Synthesis of Dicyanomethyl and Nitro Substituted *p*-Polyphenyls and Their Salts

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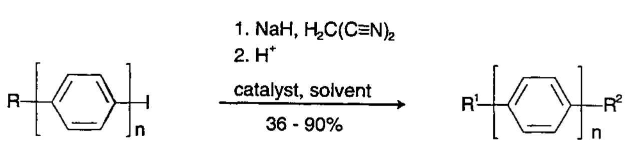
A one-step, medium-scale synthesis of several novel dicyanomethyl and nitro substituted *p*-polyphenyls from 4,4′- or 4″-diiodo-*para*-polyphenyls and from 4-iodo-4′- or 4″-nitro-*p*-polyphenyls, respectively, and sodium malononitrile using a palladium catalyst is reported. A number of organic cation salts of these extended π-electron systems are likewise described.

As part of an ongoing project involving a search for better and more stable organic conductors[2–10] and materials exhibiting non-linear optical properties[11–13] a rapid and efficient synthesis of extended π-electron systems consisting of the *p*-polyphenyl nucleus with dicyano and nitro groups in *para* positions was desirable.

Addison and co-workers[10] first synthesized 4,4′-bis(dicyanomethyl)biphenyl (**2**) in low overall yield via a four-step sequence starting from biphenyl. A more convenient method for preparing **2** was later developed by Uno and co-workers[14] whereby the latter compound was obtained in 57% yield in a one-pot reaction from 4,4′-diiodobiphenyl and the malononitrile anion. The aromatic substitution reaction catalyzed by bis(triphenylphosphine)palladium(II) chloride occurred over 33 hours in refluxing THF. Under more controlled conditions and with a shorter reaction period, the reaction gave us fewer side products and afforded **2** in 90% yield. Using a similar approach, we were able to synthesize the next higher homolog, 4″-bis(dicyanomethyl)-*p*-terphenyl (**4**) from **3** and the malononitrile anion in either THF or DMSO. Compound **4** was isolated as a light-green flaky material. Mass spectroscopic evidence was also obtained for the presence of 4″-iodo-*p*-terphenylmalononitrile ($M^+ = 420.251$) in small amounts in some crude samples of **4**. Therefore the reaction most probably occurs in two stages in which the substitution of iodine in **2** by a dicyanomethyl anion does not impede subsequent attack of the remaining iodine atom by an additional malononitrile anion. Compound **4** could not be dehydrogenated by any of the known oxidizing agents such as aqueous bromine, bromine in pyridine, hydrogen peroxide or even *N*-iodosucinimide in acetonitrile[4,5,7] to afford tetracyano-*p*-terphenoquinodimethane, an analog of TCNQ.

However, in the synthesis of the nitro substituted *p*-polyphenyls **6, 8** it was necessary to modify Uno's method. The above novel compounds could only be synthesized using higher reaction temperatures and the strongly polar aprotic solvent DMSO (Scheme 1). Thus, 4-(dicyanomethyl)-4′-nitrobiphenyl (**6**) and 4-(dicyanomethyl)-4″-nitro-*p*-terphenyl (**8**) were prepared from the appropriate iodonitro-*p*-polyphenyls[15,16] **5, 7** and sodium malononitrile. The presence of DMSO most likely favours hybrid formation in which the contribution of structure (Fig. 1) is less important, and the reactions proceed as expected but with considerably lower yield. All attempts to synthesize **6** and **8** in HMPA or acetonitrile were unsuccessful. Table 1 lists the substitution products obtained and their properties. The chloro and bromo analogs of all the aforementioned substrates (**3, 5, 7**) proved to be unreactive under similar reaction conditions which is in agreement with earlier work[14] regarding the decreasing order of the reactivity of halides, I > Br ≫ Cl, in similar nucleophilic substitution reactions.



Reactant	R	n
1	I	2
3	I	3
5	NO_2	2
7	NO_2	3

Product	R¹	R²	n
2	$CH(CN)_2$	$CH(CN)_2$	2
4	$CH(CN)_2$	$CH(CN)_2$	3
6	NO_2	$CH(CN)_2$	2
8	NO_2	$CH(CN)_2$	3

Scheme 1

Table 1. Reactions of Iodo- and Diiodo-*p*-polyphenyls with Malononitrile Anion in the Presence of Palladium Catalyst

Reactant	Catalyst molar ratio[a]	Solvent	Temp. (°C)	Reaction Time (h)	Product	mp (°C) (solvent)	Yield[b] (%)
3	25 : 1	THF	reflux	44	**4**	> 300 (MeCN)	73
3	50 : 1	DMSO	120	12	**4**	> 300 (DMSO/MeOH)	62
5	150 : 1	DMSO	120	28	**6**	148–150 (C_6H_6/n-C_7H_{16})	42
7	100 : 1	DMSO	120	12	**8**	212–214 (AcOH)	36

[a] Molar ratio of substrate to catalyst.

[b] Yield based on iodo- and diiodopolyphenyls.

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Table 2. Spectral Data of *p*-Polyphenylmalononitriles Prepared

Compound[a]	MS (m/z)	IR (KBr) ν (cm^{-1})	^{1}H NMR δ
4 $(NC)_2CH(C_6H_4)_3CH(CN)_2$	358.123	3050, 3040, 3010, 2890 (CH), 2265, 2260 (C≡N), 1490 (C=C), 1018, 1001, 830, 780 (CH)	4.7 (s, 2H), 7.6, 7.8, 7.9 (m, 12H)
6[b] $(NC)_2CH(C_6H_4)_2NO_2$	263.069	3074, 2857 (CH), 2215, 2145 (C≡N), 1576 (C=C), 1515, 1341 (NO_2), 856, 844 (CH)	4.5 (s, 1H), 6.9, 7.5 (m, 8H)
8 $(NC)_2CH(C_6H_4)_3NO_2$	339.100	3079, 3050, 3019 (C—H), 2260, 2210, 2195 (C≡N), 1600, 1595, 1501 (CH), 1510, 1347 (NO_2), 855, 828 (CH)	4.0 (s, 1H), 6.5, 7.5, 7.9 (m, 12H)

[a] All compounds including **3** gave satisfactory microanalyses: C ±0.29, H ±0.06, N ±0.09.

[b] X-ray data are available from the authors.

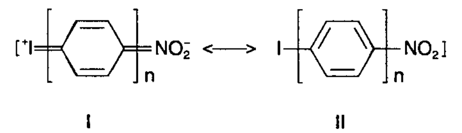

Figure 1. Hybrid structure of 4-iodo-4′/4″-nitro-*p*-polyphenyls

The tertiary hydrogens in **6** and **8** are sufficiently acidic to readily react by metathesis with weak bases such as tetrabutylammonium (TBA) hydroxide in aqueous methanol to afford the corresponding salts **11** and **12** (Table 3). However, salt **10** could only be obtained by first dissolving **4** in excess aq NaOH followed by treatment with slightly more than 2 equiv of TBA hydroxide. Salts of the anions of **11** and **12** were then prepared by displacement with various organic cations in solution, and readily precipitated from the reaction medium. Thus **13** and **14** were obtained from **11** and 4-*N*,*N*-dimethylanilinopyridinium chloride and methyl 4-*N*,*N*-dimethylanilinopyridinium iodide,[18] respectively. In solution, the organic cations of the above halides also combined with the anion in **12** to yield salts **15** and **16**. 4,4′-Bipiperidinium(2$^+$) 4″-nitro-*p*-terphenylmalononitrile (**17**) was prepared in much the same way but was difficult to purify. To this effect **17** had to be first dissolved in hot DMSO and then diluted with hot MeCN to a 1 : 1 vol. to yield a brick-red microcrystalline material on cooling.

We found the infrared absorption spectra taken in KBr significant in two aspects. Firstly, the highest C≡N stretching frequency in the dicyanomethyl substituted compounds **4** and **8** (n = 3) appear some 50–60 cm^{-1} higher than in **6** (n = 2) thus indicating that the π-electron system appears to be more localized in the *p*-terphenyl skeleton than in its lower homolog. This observation is in agreement with a bond length comparison study of the TBA salts of the above compounds using crystallographic analysis.[19] Secondly, in all the above reported *p*-polyphenylmalononitrile salts, the $\nu_{C=N}$ absorption bands consistently appear at 2175 cm^{-1} or lower, unlike

Table 3. Salts of *p*-Polyphenylmalononitriles Prepared

Salt[a]	Name	mp (°C) (solvent)	Yield[b] (%)	IR (KBr) ν (cm^{-1})
10	bis(tetrabutylammonium) *p*-terphenyl-4,4″-dimalononitrile	222–223 (MeCN)	89	3062, 3010, 2959, 2930, 2870 (CH), 2162, 2110 (C≡N), 1600 (C=C arom), 1490, 1315 (NO_2), 815 (CH)
11	tetrabutylammonium 4′-nitrobiphenylmalononitrile	132–133 (MeOH/H_2O)	100	3055, 3010, 2915, 2904, 2872 (CH), 2164, 2121 (C≡N), 1600 (C=C arom), 1583, 1338 (NO_2), 870 (CN), 850, 832, 821 (CH)
12	tetrabutylammonium 4″-nitro-*p*-terphenylmalononitrile	151–152 (MeCN/H_2O)	60	3060, 3020, 2960, 2930, 2870 (CH), 2165, 2120 (C≡N), 1600, 1590 (C=C arom), 1482, 1335 (NO_2), 850, 840, 815 (CH)
13	4-(*N*,*N*-dimethylanilino)pyridinium 4′-nitrobiphenylmalononitrile	216–218 (MeCN)	100	3080, 3037, 2921, 2875 (CH), 2460, 2372, 2338, 2303 (C—N$^+$—H), 2179, 2128 (C≡N), 1636 (C=N$^+$), 1590 (CH arom), 1493, 1329 (NO_2), 824, 799 (CH)
14	methyl 4-(*N*,*N*-dimethylanilino)pyridinium 4′-nitrobiphenylmalononitrile	257–259 (MeCN)	75	3062, 3010, 2950, 2910 (CH), 2175, 2138 (C≡N), 1640 (C=N$^+$), 1596, 1502 (C=C arom), 1572, 1320 (NO_2), 850, 810 (CH)
15	4-(*N*,*N*-dimethylanilinopyridinium 4″-nitro-*p*-terphenylmalononitrile	224–225 (MeCN)	71	3020, 2910 (CH), 2160, 2115 (C≡N), 1630 (C=N$^+$), 1585, 1490 (C=C), 1572, 1336 (NO_2), 850, 815, 804 (CH)
16	methyl 4-(*N*,*N*-dimethylanilinopyridinium) 4″-nitro-*p*-terphenylmalononitrile	268–270 DMF/MeCN	47	3070, 3030, 2940, 2890 (CH), 2160, 2118 (C≡N), 1595 (C=C arom), 1485, 1335 (NO_2), 850, 840, 810 (CH)
17	bipiperidinium(2$^+$) 4″-nitro-*p*-terphenylmalononitrile · DMSO	267–268 DMSO/MeCN	100	3062, 3010, 2956, 2920 (CH), 2810, 2740 (N^+H_2), 2170, 2120 (C≡N), 1590, 1500 (C=C), 1575 (N^+H_2), 1510, 1335 (NO_2), 1392, 1385 (*gem*-CH_3), 1035 (S=O), 812, 755 (CH)

[a] Satisfactory analyses obtained: C ±0.34, H ±0.11, N ±0.27, O ±0.26. X-ray data are available from the authors.

[b] Yield based on the TBA salt.

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Table 4. UV/VIS Data on **2**, **4**, **6**, **8** and Their TBA Salts in MeOH

Compound	λ_{max} (nm)	$\log \varepsilon$	Salt	λ_{max} (nm)	$\log \varepsilon$
2	224	4.48	**9**	222	4.65
	356	4.59			
4	224	3.31	**10**	227	4.56
	369	3.49		370	4.82
6	224	4.17	**11**	228	4.36
	299	4.25		299	4.29
	450	4.01		450	4.31
8	224	4.23	**12**	226	4.36
	318	4.37		323	4.40
	410	3.99		370	4.23
				405[a]	4.27

[a] Obtained in MeCN.

the molecular compounds where the stretching modes usually appear above 2200 cm^{-1}; thus indicating considerably more π-electron delocalization in the anions than in the molecular compounds.

Finally, we compared the UV/VIS data of all the prepared compounds and their TBA salts (Table 4) with those of biphenyl[20] and *p*-terphenyl.[21] In general, the absorption spectra of the neutral compounds and their TBA salts are almost identical which implies that intermolecular interaction is not significant.

Compounds **2**, **4**, **6**, and **8** show a primary absorption band at $\lambda = 224$ nm while those of the TBA salts appear at or near $\lambda = 226$ nm. The resonant energies in **2**, **4**, and in the dianions of salts **9** and **10** are lower than those of **6**, **8**, **11** and **12**. Evidently, π-electron delocalization is more extensive in the bis(dicyano)polyphenyls than in the nitro and dicyano substituted polyphenyls. Furthermore, in passing from the biphenyl to the *p*-terphenyl ring system, a bathochromic shift of ca. 20 nm occurs in the case of **2**, **4** and their TBA salts, whereas for **6**, **8** and their corresponding anions, a red shift of ca. 15 nm is observed. Of particular significance are the absorptions above 400 nm obtained for **6**, **8**, **11** and **12** which seem to indicate interaction between the polyphenyl ring system and the π-electrons of the nitro substituent. The fact that **6** and **11** absorb ca. 40 nm higher than **8** and **12** implies better π-electron delocalization and less twisting of the phenyl rings[10] in the former than in the latter. This probably explains the bathochromic shift in the highest $\nu_{C=N}$ of the infrared spectrum of **6** (*vide supra*). Furthermore, a comparison of the absorption intensities in the region above 400 nm for **11** and **12** with those of **6** and **8** indicates that in polar solvents the latter apparently dissociate into weak acids. Solutions of the nitro compounds and their TBA salts also exhibit a yellow to orange colour. This is particularily evident in the case of salt **11**. Consequently, the existence of a possible quinoidal structure for the anions of **11** and **12** cannot be completely disregarded.

In conclusion, the synthesis of *p*-polyphenyls substituted with dicyanomethyl and nitro groups in the para positions (**4**, **6**, **8**) was achieved in a one-pot reaction using a Pd catalyst and the strong polar aprotic solvent DMSO. A number of their salts were also prepared and described.

Melting points are uncorrected. ^{1}H NMR measurements were made on a Bruker AC-300 (300 MHz) using TMS as an internal standard. IR spectra, taken in KBr, were recorded on a Perkin-Elmer 683 spectrophotometer. UV/VIS measurements were made with a Beckman ACTA MIV spectrometer. Mass spectroscopic analysis were performed on a ZAB-1S spectrometer at 70 eV using a direct insertion technique. All reagents were purchased from Aldrich Chemical Co. and solvents were of ACS grade or better. Solvents and liquid reagents were dried in either 3 or 4A type molecular sieve for at least 24 h prior to experimentation.

4,4″-Diiodo-*p*-terphenyl (3):

A mixture of *p*-terphenyl (4.60 g, 20 mmol), iodine (5.1 g, 20 mmol) and H_5IO_6 (98 %, 1.83 g, 8 mmol) was added to a solvent mixture of $AcOH/H_2O/H_2SO_4$ (10:2:0.3 vol., 75 mL) and heated to 100 °C with stirring. A flesh coloured precipitate gradually formed and thickened and later more of the same solvent mixture was added (38 mL) in order to maintain efficient stirring. The reaction mixture was heated for 24 h and subsequently cooled and filtered. The resulting light-tan product was washed with 10 % aq sodium thiosulfate (500 mL) and then dried. The crude product was purified by refluxing with two separate portions of toluene (2 x 250 mL) each for 15 min. The dissolved product crystallized from the hot extracts in white flaky crystals. The undissolved fraction was then extracted in a Soxhlet apparatus for 24 h using toluene (150 mL) and upon cooling yielded more of the same crystalline product. Yield, 5.7 g (60 %); mp 327–328 °C (Lit.[17] mp 307–309 °C dec.).

MS (70 eV): $m/z = 481.903$.

IR (KBr): $\nu = 3040, 3024, 2920, 2850$ (CH), 1582, 1476, 1465 (C=C arom), 1070, 1065, 1010, 1000, 805 cm^{-1} (CH).

4,4″-Bis(dicyanomethyl)-*p*-terphenyl (4):

Method A:

Into a three-necked round-bottom flask (100 mL) fitted with a thermometer, bleeding tube and magnetic stirrer, were added dry nitrogen-purged THF (80 mL), malononitrile (0.66 g, 15 mmol) and sodium hydride (0.90 g, 60 %, 22.5 mmol), the resulting mixture being kept at r.t. After the reaction subsided, 4,4″-diiodo-*p*-terphenyl (1.81 g, 3.75 mmol) and finely pulverized bis(triphenylphosphine)palladium(II) chloride (0.104 g, 148 μmol) were added and the reaction mixture was refluxed for 44 h. A tan coloured solid suspension resulted and, after cooling, the solvent was removed under vacuum. To the residue was added crushed ice (150 g) which eventually melted and yielded a light-yellow suspension. The latter after filtration and acidification to pH 4 with 1 N HCl, yielded a cream coloured flaky precipitate which gradually turned into a pale-green fine solid. After adding water (100 mL) to the mixture, the product was filtered off under vacuum and dried over $MgSO_4$, to give practically pure **4** (1.1 g).

Method B:

Using the same quantities of reagents and the above catalyst, the reaction was carried in DMSO under conditions described in Table 1. The reaction mixture soon became a persistent red-brown colour and after completion of the reaction, the mixture was poured over crushed ice (200 g). When most of the ice had melted, the mixture was acidified with 1 N HCl to pH 4. The resulting precipitate was then treated as described in method A.

4-Dicyanomethyl-4′-/4″-nitro-*p*-polyphenyls 6, 8; General Procedure:

Into a three-necked round-bottom flask fitted with a thermometer, bleeding tube and magnetic stirrer were introduced dry DMSO (60 mL) and malononitrile (1.65 g, 25 mmol). Sodium hydride (1.18 g, 50 mmol) was then added while stirring and cooling to r.t. After the reaction subsided, the 4-iodo-4′/-4″-nitro-*p*-polyphenyl (10 mmol) and finely pulverized Pd catalyst in the molar ratios shown in Table 1 were each added in a single portion. A deep-red wine colour gradually developed as the temperature was increased.

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The reaction mixture was then heated according to the conditions prescribed for each of the reactants **5** and **7**. The mixture was subsequently cooled, poured over crushed ice (200 g) and acidified to pH 4 with 1 N HCl after most of the ice had melted. A semi-solid precipitate formed which was separated by vacuum filtration and the product was left to dry and solidify. The crude product was then purified by treatment on a chromatographic column as follows: a 370 mm column was packed with silica gel (60 F, 70–230 mesh) to a height of 340 mm using benzene, and the crude product was dissolved in a small amount of acetonitrile and then introduced at the top of the column. Pure **6** was obtained by first eluting with benzene and then with progressively more polar mixtures of EtOAc–benzene until a final volume ratio of 3:1 was attained. Product **8** was purified by first eluting with a MeOH–benzene (1:3 vol.) solution and then gradually shifting to more polar mixtures until finally pure methanol was employed. In both cases, the second band yielded the desired compound. After solvent evaporation, a pale-green solid of analytical quality was isolated.

Tetrabutylammonium Salts 10–12; General Procedure:
A hot solution of **4**, **6** or **8** (4 mmol) in MeOH (48 mL) was diluted with hot water (5 mL) and then treated with a warm solution of 1 M TBA hydroxide solution (4.4 mL, 4.4 mmol) diluted with water (5 mL). To the resulting deep-red solution, distilled water was added dropwise until precipitation of a dark-red product was complete. The mixture was subsequently heated for a few min with stirring, cooled in an ice bath, filtered and the residue dried. The respective salt was recrystallized from an appropriate solvent until a constant mp was attained.

4′-(*N,N*-Dimethylanilino)pyridinium 4′/4″-Nitro-*p*-polyphenylmalononitrile 13, 15; General Procedure:
4′-(*N,N*-Dimethylanilino)pyridine (0.20 g, 1 mmol) was suspended in a solution of MeOH (10 mL) and water (10 mL), and then treated with 0.25 M aq HCl (1.2 mmol). A yellow solution resulted which was then evaporated to dryness under reduced pressure to afford the hydrochloride salt as a yellow-orange solid residue. The residue was dissolved in water (50 mL) and treated with a solution of TBA salt (1 mmol) dissolved in MeOH (20 mL) and water (8 mL). A red-brown product immediately precipitated. The mixture was then heated to 50 °C while stirring to ascertain that the metathesis was complete. Upon cooling, the mixture was filtered and the product was rinsed with cold water, dried and recrystallized several times to a constant mp.

Methyl 4′-(*N,N*-Dimethylanilino)pyridinium-4′/4″-nitro-*p*-polyphenylmalononitrile 14, 16; General Procedure:
Methyl 4′-(*N,N*-dimethylanilino)pyridinium iodide hydrate[18] (0.25 g, 70 mmol) was dissolved in MeOH (8 mL) and then diluted with water (8 mL). The latter was mixed with a solution of TBA salt (77 mmol) in MeOH (20 mL). The dark-red solution rapidly yielded a brown crystalline precipitate. The mixture was then heated to 70 °C for a few minutes, cooled in ice and filtered. The dried product after three recrystallizations yielded a salt sufficiently pure for analytical purposes.

4,4′-Bipiperidinium(2^+) Bis(4″-nitro-*p*-terphenylmalononitrile) · Dimethyl Sulfoxide (17):
A solution of 4,4′-bipiperidine dihydrochloride (97%, 0.055 g, 0.22 mmol) in water (10 mL) was added to a solution of **10** (0.25 g, 0.43 mmol) dissolved in hot MeOH (10 mL) and then diluted with water (5 mL). Upon mixing of the two solutions, a brown precipitate immediately appeared. The reaction mixture was then heated for a few minutes during which more water (10 mL) was added dropwise to assure complete precipitation. The mixture was left to cool in the refrigerator for several hours and then filtered under vacuum and the residue dried. Recrystallized thrice from a DMSO/MeCN (5:3 vol.) solution, the title salt was obtained as a brick red microcrystalline material.

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