Dedicated to Full Member of the Russian Academy of Sciences N.S. Zefirov on occasion of his 75<sup>th</sup> anniversary

# Oxidation of Aromatic Compounds: XVII.\* Oxidative Cross-Dimerization of Diarylacetylenes in the System CF<sub>3</sub>CO<sub>2</sub>H–CH<sub>2</sub>Cl<sub>2</sub>–PbO<sub>2</sub>. Characteristic of Cation-Radicals of Diarylacetylenes by Cyclic Voltammetry and ESR Spectroscopy

## A.V. Vasil'ev and A.P. Rudenko

State Forestry Academy, St. Petersburg, 194021 Russia e-mail: aleksvasil@mail.ru

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Abstract—The oxidation of mixtures of diarylacetylene ArC=CAr and Ar'C=CAr' in a system  $CF_3CO_2H-CH_2Cl_2-PbO_2$  (0°C, 1.5 h) results in products of cross-dimerization, (*Z*)-1,2,3,4-tetraarylbut-2-ene-1,4-diones Ar(ArCO) C=C(COAr')Ar'. The routes of transformation of intermediate cation-radicals of diarylacetylenes [ArC=CAr]<sup>++</sup> into the final products of oxidative dimerization are elucidated. By cyclic voltammetry and ESR spectroscopy the high reactivity of the diarylacetylene cation-radicals is demonstrated, the character of their singly occupied molecular orbitals ( $a_2$  or  $b_1$ ) has been revealed by ESR method.

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The oxidation of diarylacetylenes with the lead dioxide in various acids (CF<sub>3</sub>CO<sub>2</sub>H [2–4], HF [5], HSO<sub>3</sub>F [6, 7]) gives products of the oxidative dimerization, (*Z*)-(*cis*)-1,2,3,4-tetraarylbut-2-ene-1,4-diones (unsaturated  $\gamma$ -diketones) Ar(ArCO)C=C(COAr)Ar, (*E*,*E*)-1,2,3,4tetraaryl-1,4-difluoro(or dichloro)butadienes (Ar) XC=C(Ar)–(Ar)C=CX(Ar) (X = F, Cl) formed as a result of the closure of a new carbon-carbon bond through the acetylene carbon atoms of the diarylacetylenes. It was shown by ESR spectroscopy that the primary intermediates in these reactions were diarylacetylene cationradicals [7]. The extension of the synthetic potential of these reaction consists in the oxidation of mixtures of various diarylacetylenes for the preparation of products of their mixed (cross-) oxidative dimerization.

The goal of this work was the study of substrate, regio, and stereoselectivity in the oxidative cross-dimerization of diarylacetylenes in the system  $CF_3CO_2H-CH_2Cl_2-$  PbO<sub>2</sub>, and also characterizing of the diarylacetylene cation-radicals by electrochemical analysis and ESR spectroscopy.

The oxidation of a mixture of diphenylacetylene (tolane) (I) and its monomethyl-substituted derivative II in the system  $CF_3CO_2H-CH_2Cl_2-PbO_2$  is presented in Scheme 1.

The products of this reaction III–V, VIa–VIc, and also  $\gamma$ -diketones VIII, IX and XI, XII (Schemes 2, 3), inseparable by column chromatography on silica gel were analyzed by GC-MS method like in [3]. The identification of separate components was performed by the presence in the mass spectra of characteristic ions [ArCO]<sup>+</sup> and [M– ArCO]<sup>+</sup> (see Experimental).

Alongside compounds V (oxidation product of compound I [2]) and VIa–VIc (a mixture of three oxidation products of compound II [3]) we observed the formation of  $\gamma$ -diketones III and IV, the products of the oxidative cross-dimerization of diarylacetylenes I and

<sup>\*</sup> For Communication XVI, see [1].



II (Scheme 1). The overall yield of compounds III–V, VIa–VIc was 46%. Therewith the cross-coupling products III and IV formed in approximately equal quantity (8 and 7% respectively).

VII

We ascribed to compounds **III** and **IV** and to the other related structures **VIII**, **IX**, **XIV** (Schemes 2–4) the *Z*-(*cis*)-location of substituents at the C=C bond by the analogy to the stereochemical structure of  $\gamma$ -diketones obtained from alkyl(or halo)-substituted diarylacetylenes in the system CF<sub>3</sub>CO<sub>2</sub>H–CH<sub>2</sub>Cl<sub>2</sub>–PbO<sub>2</sub> [2, 3].

The oxidation of tolane (I) in the mixture with 1,2-bis(4-methylphenyl)acetylene (VII) or 1,2-bis(4-fluo-rophenyl)acetylene (X) also provided the cross-dimerization products VIII, XI in 5 and 7% yields at the overall yield of all reaction products, including  $\gamma$ -diketones V,

IX, XII, 34 and 35% respectively (Schemes 2, 3).

As a result of the oxidation of the mixture of dialkylsubstituted diarylacetylenes **VII** and **XIII** alongside the corresponding  $\gamma$ -diketones **IX** and **XV** the cross-coupling product **XIV** was obtained in 8% yield (Scheme 4). The latter was isolated by chromatography in the individual state (see Experimental).

The attempts of cooxidation of tolane (I) with substrates possessing higher oxidation potential, 1,2-bis(4nitrophenyl)acetylene (XVI) and 1,2-bis(3-ethoxycarbonylphenyl)acetylene (XVII), did not afford the crossdimerization products. Thus the cross-coupling products formed only in the mixed oxidation of diarylacetylenes possessing close oxidation potentials (close HOMO energies) (Schemes 1–4).



The cation-radicals **A** arising from the one-electron oxidation of diarylacetylenes **XVIII** (Scheme 5) may react along the following most probable routes leading to the formation of a new carbon-carbon bond. The first route a consists in the reaction with the initial substrate **XVIII** to give cation-radical **B**, the second route b is the

dimerization of the cation-radicals into dication C. Further transformations of intermediates **B** and C result in  $\gamma$ -diketones **XIX**. These two routes may lead to the products of the oxidative cross-dimerization (Schemes 1–4). However the now available experimental data are insufficient for distinguishing these two reaction routes (cf. with the data of [1] on reactions of cation-radicals of acetylene compounds with electron-withdrawing groups).

It is hardly possible that in the studied system  $CF_3CO_2H-CH_2Cl_2-PbO_2$  an alternative route *c* materializes for cation-radicals **A** (Scheme 5), since the tolane (**I**) oxidation in the presence of a nucleophilic additive biodium or silver trifluoroacetates in the systems  $CF_3CO_2H-CF_3CO_2Na$  (or  $CF_3CO_2Ag-PbO_2$ ) results in

Scheme 3.



Scheme 4.



#### Scheme 5.



obtaining benzil PhCOCOPh. Only traces of  $\gamma$ -diketone V were found in the reaction product, whereas it is the main (yield 60%) product of tolane (I) oxidation in the system CF<sub>3</sub>CO<sub>2</sub>H–CH<sub>2</sub>Cl<sub>2</sub>–PbO<sub>2</sub> [2]. This shows that the vinyl radical Ph(CF<sub>3</sub>COO)C=CPh (species **D**, Ar = Ph, Scheme 5) formed by the reaction of the nucleophile CF<sub>3</sub>CO<sub>2</sub>– with tolane cation-radical suffers further oxidation into benzil and did not take part in the formation of the carbon-carbon bond.

Diarylacetylene cation-radicals **A** were additionally studied in this work by electrochemical analysis and ESR spectroscopy.

The characteristics of oxidation of acetylene compounds **XX–XXVII** are compiled in Table 1 obtained by cyclic voltammetry, and a typical voltammogram is shown in Fig. 1.

As follows from voltammograms, in oxidation of compounds **XXI**, **XXIII**, **XXIV**, **XXVI**, **XXVII** a totally irreversible electron transfer occurs demonstrating the

2 ų́A



**Fig. 1.** Cyclic voltammogram of bis-(pentamethylphenyl)acetylene (**XX**) oxidation (concentration 2 mmol  $l^{-1}$ ) in CH<sub>2</sub>Cl<sub>2</sub>– Bu<sub>4</sub>N+PF<sub>6</sub><sup>-</sup> (0.1 mol  $l^{-1}$ ) at 25°C, saturated calomel electrode, scanning rate 0.1 V/s.

high reactivity of the corresponding cation-radicals. Formerly an irreversible electrochemical oxidation was also observed for the other methyl- and methoxy-substituted diarylacetylenes [8, 9].

The increase in the extent of the alkyl substitution favors the stabilization of cation-radicals. A pseudoreversible electron transfer was observed in oxidation of hexa- and decamethyl derivatives **XXII** and **XX** (Fig. 1), since the difference between the potentials of the anode and cathode peaks 149 and 82 mV respectively exceeded the theoretical value for reversible processes of 57 mV. The easily oxidizable substrates **XXV**, **XXVII** have two half-waves of oxidation (Table 1). However even at high scanning rate (4 V/s) the oxidation of diarylacetylene **XXV** was not completely reversible. It means that in the given electrochemical conditions the cation-radicals gradually reacted.



Fig. 2. ESR spectrum of cation-radical  $XX^+$  generated by oxidation of bis(pentamethylphenyl)acetylene XX in a system NO+SbCl<sub>6</sub>--CH<sub>2</sub>Cl<sub>2</sub> at -70°C.

1286





1.030

1.196a

	a 1	1 10
•	Vacand	halt mana
а.	SPOOL	11/21/1=\x//21//P

XXIV

XXV

XXVI

XXVII

Cation-radicals XX<sup>++</sup>, XXII<sup>++</sup>, XXVIII<sup>++</sup> were characterized by ESR spectra registered at the oxidation of the corresponding acetylene compounds XX, XXII, XXVIII in the systems NO<sup>+</sup>SbCl<sub>6</sub><sup>-–</sup>CH<sub>2</sub>Cl<sub>2</sub> (–70°C) and HSO<sub>3</sub>F–PbO<sub>2</sub> (–75°C) (Table 2).

1.414

1.292

1.633a

1.473 (1.37 [8])

1.300 (1.17 [8])

1.453a

The ESR spectra of these cation-radicals are similar and consist of multiplets of over 20 equidistant peaks (Fig. 2). The spectra of these species are also like the spectrum of a cation-radical of 2,2',4,4',6,6'-hexamethylbiphenyl (bimesityl) with the following parameters  $a_{p-Me}^{H}$  2.1 (2.19) Gs (12H),  $a_{m-arom}^{H}$  <0.13 (0.5) Gs (4H),  $a_{p-Me}^{H}$  6.99 (8.79) Gs (6H) [10, 11]. The largest hyperfine interaction constant in the ESR spectra of species **XX**<sup>+</sup>, **XXII**<sup>+</sup>, **XXVIII**<sup>+</sup> and the bimesityl cation-radical was observed for the protons of the *para*-methyl groups,  $a^{H} \sim 7...10$  Gs. The very small value  $a^{H} \leq 0.5$  Gs characterized the moieties in the *meta*-position of the aromatic rings for protons, methyl and nitro groups. These values

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262

437

0.1

4

0.1

0.1

Cation-radical	Oxidation system	Constants of hyperfine interaction <sup>a</sup>
$\begin{bmatrix} Me & Me & Me & Me \\ Me & C \equiv C & Me \\ Me & Me & Me & Me \end{bmatrix}^{+ \cdot}$ $XX^{+ \cdot}$	NO+SbCl <sub>6</sub> <sup>-</sup> -CH <sub>2</sub> Cl <sub>2</sub> , -70°C	$a_{o-Me}^{H} 2 \text{ Gs (12H)},$ $a_{m-Me}^{H} < 0.5 \text{ Gs (12H)},$ $a_{p-Me}^{H} 8 \text{ Gs (6H)}$
$\begin{bmatrix} H & Me & Me & H \\ Me & C \equiv C & Me & H \\ H & Me & Me & H \end{bmatrix}^{+ \cdot}$ $XXII^{+ \cdot}$	NO+SbCl <sub>6</sub> CH <sub>2</sub> Cl <sub>2</sub> , -70°C	$a_{p-Me}^{H} 2 \text{ Gs (12H)},$ $a_{m-arom}^{H} < 0.5 \text{ Gs (4H)},$ $a_{p-Me}^{H} 8 \text{ Gs (6H)}$
$\begin{bmatrix} H & Me & Me & H \\ Me & C \equiv C & Me \\ O_2 N & Me & Me & NO_2 \end{bmatrix}^{+ \cdot}$ <b>XXVIII</b> <sup>+.</sup>	HSO <sub>3</sub> F–PbO <sub>2</sub> , –75°C	$a_{o-Me}^{H} 2.5 \text{ Gs (12H)},$ $a_{m-arom}^{H} < 0.5 \text{ Gs (2H)},$ $a_{NO_2}^{H} < 0.5 \text{ Gs (2N)},$ $a_{p-Me}^{H} 10 \text{ Gs (6H)}$

**Table 2.** ESR spectra of cation-radicals XX<sup>++</sup>, XXII<sup>++</sup>, XXVIII<sup>++</sup> generated from compounds XX, XXII, XXVIII) respectively in systems NO<sup>+</sup>SbCl<sub>6</sub><sup>--</sup>-CH<sub>2</sub>Cl<sub>2</sub> (-70°C) and HSO<sub>3</sub>F-PbO<sub>2</sub> (-75°C) (g-factors ~2.000)

<sup>a</sup> Ortho, meta, and para positions with respect to the acetylene substituent.

of hyperfine interaction constants indicate the singly occupied molecular orbitals (SOMOs), previous HOMOs of the neutral molecules, of the  $b_1$  type in the aromatic systems of these species. In the cation-radicals of diarylacetylenes with the singly occupied molecular orbital  $b_1$  the acetylene bond transmits well the spin density between the aromatic rings and is essentially involved in its delocalization (Fig. 3) (see also the data of [1] on the characteristic by ESR of cation-radicals of acetylene compounds with electron-withdrawing groups). The presence of the spin density on the acetylene carbon atoms results in the formation of the new carbon-carbon bond just at thgese atoms (Scheme 5).

We failed to register the ESR spectrum at the oxidation of octamethyl-substituted diarylacetylene **XXI** in the system NO+SbCl<sub>6</sub>--CH<sub>2</sub>Cl<sub>2</sub> (-70°C) since the corresponding cation-radical **XXI**<sup>+-</sup> proved to be exclusively unstable that was also confirmed by the irreversible character of the electrochemnical oxidation of compound **XXI** (Table 1). The singly occupied molecular orbital characteristic of cation-radical **XXI**<sup>+-</sup> is  $a_2$  with a weak involvement of the acetylene fragment into the delocalization of the spin density (Fig. 3).

Hence we demonstrated the high reactivity of diary-



Fig. 3. Character of singly occupied molecular orbital in cation-radicals XXI+ and XXII+ substituent...

lacetylene cation-radicals by cyclic voltammetry and ESR spectroscopy. By ESR method we established the SOMO type  $a_2$  or  $b_1$  of these species governing the direction of reaction of the cation-radicals [2–4].

### **EXPERIMENTAL**

<sup>1</sup>H NMR spectra were registered on a spectrometer Bruker AM-500 (operating frequency 500 MHz) from solution of compounds in  $CDCl_3$  and  $(CD_3)_2CO$ . The residual proton signals of solvents were used as internal references [CHCl<sub>3</sub>  $\delta_{\rm H}$  7.25 ppm, (CD<sub>3</sub>)(CD<sub>2</sub>H)CO  $\delta_{\rm H}$  2.05 ppm]. Mass spectra were recorded on an instrument MKh-1321, ionizing energy 70 eV, direct sample admission into the ion source at 100-120°C. GC-MS analysis was carried out on a mass spectrometer Hewlett Packard 5995, ionizing energy 70 eV, temperature of the separator 240°C, of the ion source, 250°C. Capillary quartz column 25 m  $\times$  0.32 mm, stationary phase Ultra-2 (95% of methylsilicone, 5% of phenylmethylsilicone) 0.53 µm. Oven temperature: starting 100°C, final 240°C, heating rate 5 deg min<sup>-1</sup>. Carrier gas helium, flow rate 1 ml min<sup>-1</sup>. The sample of 3–5% solution 2 µL. ESR spectra of cation-radicals were recorded on ESR spectrometers Varian E-109 and Bruker, the g-factor value was measured with respect to diphenylpicrylhydrazyl. The ESR spectra were simulated using the program WINEPR Sim-Fonia. The experiments of voltammetry were performed on an instrument BAS-100 Electrochemical Analyzer.

Diarylacetylenes were prepared by methods [2, 3]. The properties of compounds I, VII, X, XIII, XVI, XVII, XXIII, XXIV, XXVI, XXVII are published in [2], of compound II, in [3], of compound XXVIII, in [7].

**Bis(pentamethylphenyl)acetylene (XX),** mp 251– 253°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>), δ, ppm: 2.24 s (12H, 4Me), 2.25 s (6H, 2Me), 2.55 s (12H, 4Me). Found, %: C 90.92; H 9.60. C<sub>24</sub>H<sub>30</sub>. %: C 90.51; H 9.49.

**Bis(2,3,5,6-tetramethylphenyl)acetylene (XXI),** mp 235–237°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 2.25 s (12H, 4Me), 2.48 s (12H, 4Me), 6.92 s (2H<sub>arom</sub>). Found, %: C 91.02; H 9.00. C<sub>22</sub>H<sub>26</sub>. Calculated, %: C 90.98; H 9.02.

**Bis(2,4,6-trimethylphenyl)acetylene (XXII),** mp 128–130°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 2.31 s (6H, 2Me), 2.50 s (12H, 4Me), 6.92 s (4H<sub>arom</sub>). Found, %: C 91.67; H 8.31. C<sub>20</sub>H<sub>22</sub>. Calculated, %: C 91.55; H 8.45.

**Bis(4-methyl-2,5-dimethoxyphenyl)acetylene** (XXV), mp 143–145°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),

δ, ppm: 2.24 s (6H, 2Me), 3.81 s (6H, 2MeO), 3.89 s (6H, 2MeO), 6.71 s (2H<sub>arom</sub>), 6.98 s (2H<sub>arom</sub>). Found, %: C 73.58; H 6.88.  $C_{20}H_{22}O_4$ . Calculated, %: C 73.60; H 6.79.

Oxidation of mixtures of diarylacetylenes I, II, VII, X, XIII, XVI, XVII in the system  $CF_3CO_2H-CH_2Cl_2-PbO_2$ . To a solution of 0.43 ml of  $CF_3CO_2H$  in 4 ml of  $CH_2Cl_2$  was added while vigorous stirring at 0°C 1 mmol of each diarylacetylene. Then 478 mg (2 mmol) of PbO<sub>2</sub> was added, and the mixture was stirred for 1.5 h. On completion of the reaction the mixture was poured into 50 ml of chloroform, the extract was washed with water, with saturated solution of NaHCO<sub>3</sub>, again with water, and dried with Na<sub>2</sub>SO<sub>4</sub>. The solvents were distilled off, the residue was subjected to column chromatography on silica gel (eluent petroleum ether–ethyl ether) or was analyzed by GC-MS method.

The properties of compounds V, IX, XII, XV are published in [2], of compounds VIa–VIc, in [3].

2-(4-Methylphenyl)-1,3,4-triphenylbut-2-ene-1,4dione (III) and 1-(4-methylphenyl)-2,3,4-triphenylbut-2-ene-1,4-dione (IV) were obtained alongside compounds V (yield 10%) and compounds VIa–VIc (yield 21%) as a result of oxidation of a mixture of diarylacetylenes I and II. The mixture of reaction products was analyzed by GC-MS.

Compound **III**. Yield 8%. Mass spectrum, m/z ( $I_{OrH,}$ , %): 402 (11) [M]<sup>+</sup>, 297 (23) [M – PhCO]<sup>+</sup>, 105 (100) [PhCO]<sup>+</sup>, 77 (42) [Ph]<sup>+</sup>.

Compound IV. Yield 7%. Mass spectrum, m/z ( $I_{rel}$ , %): 402 (12)  $[M]^+$ , 297 (17)  $[M - PhCO]^+$ , 283 (19)  $[M - 4-MeC_6H_4CO]^+$ , 119 (100)  $[4-MeC_6H_4CO]^+$ , 105 (92)  $[PhCO]^+$ , 91 (67)  $[4-MeC_6H_4]^+$ , 77 (50)  $[Ph]^+$ .

**1,2-Bis(4-methylphenyl)-3,4-diphenylbut-2-ene-1,4-dione (VIII)** was obtained alongside compounds V (yield 20%) and IX (yield 9%) by oxidation of a mixture of diarylacetylenes I and VII. The mixture of reaction products was analyzed by GC-MS. Yield 5%. Mass spectrum, m/z ( $I_{rel}$ , %): 416 (14) [M]<sup>+</sup>, 311 (20) [M – PhCO]<sup>+</sup>, 297 (24) [M – 4-MeC<sub>6</sub>H<sub>4</sub>CO]<sup>+</sup>, 119 (100) [4-MeC<sub>6</sub>H<sub>4</sub>CO]<sup>+</sup>, 105 (95) [PhCO]<sup>+</sup>, 91 (70) [4-MeC<sub>6</sub>H<sub>4</sub>]<sup>+</sup>, 77 (58) [Ph]<sup>+</sup>.

**1,2-Diphenyl-3,4-bis(4-fluorophenyl)but-2-ene-1,4dione (XI)** was obtained alongside compounds V (yield 23%) and XII (yield 5%) by oxidation of a mixture of diarylacetylenes I and X. The mixture of reaction products was analyzed by GC-MS. Yield 7%. Mass spectrum, m/z ( $I_{rel}$ , %): 424 (10) [M]+, 319 (18) [M – PhCO]+, 301 (17) [M – 4-FC<sub>6</sub>H<sub>4</sub>CO]+, 123 (100) [4-FC<sub>6</sub>H<sub>4</sub>CO]+, 105 (87) [PhCO]+, 95 (60) [4-FC<sub>6</sub>H<sub>4</sub>]+, 77 (71) [Ph]+.

1,2-Bis(4-tert-butylphenyl)-3,4-bis(4-methylphenyl)but-2-ene-1,4-dione (XIV) was obtained alongside compounds IX (yield 7%) and XV (yield 10%) by oxidation of a mixture of diarylacetylenes VII and XIII. After performing the reaction the mixture of products was subjected to column chromatography on silica gel. Compound XIV. Yield 5%, mp 209-211°C. <sup>1</sup>H NMR spectrum [(CD<sub>3</sub>)<sub>2</sub>CO],  $\delta$ , ppm: 1.23 s (9H, *t*-Bu), 1.26 s (9H, *t*-Bu), 2.23 s (3H, Me), 2.29 s (3H, Me), 7.04 s (4H<sub>arom</sub>), 7.13 d (2H<sub>arom</sub>, J 8.4 Hz), 7.15 d (2Harom, J 8.2 Hz), 7.29 d (2Harom, J 8.4 Hz), 7.41 d (2H<sub>arom</sub>, J 8.3 Hz), 7.71 d (2H<sub>arom</sub>, J 8.2 Hz), 7.81 d  $(2H_{arom}, J 8.3 \text{ Hz})$ . Mass spectrum, m/z ( $I_{rel}$ , %): 528 (21)  $[M]^+, 443 (8), 409 (10) [M - 4-MeC_6H_4CO]^+, 367 (11)$  $[M - 4 - t - BuC_6H_4CO]^+$ , 161 (88)  $[4 - t - BuC_6H_4CO]^+$ , 119 (100)  $[4-MeC_6H_4CO]^+$ , 91 (25)  $[4-MeC_6H_4]^+$ . Found, %: C 86.30; H 7.70. C<sub>38</sub>H<sub>40</sub>O<sub>2</sub>. Calculated, %: C 86.32; H 7.63. M 528.30.

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