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PHOTOREACTIONS OF AROMATIC COMPOUNDS—XXVI[1]

PHOTOINDUCED REACTIONS OF BIPHENYL AND BIPHENYL DERIVATIVES WITH CYANIDE ION†

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Abstract. The photoreactions of a number of biphenyl derivatives, in particular nitrobiphenyl derivatives, with cyanide ion have been investigated. Photosubstitution by cyanide ion has been found to predominate; photoreduction is not observed. In some cases (4-nitrobiphenyl, 4,4'-dinitrobiphenyl, 3-nitrobiphenyl and similar compounds) the behaviour can be rationalized on the basis of analogy to the pattern of photocyanation of biphenyl itself (preference for substitution of the 4-position). 2-Methoxy-4-nitrobiphenyl and 2-methoxy-5-nitrobiphenyl on irradiation reacted with cyanide ion following the pattern of the nitroanisoles rather than that of the nitrobiphenyls not containing a methoxy substituent. In photoexcited 2,4,6-trimethylbiphenyls the favoured position for attack by cyanide ion appears to be at the 3-position, i.e. in the methyl substituted ring.

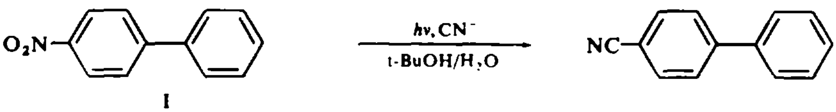
INTRODUCTION

RECENTLY, it has been reported[2,3] that nitrobenzene and 1-nitronaphthalene show different behaviour towards cyanide ion on irradiation in aqueous solution. In photoexcited nitrobenzene the nitro groups is reduced by cyanide ion, which leads to the formation of nitrosobenzene, whereas 1-nitronaphthalene undergoes photosubstitution, giving rise to the formation of 1-cyanonaphthalene. It has been suggested[3,4] that the photoreduction of nitrobenzene originates from a low-lying n, π^* excited state, whereas photosubstitution of 1-nitronaphthalene is believed to proceed through a low lying π, π^* excited state.

In view of these results, it was deemed interesting to investigate the possible photoreactions of nitrobiphenyl derivatives with cyanide ion.

RESULTS AND DISCUSSION

Irradiation of 4-nitrobiphenyl (I) with KCN in t-BuOH aq afforded 4-cyanobiphenyl as main product (44·6%‡) in addition to several by-products, each in low yield.



I

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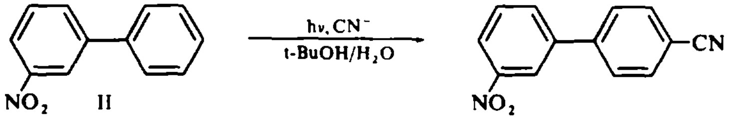
† The investigations will be described in greater detail in J. A. J. Vink, thesis, Leiden, to be published, 1972.

‡ All percentage yields are based on the amount of consumed starting material.

This reaction is analogous to that of 1-nitronaphthalene[3,5] and 1-nitroazulene.[6]

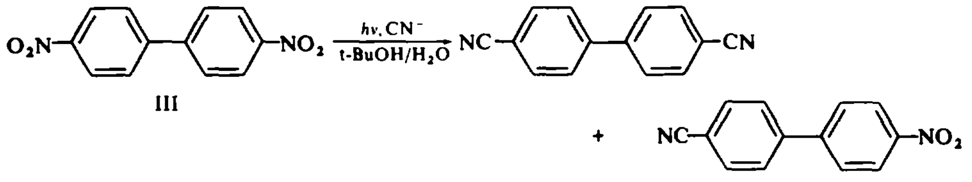
3-Nitrobiphenyl (II) is also substituted by cyanide ion on irradiation. In this case it is not the nitro group that is displaced, but the hydrogen atom at the 4′-position leading to formation of 4-cyano-3′-nitrobiphenyl in high yield (77·3%).



Neither of these two irradiations results in the formation of detectable amounts of nitrosobiphenyl.

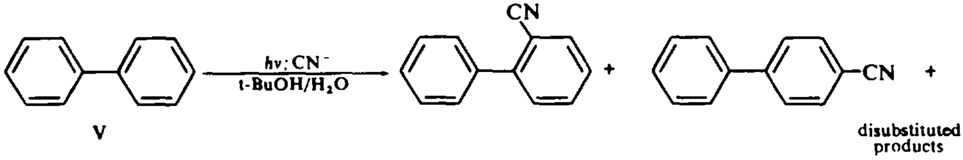
From the fact that both compounds undergo photosubstitution rather than photoreduction, it may tentatively be concluded that their reactive excited state has the same character as that of 1-nitronaphthalene.

Irradiation of 4,4′-dinitrobiphenyl (III) in t-BuOH aq with cyanide ion yielded two substitution products, viz. 4-cyano-4′-nitrobiphenyl (10·4%) and 4,4′-dicyanobiphenyl (61·4%).



However, from the irradiation of 3,3′-dinitrobiphenyl (IV) under similar conditions no cyano-substituted compounds could be isolated.

To examine whether the presence of a nitro group is required for the occurrence of photocyanation we illuminated biphenyl (V) in the same medium. The products of the reaction were 2-cyanobiphenyl, 4-cyanobiphenyl, and at least two unidentified dicyano-substituted biphenyls.

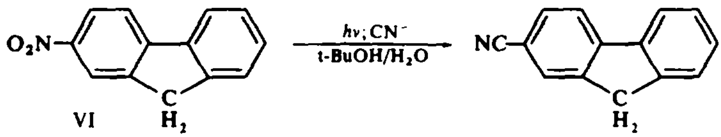


The ratio of the yield of 2-cyanobiphenyl to that of 4-cyanophenyl depends on the time of irradiation. After 10 hrs the yields of 2-cyanobiphenyl, 4-cyanobiphenyl and of the dicyanobiphenyls were respectively 13·8%, 28·6% and 10·6%. After 21 hrs the yields were respectively 14·3%, 17·5% and 22·6%.

It can be seen that the yield of 2-cyanobiphenyl is constant within experimental error. From the observation that the sum of the yields of 4-cyanobiphenyl and the dicyano-substituted biphenyls is also constant, we concluded that the dicyanobiphenyls are formed from 4-cyanobiphenyl, in which coplanarity of the phenyl rings is not hindered. This would mean that in the primary photochemical reaction the ratio of the formation of 2-cyanobiphenyl to 4-cyanobiphenyl is about 1:3. Allowing for the statistical factor the ratio of the rates of substitution at the *ortho* versus the *para* position is 1:6.

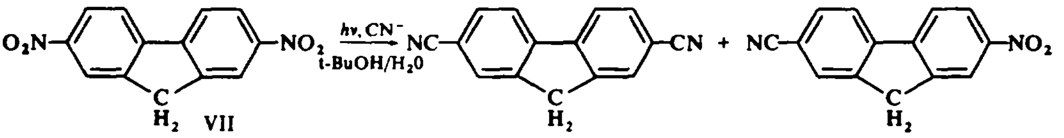
It seems consistent with the observed reactivity of the *para* position in biphenyl that in 4-nitrobiphenyl and 4,4'-dinitrobiphenyl the nitro groups are displaced. The much shorter times of irradiation needed in the case of the nitro-substituted compounds (Table 1) may be partly explained by the activating influence of the electron withdrawing nitro group and its greater leaving group tendency relative to hydrogen. The fact that in 3,3'-dinitrobiphenyl no substitution at the 4- and 4'-position could be observed, while in 3-nitrobiphenyl the *para* position in the unsubstituted ring is preferred, might for the moment be described in terms of hindrance by an *ortho* nitro group.

2-Nitrofluorene (VI) and 2,7-dinitrofluorene (VII) on irradiation in the presence of cyanide show reactions that are completely analogous to that of 4-nitrobiphenyl and 4,4'-dinitrobiphenyl, respectively, though product yields are lower and more by-products are formed. 2-Nitrofluorene (VI) afforded 2-cyanofluorene (24·9%) as main product.



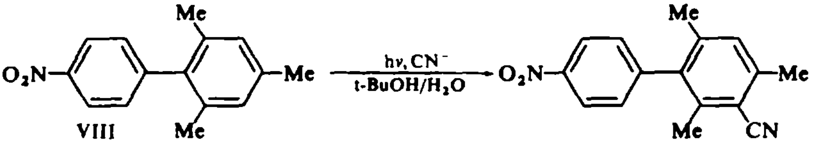
VI

From 2,7-dinitrofluorene (VII) two photosubstitution products were isolated, 2-cyano-7-nitrofluorene (2·3%) and 2,7-dicyanofluorene (24·8%).



VII

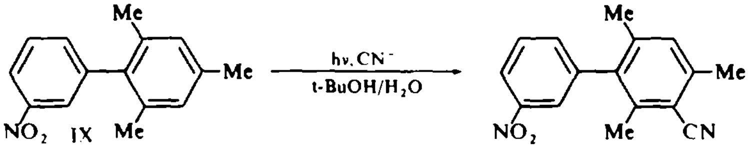
There is general consent that biphenyls in their excited states tend to assume a planar conformation.[7,8,9] Since *ortho* substituents are known to hinder the decrease of the dihedral angle, it was considered worth-while investigating the effect of methyl groups at *ortho* positions on the photoreaction of 4-nitrobiphenyl with cyanide ion. The reaction was performed with 2,4,6-trimethyl-4'-nitrobiphenyl (VIII), this compound being more easily accessible than the 2,6-dimethyl derivative.



VIII

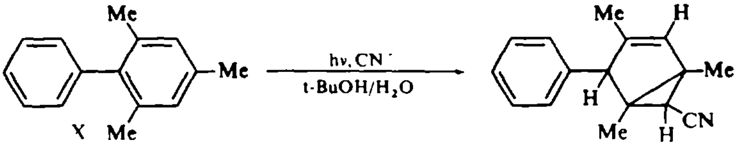
The product of the reaction, 3-cyano-2,4,6-trimethyl-4′-nitrobiphenyl, was formed in very high yield (80·6%). No trace was found of the compound in which substitution of the nitro group by cyanide had taken place, nor could formation of the nitroso compound be detected.

Evidently, introduction of the three methyl groups has a strong influence on the reaction course, but not to the effect that the compound now behaves like a substituted nitrobenzene. That, unlike in many substituted nitrobenzenes, no substitution takes place at the positions *meta* to the nitro group, may be caused by the fact that attack at these positions is hindered by the methyl groups. The non-occurrence of photoreduction of the nitro group is consistent with the idea that despite the presence of the *ortho* methyl substituents the molecule is still nearly planar in the excited state, which may thus be expected to have the same character as that of the unsubstituted nitrobiphenyls. Compared to 4-nitrobiphenyl the presence of the methyl groups causes a change in the reaction pattern. It seems plausible that the electronic properties of the methyl groups co-determine the reaction path. This view is corroborated by the behaviour of 2,4,6-trimethyl-3′-nitrobiphenyl (IX) in which substitution by cyanide was found to occur also in the methyl-substituted ring, yielding 3-cyano-2, 4, 6-trimethyl-3′-nitrobiphenyl (56·8%).


IX

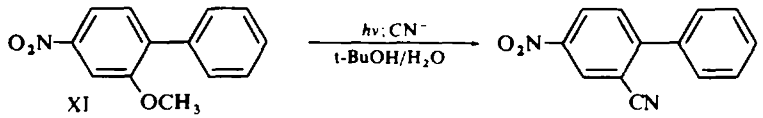
In contrast with 2,4,6-trimethyl-4′-nitrobiphenyl, compound (IX) has an unhindered *meta* position with respect to the nitro group. Yet, substitution takes place in the ring bearing the methyl groups.

In view of these results, it seemed attractive to try to effect photochemical substitution by cyanide ion in 2,4,6-trimethylbiphenyl (X). However, even upon prolonged irradiation under the same reaction conditions no cyano-2,4,6-trimethylbiphenyl could be found. Instead a product, probably the *endo-endo* isomer of 6-cyano-4-phenyl-1,3,5-trimethylbicyclo[3.1.0]hex-2-ene (~20%), formed in a process of which the overall result is photochemical addition of hydrogen cyanide, was isolated. It appears that the presence, but not the position of a nitro group in the methyl-substituted biphenyls, is essential for the occurrence of a smooth photosubstitution in the mesitylene moiety by cyanide.


X

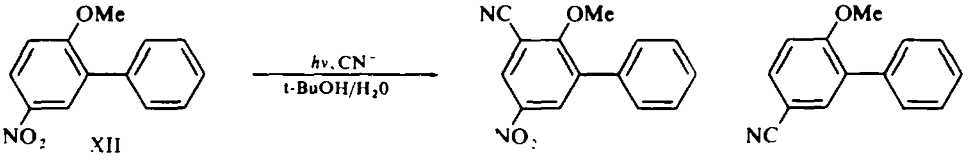
We rounded off our study of the biphenyls by investigating the reactions of 2-methoxy-4-nitrobiphenyl and 2-methoxy-5-nitrobiphenyl with cyanide ion, in order to investigate whether these compounds behave like nitroanisoles[10, 11] or like nitrobiphenyls.

2-Methoxy-4-nitrobiphenyl (XI) on irradiation in the presence of cyanide ion in t-BuOH aq afforded 2-cyano-4-nitrobiphenyl (68·5 %).



XI

The photoreaction of 2-methoxy-5-nitrobiphenyl (XII) under the same conditions proceeds not as smoothly as that of compound XI. Two cyano-substituted products could be identified, *viz.* 2-methoxy-5-cyanobiphenyl (9 %) and 2-methoxy-3-cyano-5-nitrobiphenyl (9 %).



XII

It is obvious that these two compounds behave like nitroanisoles and not like other nitrophenyls.

Summarizing it can be stated that biphenyl and nitrobiphenyls show an intriguing variety of substitution patterns in photoreactions with cyanide ion. In many cases there is analogy with reactions in the naphthalene series, but not in all. Reduction to nitroso compounds occurring in some benzene derivatives and probably indicative of a low-lying n, π^* state, is not found with nitrobiphenyls.

EXPERIMENTAL

M.ps are uncorrected. IR spectra were taken on a Beckman IR-10 spectrometer. NMR spectra were obtained using a Jeol JNM-PS-100 spectrometer. UV spectra were taken with a Cary 15 spectrophotometer. Mass spectra were obtained using a GEC AEI-MS 902 mass spectrometer. VPC analyses were made on a Hewlett-Packard HP-402 gas chromatograph with a SE-30 1 % column with a length of 1.20 m and on a Varian Aerograph Model 90-P with a OV-17 20 % column with a length of 6 m. The starting materials and the reagents were commercial products with the exception of the following compounds:

2,4,6-*Trimethyl-4'-nitrobiphenyl* (VIII) was prepared by the Gomberg–Hey synthesis[12] from 4-nitroaniline and mesitylene, m.p. 94°. Mass spectrum: $M^+ = 241{\cdot}1124$ ($C_{15}H_{15}NO_2$: 241·1103). IR (KBr): 1510, 1340 cm^{-1} (NO_2): 1464, 1378 cm^{-1} (CH_3). UV (EtOH): $\lambda_{max} = 266$ nm. NMR ($CDCl_3$): $\delta = 8{\cdot}18$ ppm (d, 2H, H 3',5'), 7·21 (d, 2H, H 2', 6'), 6·88 (s, 2H, H 3, 5), 2·34 (s, 3H, CH_3), 2·00 (s, 6H, 2 CH_3).

2,4,6-*Trimethyl-3'-nitrobiphenyl* (IX) was prepared by the Gomberg–Hey synthesis[12] from 3-nitroaniline and mesitylene, m.p. 83–84°. Mass spectrum: *m/e* (parent peak) = 241. IR (KBr): 1520, 1345 cm^{-1} (NO_2); 1460, 1378 cm^{-1} (CH_3). UV (EtOH): $\lambda_{max} = 249$ nm. NMR ($CDCl_3$): $\delta = 8{\cdot}13$ ppm (d, 1H, H 4'), 7·98 (s, 1H, H 2'), 7·55 (t, 1H, H 5'), 7·44 (d, 1H, H 6'), 6·93 (s, 2H, H 3, 5), 2·36 (s, 3H, CH_3), 2·03 (s, 6H, 2 CH_3).

2-*Nitrofluorene* (VI) was prepared by nitration of fluorene.[13] M.p. 157–159° (Lit.,[13] 156°).

2,7-*Dinitrofluorene* (VII) was prepared by nitration of fluorene.[14]

2,4,6-*Trimethylbiphenyl* (X) was prepared by the Gomberg–Hey synthesis[12] from aniline and mesitylene. b.p. 101°/5 mm Hg (Lit.,[15] 275–277°).

3,3′-*Dinitrobiphenyl* (IV) was prepared by the Ullmann synthesis[16] from 3-iodonitrobenzene, m.p. 203° (lit.,[16] 200°).

2-*Methoxy-4-nitrobiphenyl* (XI) was prepared by the Gomberg–Hey synthesis[12] from 2-methoxy-4-nitroaniline and benzene, m.p. 70° (lit.,[17] 68–70°).

2-*Methoxy-5-nitrobiphenyl* (XII) was prepared by the Gomberg–Hey synthesis[12] from 2-methoxy-5-nitroaniline and benzene, m.p. 96° (lit.,[18] 95–96).

Procedure of irradiation experiments. Preliminary experiments were performed with dilute (*ca.* 10^{-4} M) solutions in t-BuOH/H_2O mixtures in spectrophotometer cells. All preparative irradiations were performed with 200 mg of starting material in 1500 ml of a mixture of t-BuOH/H_2O, except in the case of the dinitro compounds, where the volume of the solutions was 1750 ml. In all cases the concentration of KCN was 0·05 M. Further experimental data are summarised in Table 1. A Rayonet photochemical reactor type RPR-208 was used. Upon completion of the irradiation NaCl was added to the solution and the organic layer separated. The aqueous layer was extracted twice with $CHCl_3$ and the combined organic layers dried ($CaSO_4$), filtered and solvent evaporated. The residue was subjected to column chromatography on silica gel (0·05–0·2) with $CHCl_3$ or $CHCl_3$/pentane (1:1) as eluent.

6-Cyano-4-phenyl-1,3,5-trimethylbicyclo[3.1.0]hex-2-ene was isolated by semi-preparative VPC.

TABLE 1. EXPERIMENTAL DATA ON THE PREPARATIVE SCALE IRRADIATIONS

Compound no.	Solvent composition t-BuOH:H_2O	Lamps (RUL)	Irradiation time (min)	Degree of conversion (%)
I	1:2	3500 A	15	88·5
II	1:2	2537 A	10	57
III	5:2	3000 A	60	94·5
IV	2:1	2537 A	360	—
V	1:2	2537 A	600	67·5
V	1:2	2537 A	1260	99
VI	1:1	3500 A	25	100
VII	6:1	3000 A	30	95·5
VIII	1:2	2537 A	34	93
IX	1:2	2537 A	120	93·5
X (500 mg)	1:2	2537 A	420	44·2
XI	1:2	3500 A	10	100
XII	1:2	2537 A	10	62

Identification of irriation products. All products were identified by means of their IR, NMR, UV and mass spectra, and in some cases by their m.p.s. In the case of biphenyl, a mixture of 2-cyano- and 4-cyanobiphenyl was obtained. It was analysed by means of VPC by comparison with authentic samples.

4-*Cyanobiphenyl*, m.p. 87·5–88° (lit.,[19] 88°). Mass spectrum: *m/e* (parent peak) = 179. IR (KBr): 2218 cm^{-1} (CN). UV (EtOH): λ_{max} = 268 nm. NMR ($CDCl_3$): δ = 7·56 ppm (s, 4H, H 2, 3, 5, 6), 7·2–7·5 (m, 5H, H 2′, 3′, 4′, 5′, 6′).

4-*Cyano-3′-nitrobiphenyl*, m.p. 175–176°. Mass spectrum: *m/e* (parent peak) = 224. IR (KBr): 2214 cm^{-1} (CN); 1518, 1347 cm^{-1} (NO_2). UV (EtOH): λ_{max} = 259 nm. NMR ($CDCl_3$): δ = 8·40 ppm (s, 1H, H 2′), 8·24 (d, 1H H 4′), 7·89 (d, 1H, H 6′), 7·74 (s, 4H, H 2, 3, 5, 6), 7·65 (t, 1H, H 5′); $J_{4'5'} = J_{5'6'}$ = 7·5 Hz.

3-*Cyano-2,4,6-trimethyl-4′-nitrobiphenyl*, m.p. 133–134°. Mass spectrum: *m/e* (parent peak) = 266. IR (KBr): 2200 cm^{-1} (CN); 1512, 1340 cm^{-1} (NO_2). UV (EtOH): λ_{max} = 242, 270 nm. NMR ($CDCl_3$): δ = 8·10 ppm (d, 2H, H3′, 5′) 7·33 (d, 2H, H 2′, 6′), 7·13 (s, 1H, H 5), 2·53 (s, 3H, CH_3), 2·22 (s, 3H, CH_3), 2·05 (s, 3H, CH_3).

3-*Cyano-2,4,6-trimethyl-3′-nitrobiphenyl*, m.p. 134–135°. Mass spectrum: *m/e* (parent peak) = 266. IR (KBr): 2198 cm^{-1} (CN); 1522, 1342 cm^{-1} (NO_2). UV (EtOH): λ_{max} 207, 240 (sh)nm. NMR ($CDCl_3$): δ = 8·25 ppm (d, 1H, H4′), 7·89 (s, 1H, H 2′), 7·61 (t, 1H, H 5′), 7·40 (d, 1H, H 6′), 7·05 (s, 1H. H 5), 2·58 (s, 3H, CH_3), 2·25 (s, 3H, CH_3), 2·07 (s, 3H, CH_3); $J_{4'5'}$ = 7·5 Hz.

4,4′-*Dicyanobiphenyl*, m.p. 230° (lit.,[20] 234°). Mass spectrum: *m/e* (parent peak) = 204. IR (KBr): 2218

cm^{-1} (CN); 813 cm^{-1} (*para*-substitution). UV (EtOH): λ_{max} = 274 nm. NMR ($CDCl_3$): δ = 7·76 ppm (d, 4H, H3, 5, 3′, 5′), 7·68 (d, 4H, 2, 6, 2′, 6′).

4-*Cyano-4′-nitrobiphenyl*, m.p. 181–182°. Mass spectrum: *m/e* (parent peak) = 224. IR (KBr): 2218 cm^{-1} (CN); 1510, 1343 cm^{-1} (NO_2). UV (EtOH): λ_{max} = 295 nm. NMR ($CDCl_3$): δ = 8·27 ppm (d, 2H, H 3′, 5′), 7·83–7·59 (m, 6H, H2, 3, 5, 6, 2′, 6′).

2-*Cyanofluorene*, m.p., 89° (lit.,[21] 88°). Mass spectrum: *m/e* (parent peak) = 191. IR (KBr): 2212 cm^{-1} (CN). UV (EtOH): λ_{max} = 283, 295, 307 nm. NMR ($CDCl_3$): δ = 7·83–7·29 ppm (m, 7H, H 1, 3, 4, 5, 6, 7, 8) s = 3·87 (s, 2H, CH_2).

2,7-*Dicyanofluorene*, m.p. 264°. Mass spectrum: *m/e* (parent peak) = 216. IR (KBr): 2205 cm^{-1} (CN). UV (EtOH): λ_{max} = 288, 299, 304, 312 nm. NMR ($CDCl_3$): δ = 7·88 ppm (d, 2H, H 3, 6), 7·83 (s, 2H, H1, 8), 7·68 (d, 2H, H4, 5), 4·04 (s, 2H, CH_2).

2-*Cyano-7-nitrofluorene*, m.p. 277–278° (lit.,[22] 281–282°). Mass spectrum: *m/e* (parent peak) = 236. IR (KBr): 2210 cm^{-1} (CN); 1516, 1346 cm^{-1} (NO_2). UV (EtOH): λ_{max} = 321 nm.

2-*Cyano-4-nitrobiphenyl*, m.p. 146°. Mass spectrum: *m/e* (parent peak) = 224. IR (KBr): 2225 cm^{-1} (CN); 1522, 1351 cm^{-1} (NO_2). UV (EtOH): λ_{max} = 297 nm. NMR ($CDCl_3$): δ = 8·57 ppm (s, 1H, H 3), 8·42 (d, 1H, H5), 7·68 (d, 1H, H 6), 7·62–7·42 (m, 5H, H 2′, 3′, 4′, 5′, 6′).

2-*Methoxy-5-cyanobiphenyl*. Mass spectrum: *m/e* (parent peak) = 209. IR (KBr): 2218 cm^{-1} (CN); 1262 cm^{-1} (OCH_3). UV (EtOH): λ_{max} = 235 nm. NMR ($CDCl_3$): δ = 7·65–7·35 ppm (m, 7H, H4, 6, 2′, 3′, 4′, 5′ 6′), 7·05 (d, 1H, H3), 3·90 (s, 3H, OCH_3).

2-*Methoxy-3-cyano-5-nitrobiphenyl*. Mass spectrum: *m/e* (parent peak) = 254. IR (KBr): 2235 cm^{-1} (CN); 1527, 1347 cm^{-1} (NO_2); 1258 cm^{-1} (OCH_3). UV (EtOH): λ_{max} = 266 nm. NMR ($CDCl_3$): δ = 8·43 and 8·37 ppm (two s, each 1H, H4 and H 6), 7·62–7·41 (m, 5 H, H 2′, 3′, 4′, 5′, 6′), 3·84 (s, 3H, OCH_3).

6-*Cyano-4-phenyl*-1,3,5-*trimethylbicyclo*[3.1.0]*hex-2-ene*. Mass spectrum: M^+ = 223·1361 ($C_{16}H_{17}N$: 223·1361). IR (KBr): 2228 cm^{-1} (CN). UV (EtOH): λ_{max} = 218 nm. NMR (CCl_4): δ = 7·35–6·93 ppm (m, 5H, Ph). 4·95 (q, 1H, H2), 2·10 (d, 1H). 1·89 (d, 3H, CH_3). 1·68 (d, 1H), 1·53 (s, 3H, CH_3), 1·14 (s, 3H, CH_3). J_{46} = 3·0 Hz, H_{23} = 1·5 Hz.

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