present poly(amide–imide)s are considered to be new candidates for processable high-performance polymeric materials.

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