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REACTIONS OF ACETYLENES WITH TRANSITION METALS

VII *. STERIC EFFECTS IN THE OLIGOMERIZATION OF DIARYLACETYLENES

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Summary

The synthesis of *ortho*-substituted diphenylacetylenes and their behaviour toward dichlorobis(benzonitrile)palladium(II) are described. The influence of steric factors on the oligomerization is discussed.

Introduction

Several studies have been carried out on the behaviour of acetylenes toward palladium chloride [2]. The importance of steric control resulting from the size of the acetylenic substituents on the course of the reaction has been noted [3].

In the course of investigations on acetylenes substituted with bulky groups [4] it was shown [5,6] that phenyl-t-butylacetylene, with one moderately bulky and one very bulky substituent, yields a complex of cyclobutadiene with $PdCl_2$ in which the two t-butyl groups are in vicinal positions. This indicates that the size of the t-butyl groups is not sufficient to prevent the formation of a bond between the carbons bearing these bulky groups.

In order to obtain further information on the importance of the size of substituents, a study of acetylenes having substituents of increasing bulk seemed appropriate. In order to produce a controlled crowding around the complexed triple bond, *ortho*-methyl-substituted diphenylacetylenes were used.

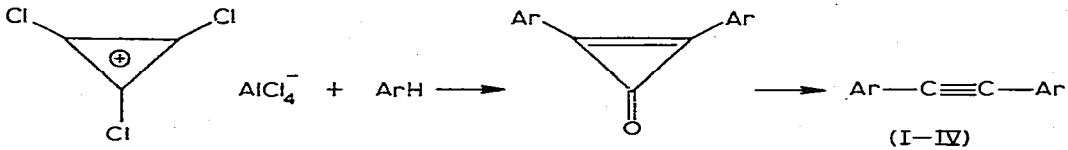
In this paper we report on the syntheses and behaviour toward dichlorobis-(benzonitrile)palladium (BNP) of the following diphenylacetylenes: (a) symmetrically substituted with the bulky t-butyl group in *para*, but without any substituent in *ortho* (di-4-t-butylphenylacetylene, I); (b) symmetrically di- and tetra-substituted in *ortho* positions (di-2,4-dimethylphenyl- and di-2,5-dimethylphenyl-acety-

* Part VI, see ref. 1.

lene, II and III, respectively, and dimesitylacetylene, IV); (c) asymmetrically trisubstituted in *ortho* position (xylylmesitylacetylene, V).

Synthesis of acetylenes

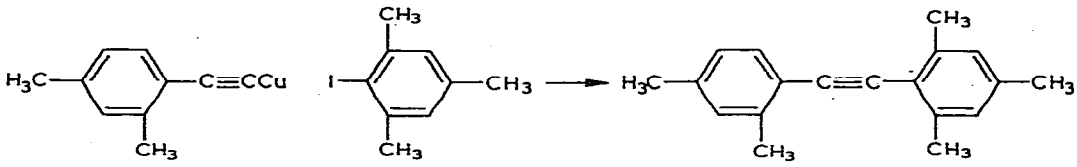
The symmetrically substituted diphenylacetylenes I—IV were prepared by treatment of trichlorocyclopropenylium chloroaluminate (VI) with the appropriate aromatic hydrocarbons [7], followed by the photochemically induced decarbonylation [7,8] of the diarylcyclopropenones VII—X.



(VI) (VII – X) (I—IV)

I, VII : Ar = 4-t-butylphenyl
II, VIII : Ar = 2,4-dimethylphenyl
III, IX : Ar = 2,5-dimethylphenyl
IV, X : Ar = mesityl

The asymmetrically substituted diphenylacetylene V was obtained by adapting the general method [9] based on the coupling of a cuprous arylacetylide (XI) with iodomesitylene (XII).



(XI) (XII) (V)

Results and discussions

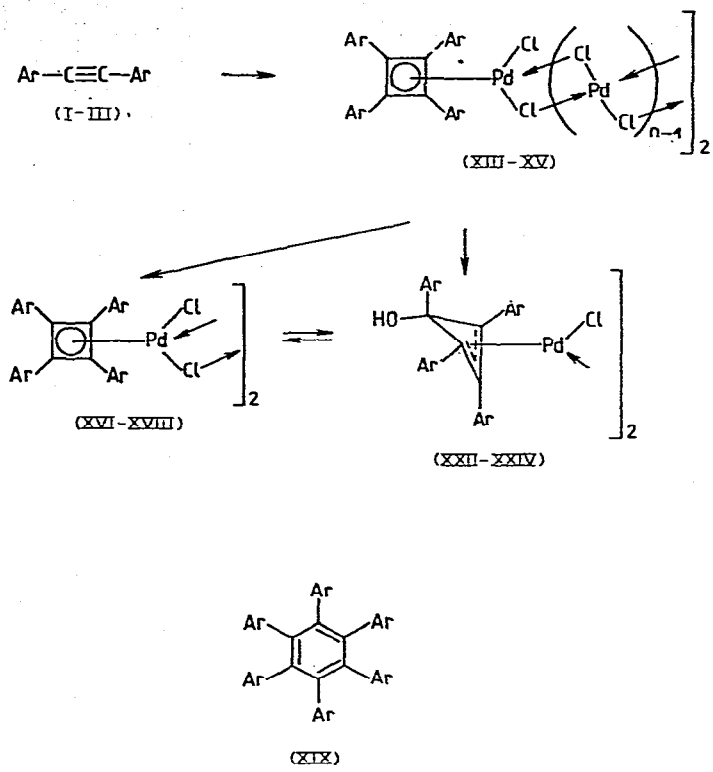
The oligomerization experiments were carried out using a standard procedure, involving treatment of the acetylene with BNP in the molar ratio 2 : 1 in benzene solution for several hours at room temperature.

Under these conditions di-4-t-butylphenylacetylene (I) and the two dixylylacetylenes II and III yielded the complexes XIII, XIV and XV, respectively, which have a composition corresponding to the pressure of acetylene residues and an excess of palladium chloride. Removal of the excess of $PdCl_2$, gave the dimeric complexes of tetraarylcyclobutadienes XVI, XVII and XVIII, respectively (Scheme 1).

From the reaction of I, the hexaarylbenzene XIX was also isolated.

The cyclobutadiene structure of complexes XVI, XVII and XVIII was ascribed on the basis of spectral data and chemical behaviour. The UV spectra (Fig. 1) are closely similar to those of tetraphenylcyclobutadiene · $PdCl_2$ (XX) [10];

SCHEME 1



I, XIII, XVI, XIX, XXII : Ar = 4-t-butylphenyl
II, XIV, XVII, XXIII : Ar = 2,4-dimethylphenyl
III, XV, XVIII, XXIV : Ar = 2,5-dimethylphenyl

for comparison the spectrum of 1,2-diphenyl-3,4-di-t-butylcyclobutadiene · $PdCl_2$ (XXI) complex are noted.

The proposed structures were also in agreement with the chemical behaviour. By treating a dimethylformamide solution of XIII—XV or XVI—XVIII, with water, the hydroxycyclobutenylic complexes XXII, XXIII and XXIV, respectively, were obtained. With hydrogen chloride XXII—XXIV regenerated the cyclobutadienic complexes XVI—XVIII.

The UV spectra of the hydroxy derivatives XXII—XXIV and those of the hydroxydiphenyl-di-t-butylcyclobutenylic complex XXV are shown in Fig. 2.

As expected, the controlled increase of the size of the acetylene substituents by introduction of methyl groups in *ortho* position of the phenyl rings supplies information on the limits of the steric control in the steps of the oligomerization. Thus, the introduction of a t-butyl group in the *para* position of the phenyl rings in I has no effect on the oligomerization process. The absence of *ortho* substituents in this acetylene, as in other similar diphenylacetylenes earlier studied [11], allowed the formation of the dimeric cyclobutadiene · $PdCl_2$ complex XVI as well as the trimerisation product XIX.

The fact that dixylylacetylenes II and III with only two *ortho*-substituted

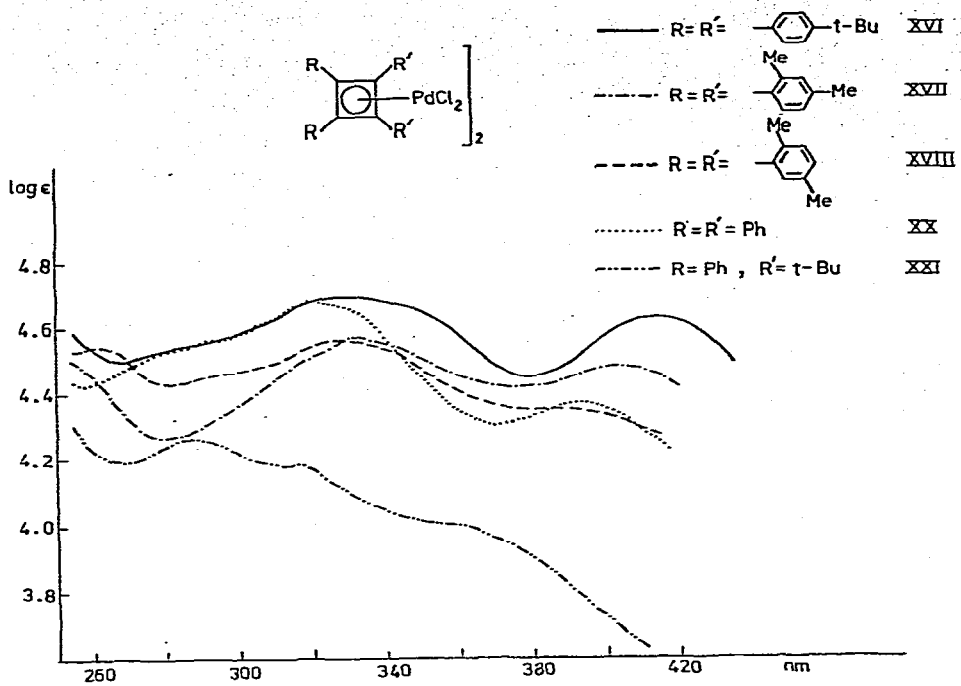


Fig. 1. UV spectra of cyclobutadienic complexes XVI—XXI.

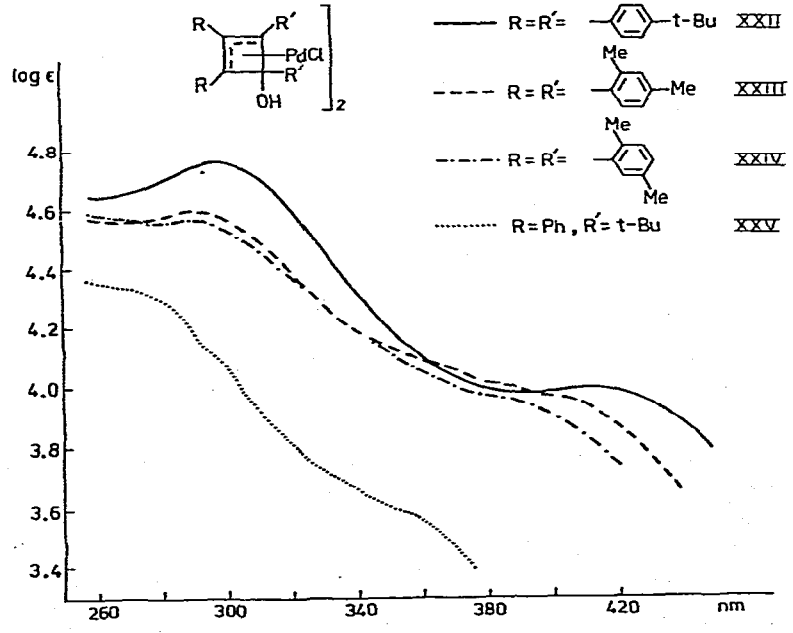


Fig. 2. UV spectra of hydroxycyclobutenylic complexes XXII—XXV.

positions yield the cyclobutadienic complexes XVII and XVIII, respectively, shows that the formation of the four-membered ring complexes occurs when the methyl-substituted diphenylacetylenes have at least one free *ortho* position on each benzene ring. With molecular models it can be observed that the rotation of the phenyl rings may lead to favourable, sterically unhindered arrangements which permit the C—C coupling for dimerisation as well as for cyclisation. Further reaction of the dimer with another molecule of acetylene is therefore disfavoured sterically and no trimer is observed. The fact that III behaves like II shows that the methyl groups in *meta*-position produce no steric hindrance.

The failure to oligomerize the diphenylacetylenes IV and V bearing four and three *ortho*-methyl groups, respectively, indicates that the steric hindrance operates even in the first steps of the reaction. This behaviour resembles that of di-t-butylacetylene which also fails to give oligomerization products [12].

Additional features of the steric effect of bulky substituents on the palladium chloride catalyzed reaction of acetylenes are under investigation.

Experimental

Microanalyses were performed by Miss E. Sliam and Mrs. V. Daniel. IR spectra were run on a double-beam UR-20-Zeiss-Jena spectrophotometer and UV spectra on a VSU-Zeiss-Jena spectrophotometer. NMR spectra were determined with a Varian A-60A spectrometer (TMS as internal standard).

Di-4-t-butylphenylcyclopropenone (VII)

A suspension of 8.0 g (60 mmol) anhydrous aluminium chloride in 13.0 g (72 mmol) tetrachlorocyclopropene [13] was locally heated in order to initiate the complexation [14]. The complex VI formed was treated with 17.7 g (132 mmol) t-butylbenzene in small portions with ice-water cooling during a period of 30 min. The mixture was then gradually heated to 70°C during 30 min, and was maintained at this temperature for 90 min. After cooling, ice water was added and the organic solid was extracted with dichloromethane. The solution was washed with water and dried, and the solvent was evaporated. The brown residue was chromatographed on alumina using petroleum ether, ether and finally dichloromethane as eluents. From the last, after distillation of the solvent, crude VII was obtained, which was purified by recrystallization from methanol, yielding 9.2 g (48.2% based on aluminium chloride) pure VII, m.p. 157°C (lit. [15] m.p. 155—156°C). IR [16] (CCl_4, cm^{-1}): ν(C=C) 1620, 1640; ν(C=O) 1868. NMR ($CDCl_3$, δ, ppm): 1.40 (s, 18H, t-butyl groups), 7.58—7.95 (*AB* system, 8H arom., *J* 8.2 Hz).

Analysis found: C, 86.55; H, 8.23. $C_{23}H_{26}O$ calcd.: C, 86.76; H, 8.23%.

Di-2,4-dimethylphenylcyclopropenone (VIII)

Using the procedure above, from 11.0 g (62 mmol) tetrachlorocyclopropene, 6.6 g (50 mmol) aluminium chloride and 11.7 g (110 mmol) *m*-xylene, 7.2 g (54.9% based on aluminium chloride) VIII were obtained, m.p. 164—165°C (methanol). IR * (CCl_4, cm^{-1}): ν(C=C) 1620; ν(C=O) 1868. NMR (CCl_4, δ,

* IR characteristic bands of cyclopropenones were assigned as suggested in ref. 16.

ppm): 2.30 (s, 6H, 2 CH_3), 2.47 (s, 6H, 2 CH_3), 7.01 (s, 2H arom.), 7.40 and 6.95 (*AB* system, 4H arom., *J* 8.0 Hz). UV (acetonitrile): λ_{max} 298.0 nm (log ϵ 4.320), 311 (4.379), 323 (4.293).

Analysis found: C, 86.60; H, 7.01. $C_{19}H_{18}O$ calcd.: C, 86.99; H, 6.91%.

Di-2,5-dimethylphenylcyclopropenone (IX)

Similarly, from 11.5 g (64 mmol) tetrachlorocyclopropene, 7.0 g (52 mmol) aluminium chloride and 11.0 g (104 mmol) *p*-xylene, 6.3 g (45.3% based on aluminium chloride) IX were obtained, m.p. 139—140°C (methanol). IR [16] (CCl_4, cm^{-1}): ν(C=C) 1625; ν(C=O) 1852, 1835. NMR ($CDCl_3$, δ, ppm): 2.42 (s, 6H, 5-CH_3 and 5'-CH_3), 2.67 (s, 6H, 2-CH_3 and 2'-CH_3), 7.23 (s, 4H arom., 3-H, 3'-H, 4-H, 4'-H), 7.66 (s, 2H arom., 6-H and 6'-H). UV (acetonitrile): λ_{max} 287 nm (log ϵ 4.288), 300 (4.287), 311 (4.281), 323 shoulder (4.140).

Analysis found: C, 87.25; H, 7.08. $C_{19}H_{18}O$ calcd.: C, 86.99; H, 6.91%.

Dimesitylcyclopropenone (X)

Similarly, from 6.5 g (45 mmol) tetrachlorocyclopropene, 4.0 g (30 mmol) aluminium chloride and 8.0 g (68 mmol) mesitylene, 5.0 g (57.4% based on aluminium chloride) X *, m.p. 189—190°C, were obtained. IR [16] (CS_2, CCl_4, cm^{-1}): ν(C=C) 1625; ν(C=O) 1848. NMR ($CDCl_3$, δ, ppm): 6.95 (s, 4H arom.), 2.33 (s, 18H, 6 CH_3). UV (acetonitrile): λ_{max} 279 nm (log ϵ 4.306), 294 shoulder (4.192), 314 (4.009), 332 (3.855).

Analysis found: C, 86.56; H, 7.87. $C_{21}H_{22}O$ calcd.: C, 86.85; H, 7.64%.

Di-4-t-butylphenylacetylene (I)

A solution of 1.0 g (3.1 mmol) VII in 25 ml benzene was irradiated under nitrogen with a high pressure UV mercury lamp (Hannau 75 W) for 12 h. The solvent was removed under vacuum and the residue was chromatographed on alumina with petroleum ether. Removal of the solvent and recrystallization from methanol yielded 0.76 g (92%) I, m.p. 175°C. NMR (CCl_4, δ, ppm): 1.34 (s, 18H, 2 t-butyl groups), 7.35 (broad s, 8H arom.). UV (cyclohexane): λ_{max} 272.0 nm (log ϵ 4.407), 280.0 (4.438), 288.0 (4.546), 297.0 (4.402), 307.0 (4.501).

Analysis found: C, 90.89; H, 9.16. $C_{22}H_{26}$ calcd.: C, 90.98; H, 9.02%.

Di-2,4-dimethylphenylacetylene (II)

A solution of 6.0 (23 mmol) VIII in 75 ml benzene was irradiated as described above and working up gave 5.2 g (96.5%) II, m.p. 78°C. NMR (CCl_4, δ, ppm): 2.30 (s, 6H, 2 CH_3), 2.47 (s, 6H, 2 CH_3), 7.01 (s, 2H arom.), 6.95 and 7.40 (*AB* system, 4H arom., *J* 8.0 Hz). UV (cyclohexane): λ_{max} 240.7 nm (log ϵ 4.272), 256.0 (4.184), 330.6 (4.267), 404.6 (4.175).

Analysis found: C, 92.16; H, 7.90. $C_{18}H_{18}$ calcd.: C, 92.26; H, 7.74%.

Di-2,5-dimethylphenylacetylene (III)

The photochemical decarbonylation of IX, carried out as described above,

* The preparation of cyclopropenone X is reported in a short communication [7] but no experimental detail or m.p.'s and spectral details are given.

gave III in 94% yield; m.p. 120°C. NMR (CCl_4, δ, ppm): 2.32 (s, 6H, 2 CH_3), 2.46 (s, 6H, 2 CH_3), 7.00 (broad s, 4H arom.), 7.25 (broad s, 2H arom.). UV (cyclohexane): λ_{max} 288.0 nm (log ε 4.411), 301.0 (4.275), 312.0 (4.362).

Analysis found: C, 92.10; H, 7.89. $C_{18}H_{18}$ calcd.: C, 92.26; H, 7.74%.

Dimesitylacetylene (IV)

Similarly, from 1.0 g (3.45 mmol) X, 0.80 g (89%) IV were obtained, m.p. 130°C (lit. [17] 127—130°C). NMR (CCl_4, δ, ppm): 2.26 (s, 6H, 2 CH_3), 2.46 (s, 12H, 4 CH_3), 6.80 (broad s, 4H arom.). UV (cyclohexane): λ_{max} 280.0 nm (log ε 4.346), 286.0 (4.389), 296.0 (4.534), 303.0 (4.428), 315.0 (4.515).

Analysis found: C, 91.79; H, 8.37. $C_{20}H_{22}$ calcd.: C, 91.55; H, 8.45%.

2,4-Dimethylphenylmesitylacetylene (V)

By condensing cuprous 2,4-dimethylphenylacetylide (XI) * with iodomesitylene (XII) ** in Stephens procedure [9], the acetylene V was obtained in 60% yield; m.p. 65°C. NMR (CCl_4, δ, ppm): 2.25 and 2.29 (2 s, 6H, 2 *p*-CH_3), 2.45 (s, 9H, 3 *o*-CH_3), 6.80 (s, 2H arom. from mesityl), 6.95 (s, 1H *meta* from xylyl), 6.89 and 7.34 (*AB* system for vicinal 2H *ortho* and *meta* from xylyl, J_{AB} 7.0 Hz). UV (cyclohexane): λ_{max} 279.0 nm (log ε 4.389), 284.0 (4.430), 293.7 (4.530), 301.0 (4.425), 312.7 (4.480).

Analysis found: C, 92.10; H, 8.22. $C_{19}H_{20}$ calcd.: C, 91.88; H, 8.12%.

Reaction of di-4-t-butylphenylacetylene (I) with BNP

Complex XIII. To a solution of 0.72 g (1.88 mmol) BNP in 50 ml benzene was added a solution of 1.10 g (3.74 mmol) acetylene I in 10 ml benzene. The reaction mixture was left at room temperature with occasional shaking. After 24 h the red-brick precipitate was filtered off and washed with benzene and petroleum ether, yielding 0.70 g (90% based on BNP and 37% based on I) complex XIII, n = 2.5, m.p. 280—300°C (dec.). NMR ($CDCl_3$, δ, ppm): 1.36 (s, 36H, 4 t-butyl groups), 7.56 (m, 16H, arom.).

Analysis found: C, 51.15; H, 5.18; Cl, 18.01; Pd, 26.33. $C_{44}H_{52}(PdCl_2)_{2.5}$ calcd.: C, 51.60; H, 5.12; Cl, 17.31; Pd, 25.97%.

From the filtrates 0.55 g (50%) hexa-4-t-butylphenylbenzene (XIX), m.p. >360°C, was isolated. NMR (C_6D_6, δ, ppm): 1.06 (s, 54H, 6 t-butyl groups), 6.90 and 7.08 (*AB* system, 24H, 6 benzenic rings *p*-disubstituted, *J* 8.0 Hz). UV (hexane): λ_{max} 253.8 nm (log ε 4.765), 276.8 (4.520).

Cyclobutadienic complex XVI. The complex XIII (0.30 g) was dissolved in 10 ml dimethylformamide and the filtered solution was treated with 3 ml concentrated hydrochloric acid with cooling. The product was filtered off, washed with petroleum ether and dried, yielding 0.19 g (85%) XVI, m.p. 285°C (dec.). NMR ($CDCl_3$, δ, ppm): 1.38 (s, 36H, 4 t-butyl groups), 7.48 and 7.83 (*AB* system, 16H arom., 4 *p*-disubstituted benzene rings, *J* 8.0 Hz). UV ($CHCl_3$): λ_{max} 327.0 nm (log ε 4.700), 413.0 (4.623).

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* 2,4-Dimethylphenylacetylene was prepared by the method used for *p*-tolylacetylene [18a]. The corresponding cuprous acetylide was obtained using the method described in ref. 18b.

** Iodomesitylene was obtained from mesitylene, iodine and peracetic acid, following the method of iodination of benzene and xylenes described in the literature [19].

Analysis found: C, 68.12; H, 7.07; Cl, 10.50; Pd, 14.30. $C_{44}H_{52}Cl_2Pd$ calcd.: C, 69.70; H, 6.91; Cl, 9.35; Pd, 14.03%.

Hydroxycyclobutenylic complex XXII. By treating a solution of 0.30 g complex XIII in 10 ml dimethylformamide with an excess of water, 0.17 g (78%) complex XXII was obtained, m.p. 180°C (dec.). IR (CH_2Cl_2, cm^{-1}): ν(OH) 3571. NMR ($CDCl_3$, δ, ppm): 1.18 (s, 18H, 2 t-butyl groups), 1.38 and 1.43 (2s, 18H, 2 t-butyl groups), 7.20—8.00 (m, 16H arom.). UV ($CHCl_3$): λ_{max} 296.5 nm (log ϵ 4.760), 408.0 (4.004).

Analysis found: C, 70.42; H, 7.21; Pd, 14.68. $C_{44}H_{53}ClOPd$ calcd.: C, 71.44; H, 7.22; Pd, 14.38%.

The hydroxy complex XXII regenerated the complex XVI on treatment in dichloromethane solution with gaseous anhydrous hydrogen chloride.

Reaction of di-2,4-dimethylphenylacetylene (II) with BNP

Complex XIV. To a solution of 1.9 g (4.9 mmol) BNP in 50 ml benzene a solution of 2.3 g (9.8 mmol) II in 10 ml benzene was added and the mixture was kept at room temperature with occasional shaking, for 72 h. The brown precipitate formed was filtered off and triturated with petroleum ether, yielding 1.4 g (83% based on BNP and 30% based on II) complex XIV, n = 2.8, brown crystals with m.p. 200°C (dec.).

In other runs, using different reaction times, values between 2 and 2.5 were found for n in the formula XIV.

Analysis found: C, 45.28; H, 4.19; Cl, 20.07; Pd, 31.12. $C_{36}H_{36}(PdCl_2)_{2.8}$ calcd.: C, 44.80; H, 3.76; Cl, 20.57; Pd, 30.87%.

Hydroxycyclobutenylic complex XXIII. 0.20 g crude complex XIV corresponding to the formula $C_{36}H_{36}(PdCl_2)_{2.8}$ was dissolved in 10 ml dimethylformamide and the filtered solution was added in portions, with stirring, to 100 ml water. After filtration the precipitate was dried to give 0.12 g (92%) of yellow crystals, which were purified by chromatography on alumina. Elution with acetone and evaporation of the solvent yielded light yellow crystals of XXIII, m.p. 195°C. IR (KBr, cm^{-1}): ν(OH) 3570. NMR ($CDCl_3$, δ, ppm): 1.76, 1.94, 2.17, 2.26, 2.33 (5 s corresponding to 24H, 8 CH_3), 6.66—7.10 (m, 9H arom.), 7.38 (d, 2H arom., J 8.0 Hz), 7.71 (d, 1H arom., J 8.0 Hz). UV ($CHCl_3$): λ_{max} 290.0 nm (log ϵ 4.592), 402.9 (3.968).

Analysis found: C, 68.13; H, 6.30; Cl, 5.51; Pd, 16.88. $C_{36}H_{37}ClOPd$ calcd.: C, 68.90; H, 5.94; Cl, 5.65; Pd, 16.95%.

Cyclobutadienic complex XVII. A stream of gaseous anhydrous hydrogen chloride was bubbled for 2 h through a solution 0.20 g hydroxy complex XXIII in methylene chloride and the closed flask was then allowed to stand overnight. Removal of the solvent in vacuum gave 0.16 g (77%) XVII, as red crystals, m.p. 300°C (dec.). NMR ($CDCl_3$, δ, ppm); 2.08 (s, 12H, 4 CH_3), 2.30 (s, 12H, 4 CH_3), 6.90—7.20 (m, 8H arom.), 7.83 (d, 4H arom., J 8.0 Hz). UV ($CHCl_3$): λ_{max} 256.0 nm (log ϵ 4.184), 330.6 (4.267), 404.6 (4.175).

Analysis found: C, 66.16; H, 6.17; Cl, 11.47; Pd, 16.35. $C_{36}H_{36}Cl_2Pd$ calcd.: C, 66.93; H, 5.62; Cl, 10.98; Pd, 16.47%.

The complex XVII was also obtained by treating a filtered solution of 0.2 g XIV in 5 ml dimethylformamide with 5 ml concentrated hydrochloric acid.

Reaction of di-2,5-dimethylphenylacetylene (III) with BNP

Complex XV. To a solution of 0.82 g (2.13 mmol) BNP in 50 ml benzene was added a solution of 1.0 g (4.27 mmol) acetylene III in 10 ml benzene. The mixture was left at room temperature for 72 h. The solid deposit formed on the walls of the flask was filtered off and triturated with petroleum ether to give 0.72 g (26% based on BNP and 17% based on III) of brown crystals of complex XV, $n = 1.5$.

Analysis found: C, 57.90; H, 5.14; Cl, 14.44; Pd, 22.64. $C_{36}H_{36}(PdCl_2)_{1.5}$ calcd.: C, 58.86; H, 4.94; Cl, 14.48; Pd, 21.72%.

In other runs values between 1.5 and 3 were found for n.

Analysis found: C, 42.59; H, 3.83; Cl, 20.36; Pd, 32.87. $C_{36}H_{36}(PdCl_2)_3$ calcd.: C, 43.21; H, 3.62; Cl, 21.26; Pd, 31.90%.

Cyclobutadienic complex XVIII. The chromatography of complex XV on alumina, using dichloromethane as eluent, gave complex XVIII, m.p. 285°C (dec.). NMR ($CDCl_3$, δ, ppm): 2.06 (s, 12H, 4 CH_3), 2.23 (s, 12H, 4 CH_3), 7.15 and 7.30 (*AB* system, 8H arom., J 8.0 Hz). UV ($CHCl_3$): λ_{max} 262.0 nm (log ϵ 4.239), 328.0 (4.263), 397.0 (4.026).

Analysis found: C, 67.37; H, 6.66; Cl, 9.04; Pd, 17.44. $C_{36}H_{36}PdCl_2$ calcd.: C, 66.93; H, 5.62; Cl, 10.98; Pd, 16.47%.

Hydroxycyclobutenylic complex XXIV. A solution of 0.30 g XV in 6 ml dimethylformamide was treated with 80 ml water, with stirring. The crude complex was filtered off, dried and chromatographed with ether on alumina affording 0.16 g (62%) XXIV, m.p. 210°C (dec.). IR (KBr, cm^{-1}): ν(OH) 3575—3610. NMR ($CDCl_3$, δ, ppm): 1.70 (s, 3H, CH_3), 1.98 (s, 6H, 2 CH_3), 2.13 (s, 15H, 5 CH_3), 6.75—7.20 (m, 9H arom.), 7.26 (s, 2H arom.), 7.65 (s, 1H arom.). UV ($CHCl_3$): λ_{max} 290.0 nm (log ϵ 4.565), 385.0 (3.960).

Analysis found: C, 69.14; H, 6.19; Cl, 5.90; Pd, 17.14. $C_{36}H_{37}ClOPd$ calcd.: C, 68.90; H, 5.94; Cl, 5.65; Pd, 16.95%.

Reaction of dimesitylacetylene (IV) with BNP

A solution of 2.62 g (10 mmol) IV in 10 ml benzene was added to a solution of 1.92 g (5 mmol) BNP in 50 ml benzene. The mixture was left at room temperature 60 h. After the removing of the solvent, the residue was triturated with petroleum ether. From the petroleum ether solutions the unchanged acetylene IV (identified by NMR spectrum) was isolated. The solid residue proved to be BNP identified also spectroscopically.

Reaction of 2,4-dimethylphenylmesitylacetylene (V) with BNP

A solution of 0.38 g (1 mmol) BNP in 13 ml benzene was added to a solution of 0.50 g (2 mmol) V in 5 ml benzene and the mixture was left at room temperature for 72 h. The solvent was then removed and the residue was triturated with petroleum ether. From the petroleum ether solutions the unchanged acetylene V was recovered; the solid residue was identified spectroscopically as BNP.

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