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BIPHENYLENES—XXX[1]

SYNTHESIS AND SOME REACTIONS OF 5,10-DIAZABENZO[*b*]BIPHENYLENES

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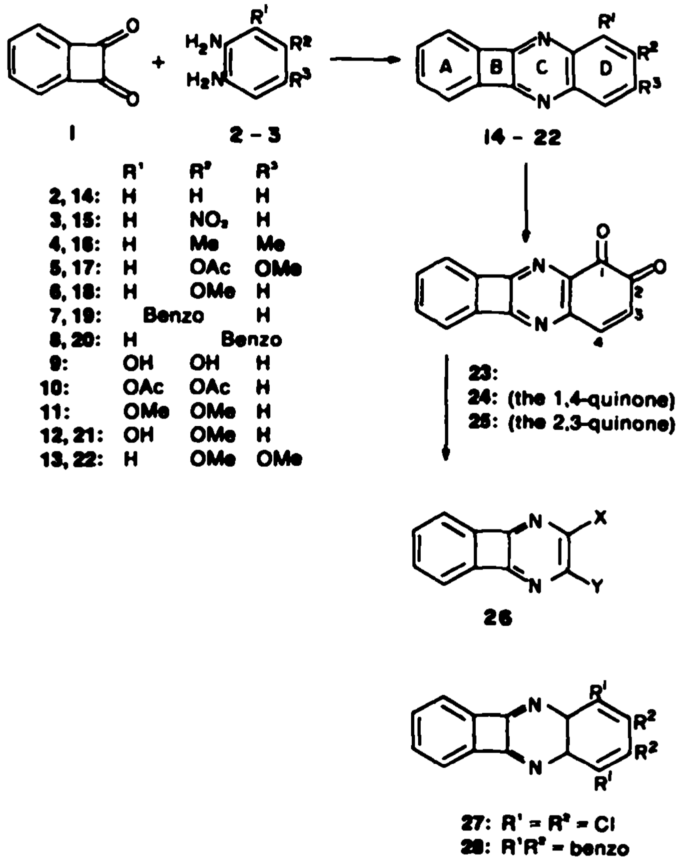
Abstract—Condensation of benzocyclobutene-1,2-dione with 10 derivatives of *o*-phenylenediamine gave the corresponding benzo-substituted derivatives of 5,10-diazabenzo[*b*]biphenylene. Attempted oxidative demethylation of 2,3-dimethoxy-5,10-diaza benzo[*b*]biphenylene gave 2-methoxy-5-phthalimido benzo-1,4-quinone. Other attempts to demethylate methoxy diazabenzobiphenylenes are described. 5,10-Diazabenzo[*b*]biphenylene was not oxidised by air, lead tetra-acetate, or phenyliodoso diacetate, but with peracetic acid it gave dibenzodiazocine-5,12-dione and *o*-nitroaniline. Reduction of the diazabiphenylene with Raney nickel gave 2-phenylquinoxaline. The diazabiphenylene is readily hydrolysed by nitric and hydrochloric acid to regenerate benzocyclobutene-1,2-dione and *o*-phenylenediamine which then recondense to give a mixture of products. A similar hydrolysis occurs with sodium hydroxide.

The first nitrogen analogue of biphenylene, 5,10-diazabenzo[*b*]biphenylene **14**, was made in 1957 by the condensation of benzocyclobutene-1,2-dione (BBD) **1** with *o*-phenylenediamine **2**.[2] More recently 1,4,5,8-tetra-azadibenzo[*b,e*]biphenylene was made in three stages from squaric acid and *o*-phenylenediamine,[3] and the following azabiphenylenes have been prepared by the pyrolysis or photolysis of appropriate precursors: 2,6-diaza-,[4] hexafluoro-2,6- and -2,7-diaza-,[5] 2,7-diaza-,[6] 1- and 2-aza, also 1,8-diaza-.[7] The present paper records the synthesis of ten derivatives of 5,10-diazabenzo[*b*]biphenylene and attempts to degrade some of them to give 2,3-disubstituted 1,4-diazabiphenylenes **26** which might be convertible into the unsubstituted compound **26** ($X = Y = H$) (see Scheme 1). Since the cleavage of a benzene ring usually requires vigorous conditions whereas the cleavage of *ortho*-quinones can be effected under mild conditions (e.g. oxidatively or by Beckmann fragmentation of quinone monoximes) we studied mainly the preparation of methoxy- and hydroxy-derivatives of 5,10-diazabenzo[*b*]biphenylene in the hope of preparing the quinones **23–25** from them. Although the desired opening of ring D was not achieved we have more recently prepared a 2,3-disubstituted 1,4-diazabiphenylene by a modification of Scheme 1.[8] After completion of the work described here the synthesis of 1,4-diazabiphenylene by pyrolysis of 1,4,9,10-tetra-azaphenanthrene was reported.[9]

Preparation of the diazabiphenylenes. The relatively stable *o*-phenylenediamines **2–8**, also tetrachloro-*o*-phenylenediamine and 9,10-diaminophenanthrene reacted with BBD **1** in ethanol containing a few drops of acetic acid to give the corresponding 5,10-diazabenzo[*b*]biphenylenes **14–20**, **27** and **28** in 68–92% yield. The air-sensitive diamines **9–13** were made by catalytic reduction of the corresponding *o*-dinitro compounds and, without isolation, allowed to react with BBD under nitrogen. This method was unsuccessful with **9–11** but the 3-hydroxy-4-methoxy- and 3,4-dimethoxy-diamines, **12** and **13**, gave the corresponding diazabenzobiphenylenes **21** and **22** in 47 and 72% yields respectively. The structures of the products follow from their method of preparation and are in agreement with their UV spectra, details of which are given in the Experimental section.

Demethylation, oxidation and reduction experiments. Attempts to demethylate the 1-hydroxy-2-methoxy compound **21** using hydrogen bromide in acetic acid or pyridine hydrochloride gave intractable solids instead of the desired 1,2-dihydroxy compound which might have been oxidisable to the *o*-quinone **23**. No reaction occurred when the compound **21** was treated with Fremy salt, possibly because of the very low solubility of the phenol in the aqueous reaction medium. Attempted demethylation of the 2-methoxy compound **18** as above gave oily products while reaction with boron tribromide caused cleavage of the C ring to regenerate the dione **1** in 91% yield.

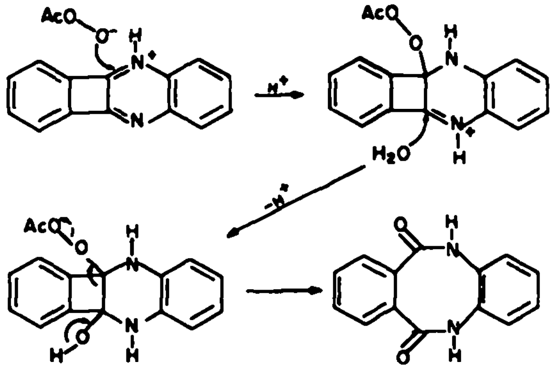
Treatment of the 2,3-dimethoxy compound **22** with bromine gave a deep red bromine-containing complex which did not react further to give the 2,3-quinone **25** but which regenerated the dimethoxy compound **22** when treated with water or ethanol. 2,3-Dimethoxybiphenylene gives a similar red complex with bromine but the red complex gradually reacts further to give the very stable biphenylene-2,3-quinone.[10] Quinone **25** may be regarded as a derivative of naphthalene-2,3-quinone, which is expected to be highly reactive and no derivatives of it have yet been isolated. For this reason it is not surprising that attempted oxidative demethylation[11] of compound **22**, with nitric acid in ethanol, did not give the 2,3-quinone **25**; instead an extensive oxidation and rearrangement occurred to give 2-methoxy-5-phthalimido-1,4-benzoquinone. The structure assigned to the benzoquinone on spectroscopic evidence was confirmed by its synthesis from 5-amino-1,2,4-trimethoxybenzene. This was converted into the corresponding 5-phthalimido compound and thence, by oxidative demethylation with nitric acid, into the required quinone. Two other routes were tried but were unsuccessful: 1,2-dimethoxy-4-phthalimidobenzene was nitrated by nitric acid instead of undergoing oxidative demethylation and insertion of an oxygen atom to give the required quinone (compare Ref. 11), and 2-methoxy-5-phthal-



	R'	R²	R³
2, 14:	H	H	H
3, 15:	H	NO_2	H
4, 16:	H	Me	Me
5, 17:	H	OAc	OMe
6, 18:	H	OMe	H
7, 19:	Benzo		H
8, 20:	H	Benzo	
9:	OH	OH	H
10:	OAc	OAc	H
11:	OMe	OMe	H
12, 21:	OH	OMe	H
13, 22:	H	OMe	OMe

Scheme 1.

imidophenol did not react with potassium nitrosodisulphonate (Fremy salt) to give the quinone. Several mechanisms can be devised for the oxidative conversion of compound 22 into the phthalimidoquinone but there is insufficient evidence to decide between them. The stability of the parent compound 5,10-diazabenzo[*b*]biphenylene 14 towards oxidising agents was studied. With hydrogen peroxide in acetic acid an oxidative cleavage of the bond common to rings B and C occurred to give the dibenzo-diazocine 29 together with a small amount of *o*-nitroaniline. A possible mechanism for this reaction is shown in Scheme 2, although the addition of water may precede attack by peracetic acid or its anion. The *o*-nitroaniline probably arises by oxidation of *o*-phenylenediamine formed by hydrolysis of some of the diazabiphenylene. Compound 14 was not oxidised when air was bubbled through a solution of it in 1,2-dichloroethane for 12 hr, nor was it oxidised by lead tetra-acetate or phenyliodoso diacetate in hot dichloromethane.



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Scheme 2.

Reduction of compound 14 by heating it with Raney nickel in ethanol gave 2-phenylquinoxaline by fission of ring B. Similar fission of the 4-membered ring occurs with biphenylene itself and with substituted biphenylenes.[12]

Hydrolysis of 5,10-*diazabenzo*[b]*biphenylene* 14. After completion of the work described above it was discovered that the diazabiphenylene 14 is readily hydrolysed and this explains the failure of some of our attempts to demethylate the methoxy derivatives. Treatment of compound 14 with nitric acid in ethanol (under the conditions used with the dimethoxy compound 22) gave a complex mixture of white products. With cold 2 M HCl a similar mixture of products was obtained and the crude mixture was almost identical (TLC, IR, NMR, mass spec) with that obtained by mixing equimolar amounts of BBD 1 and *o*-phenylenediamine in 2 M HCl. These mixtures were not further investigated. However, more direct proof that hydrolysis to the dione and diamine had occurred was obtained by treating the diazabiphenylene 14 with 2,4-dinitrophenylhydrazine in hydrochloric acid when a mixture of the E- and Z-isomers of BBD mono-2,4-dinitrophenylhydrazone was formed. The structure of these and related hydrazones will be discussed in a separate paper.[13]

The rate of hydrolysis of the diazabiphenylene 14 was followed spectrophotometrically by following the decrease in intensity of the peak at λ_{max} 372 nm in aqueous buffers at 25°. At pH 3 and pH 4 $t_{1/2}$ was 5 min and 3.25 h respectively. Evidently the hydrolysis does not follow exact second order kinetics.

Surprisingly, the diazabiphenylene 14 is hydrolysed by dilute sodium hydroxide and gives a mixture of at least five products (TLC). The hydrolysis by alkali is much slower than by acid but its rate could not be measured accurately because of the formation of a precipitate, however the *approximate* $t_{1/2}$ at 25° in 0.5 M sodium hydroxide was 5 h. The regenerated BBD 1 is relatively stable to acid but is rapidly cleaved by sodium hydroxide to give the sodium salt of phthalaldehydic acid.[2] The latter acid is known to condense with *o*-phenylenediamine to give a variety of products depending on the conditions.[14]

EXPERIMENTAL

UV spectra were recorded as solutions in 95% EtOH and IR spectra as Nujol mulls. Petroleum refers to light petroleum (b.p. 60–80°).

5,10-*Diazabenzo*[b]*biphenylene* 14. UV λ_{max} 233, 239, 271, 293, 356, 372 and 392 nm (log ε 4.46, 4.53, 4.67, 4.26, 4.14, 4.31 and 4.29).

General method for preparation of 5,10-diazabenzo[b]*biphenylenes.* Solns of approximately equimolar amounts of BBD 1 and the appropriate aromatic 1,2-diamine in the minimum volume of EtOH were mixed and a few drops of AcOH were added. After a few hours the crystalline product was collected, washed with petroleum, then dried in air and recrystallised. The following compounds were made in this way (recrystallisation solvent and yield): 2-*nitro*-15 (toluene, 74%), yellow plates, m.p. 268–269°. (Found: C, 66.8; H, 2.9; N, 16.5. $C_{14}H_7N_3O_2$ requires: C, 67.5; H, 2.8; N, 16.9%); UV λ_{max} 233, 271, 274 sh, 310, 364, 378 and 396 nm (log ε 4.29, 4.51, 4.50, 4.13, 3.91, 4.13 and 4.10); 2,3-*dimethyl*- 16 (acetone, 92%) needles, m.p. 262–263°. (Found: C, 82.2; H, 5.5; N, 12.1. $C_{16}H_{12}N_2$ requires: C, 82.7; H. 5.2; N, 12.1%), UV λ_{max} 226, 239, 247, 271sh, 279, 296, 368sh, 384 and 405 nm (log ε 3.89, 4.05, 4.00, 3.91, 4.02, 3.64, 3.42, 3.73 and 3.76); 2-*acetoxy*-3-*methoxy*- 17 (benzene, 87%), yellow prisms, m.p. 216–217°. (Found: 69.3; H, 4.2; N, 9.5. $C_{17}H_{12}N_2O_3$ requires: C, 69.9; H, 4.1; N, 9.6%; 2-*methoxy*- 18 (benzene-petroleum, 75%), yellow needles, m.p. 140–142°. (Found: C, 77.3; H, 4.3; N, 11.9. $C_{15}H_{10}N_2O$ requires: C, 76.9; H, 4.3; N, 12.0%), UV λ_{max} 231, 244, 249, 277, 296sh, 392 and 409 nm (log ε 4.20, 4.26, 4.29, 4.37, 3.97, 4.03 and 4.03); 1,2,3,4-*tetrachloro*- 27 (benzene, 85%), needles, m.p. 303–304°. (Found: C, 49.0; H, 1.3; N, 8.0. $C_{14}H_4Cl_4N_2$ requires: C, 49.2; H, 1.2; N,.8.2%), UV λ_{max} 218, 275, 287, 309, 346, 365, 382 and 401 nm (log ε 4.07, 4.29, 4.34, 4.00, 3.51, 3.63, 3.94 and 3.97); *naphtho*[1,2-b]-1,4-*diazabiphenylene* 19 (benzene–petroleum, 76%), yellow needles, m.p. 208–209°. (Found: C, 85.1; H, 4.1; N, 11.1. $C_{18}H_{10}N_2$ requires: C, 85.0; H. 4.0; N, 11.0%), UV λ_{max} 226, 254, 270sh, 280, 321sh, 328, 381sh, 399 and 421 nm (log ε 4.26, 4.34, 4.24, 4.27, 3.95, 4.08, 3.77, 4.07 and 4.16); *naphtho*[2,3-b]-1,4-*diazabiphenylene* 20 (sublimed, 72%), yellow plates, m.p. 312–314°. (Found: C, 85.7; H, 4.2; N, 10.9%), UV λ_{max} 257, 267, 283, 294, 317sh, 325, 378, 396 and 417 nm (log ε 4.51, 4.48, 4.51, 4.62, 4.58, 4.65, 4.03, 4.06 and 3.80); *phenanthro*[9,10-b]-1,4-*diazabiphenylene* 28 (sublimed, 68%), yellow needles, m.p. 308–309°. (Found: C, 86.1; H, 4.1; N, 9.0. $C_{22}H_{12}N_2$ requires: C, 86.9; H, 4.0; N, 9.2%), UV λ_{max} 223, 247sh, 253, 276sh, 287, 298, 357, 403 and 425 nm (log ε 4.10, 4.55, 4.58, 4.17, 4.14, 4.00, 3.56, 3.99 and 4.08).

1 - *Hydroxy* - 2 - *methoxy* - 5,10 - *diazabenzo*[b]*biphenylene* 21. A soln of 5,6-dinitroguaiacol[15] (642 mg) in EtOH (60 ml) was hydrogenated at 3 atm in the presence of PtO_2 (100 mg) during 3 hr. The resulting soln was filtered under N_2 into an ethanolic soln of BBD 1 (396 mg) containing a few drops of AcOH. The mixture was warmed on a water bath for 0.5 hr then kept overnight at 0°. The solid was collected and recrystallised from $CHCl_3$ to give the *diazabiphenylene* 21 (352 mg, 47%) as yellow needles, m.p. 274–276°. (Found: C, 71.4; H, 4.1; N, 11.3. $C_{15}H_{10}N_2O_2$ requires: C, 80.0; H, 4.0; N, 11.2%).

2,3-*Dimethoxy*-5,10-*diazabenzo*[b]*biphenylene* 22. This was made from 4,5-dinitroveratrole[16] as in the preceding experiment. The *diazabiphenylene* 22 (72%) formed yellow needles (from ethanol), m.p. 228–230°. (Found: C, 72.7; H, 4.6; N, 10.2. $C_{16}H_{12}N_2O_2$ requires: C, 72.7; H, 4.6; N, 10.6%), UV λ_{max} 230, 256, 259, 283, 290, 299sh, 403 and 422 nm (log ε 4.04, 4.27, 4.28, 3.93, 3.93, 3.75, 3.98 and 4.06).

1,2-*Diacetoxy*-3,4-*dinitrobenzene.* 3,4-Dinitrocatechol[17] was acetylated with Ac_2O and pyridine. The *diacetate* formed plates, m.p. 123–124°. (Found: C, 41.7; H, 2.7; N, 9.7. $C_{10}H_8N_2O_8$ requires: C, 42.3; H, 2.8; N, 9.9%).

Reaction of 2 - *methoxy* - 5,10 - *diazabenzo*[b]*biphenylene* 18 *with boron tribromide.* Boron tribromide[18] (2.6 ml) in methylene chloride (2 ml) was added to a stirred soln of 18 (200 mg) in the same solvent at −78°. The mixture was allowed to warm up to room temp. while being stirred during 20 hr. Water (10 ml) was added and the product was extracted into ether (2 × 25 ml). Removal of solvent gave BBD (103 mg, 91%).

Reaction of 2,3 - *dimethoxy* - 5,10 - *diazabenzo*[b]*biphenylene* 22 *with nitric acid*

(a) The addition of the dimethoxy compound 22 to HNO_3 (*d* 1.4) gave an orange ppt which, on washing with water, gave a quantitative recovery of starting material.

(b) The dimethoxy compound (250 mg) was added to a mixture of HNO_3 (5 ml, *d* 1.2) and EtOH (5 ml). The resulting orange soln was warmed gently until a vigorous reaction occurred, after which the resulting red soln was poured into ice-water (25 ml). The solid was collected and gave 2 - *methoxy* - 5 - *phthalimido* - 1,4 - *benzoquinone* (196 mg, 74%) as yellow needles (from benzene–petroleum), m.p. 260–262°. (Found: M^+, 283.0485; C, 63.6; H, 3.3; N, 4.9. $C_{15}H_9NO_5$ requires: *M*, 283.0481; C, 63.6; H, 3.2; N, 5.0%), ν_{max} 1780w, 1725s, 1676s, 1655s, 1600s and 1222s cm^{-1}, τ ($CDCl_3$) 2.08 (m, 4H), 3.14 (s, 1H), 3.91 (s, 1H) and 6.12 (s, OCH_3).

1,2,4-*Trimethoxy*-5-*phthalimidobenzene.* A finely-ground mixture of 5 - amino - 1,2,4 - trimethoxybenzene[19] (1.0 g), and phthalic anhydride (2.0 g) was heated in an oil-bath at 180–185°, with stirring, until effervescence ceased (*ca.* 10 min). The dark melt was cooled then stirred with warm EtOH. The gray solid was recrystallised from CH_2Cl_2/petroleum (charcoal) and gave the *phthalimido benzene* (1.4 g, 82%) as yellow crystals, m.p. 205–206°. (Found: C, 65.1; H, 4.9; N, 4.5. $C_{17}H_{15}NO_5$ requires: C, 65.2; H, 4.8; N, 4.5%), τ ($CDCl_3$) 2.03–2.29 (AA′BB′, 4H), 3.22 (s, ArH), 3.33 (s, ArH), 6.07, 6.17 and 6.23 (s, 3 × OMe).

2 - *Methoxy* - 5 - *phthalimido* - 1,4 - *benzoquinone.* Conc HNO_3 (3 ml) in AcOH (6 ml) was added to the above phthalimidobenzene (0.5 g) in acetic acid (10 ml) and the mixture was heated to 60° for 15 min. The mixture was cooled in ice and the solid collected. It was chromatographed on a silica gel M.F.C. column in $CHCl_3$ and gave the quinone (0.20 g, 44%) as yellow needles (from MeOH), m.p. 259–262° (dec). (Found: C, 63.3; H, 3.3; N, 5.0. Calc. for $C_{15}H_9NO_5$: C, 63.6; H, 3.2; N, 5.0%). It was identical (IR, NMR) with the quinone from diazabiphenylene 22.

1,2 - *Dimethoxy* - 4 - *nitro* - 5 - *phthalimidobenzene.* A mixture of 3,4-dimethoxy aniline (750 mg) and phthalic anhydride (1.5 g) was heated at 180–185° until reaction ceased (*ca.* 8 min). The cooled product was triturated with hot EtOH then collected by filtration. The 1,2 - *dimethoxy* - 4 - *phthalimidobenzene* (1.19 g, 85%) formed needles (from CH_2Cl_2/petroleum), m.p. 204–205°. (Found: C, 68.2; H, 4.5. $C_{16}H_{13}NO_4$ requires: C, 67.8; H, 4.6%).

This phthalimido compound (250 mg) was added to a mixture of HNO_3 acid (5 ml, *d* 1.2) and EtOH (2.5 ml), and the soln was warmed carefully until a vigorous reaction set in. After 1 min ice-water was added and the solid was collected. Recrystallisation from CH_2Cl_2/petroleum gave 1,2 - *dimethoxy* - 4 - *nitro* - 5 - *phthalimidobenzene* (262 mg, 90%) as yellow needles, m.p. 185–186°. (Found: C, 58.5; H, 3.7. $C_{16}H_{12}N_2O_6$ requires: C, 58.9; H, 3.7%).

2 - *Methoxy* - 5 - *phthalimidophenol.* 2 - Methoxy - 5 - nitrophenol (4 g) was dissolved in a boiling mixture of EtOH (20 ml) and 100% hydrazine hydrate (5 ml). Pd-black catalyst

(0.3 g) was added in portions to maintain gentle refluxing during 90 min. Anhyd. Na_2SO_4 was added to the cooled soln. After 2 hr the mixture was filtered and the filtrate evaporated to dryness under reduced pressure. The crude amine was heated with about twice its weight of phthalic anhydride at 180–185° for 10 min. The cooled product was triturated with ethanol and the insoluble portion consisted of 2 - *methoxy* - 5 - *phthalimidophenol* (2.6 g, 41%) which formed plates (from CH_2Cl_2/petroleum), m.p. 201–202°. (Found: C, 66.8; H, 4.1; N, 5.2. $C_{15}H_{11}NO_4$ requires: C, 66.9; H, 4.1; N, 5.2%).

An attempt to oxidise this phenol with Fremy salt was unsuccessful.

Oxidation of 5,10-*diazabenzo*[b]*biphenylene*. A soln of the diazabiphenylene (160 mg) in AcOH (2 ml) and 30% H_2O_2 (0.25 ml) was kept at 50–55° overnight. The mixture was cooled and the white crystals collected and washed with EtOH. By comparison with an authentic sample the product was shown to be 29 (63 mg, 31%), m.p. 259–262° (lit.[20] m.p. 262–265°), τ(Me_2SO), −0.06 (s, 2NH replaceable by D_2O), 2.6–3.0 (m, 8ArH). Evaporation of the filtrate from above, followed by preparative TLC on Kieselgel G (Merck) gave a small amount of *o*-nitro-aniline (*ca.* 1%).

Reduction of 5,10-*diazabenzo*[b]*biphenylene*. The diazabiphenylene (50 mg) and Raney Ni (100 mg) in hot EtOH (3 ml) were stirred for 30 min. The hot mixture was filtered, then water was added. The resulting ppt (78%) was identified as 2-phenylquinoxaline, m.p. 75–76° (lit.[21] m.p. 78°) by comparison with an authentic sample.

Hydrolysis of 5,10-*diazabenzo*[b]*biphenylene*. The diazabiphenylene (50 mg) was added to a warm soln of 2,4-dinitrophenylhydrazine (50 mg) in 5 M HCl (3 ml). A yellow ppt formed immediately. After 15 min the ppt was collected and washed successively with 2 M HCl, water, and EtOH. The *mono-2,4-dinitrophenylhydrazone of BBD* (50 mg, 65%) had m.p. 249–250°. (Found: C, 54.1; H, 2.3; N, 17.6. $C_{14}H_8N_4O_5$ requires: C, 53.9; H, 2.6; N, 18.0%). TLC showed that it consisted of E- and Z-isomers.[13]

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