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Oxidation of Aromatic Compounds: XV.* Oxidation of Arylacetylenes in a System HF–PbO_2

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Abstract—The oxidation of symmetrically substituted diarylacetylenes ArC≡CAr (Ar=C_6H_4R) containing strong electron-withdrawing groups R = 4-COMe, 4-CO_2Me, 3-CO_2Et, and 4-NO_2 in a system HF–PbO_2 at –10÷–20°C led within 0.5–3 h to the formation of Z,Z-1,2,3,4-tetrakis(aryl)-1,4-difluorobuta-1,3-dienes ArFC=C(Ar)–(Ar)C=CFAr. The butadiene structures obtained exist in solutions as *s-cis*- and *s-trans*-conformers and in the crystalline state are present in the stable *s-cis*-form.

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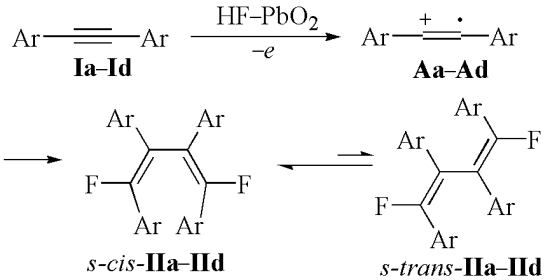
Alkynes oxidation is widely employed in the organic synthesis [2]. Single-electron transfer reactions involving acetylene derivatives make it possible to prepare quite a number of synthetically valuable multifunctional compounds [1, 3–7].

We formerly demonstrated that the oxidation of diarylacetylenes in a system CF_3CO_2H–CH_2Cl_2–PbO_2 proceeding through an intermediate formation of diarylacetylenes cation radicals resulted in the products of oxidative dimerization, 1,2,3,4-tetraarylbut-2-ene-1,4-diones [4–6]. The essential drawback of this system was insufficient oxidative potential of the lead dioxide for efficient activation of diarylacetylenes into the cation radical state when the diarylacetylenes contained two strong electron-withdrawing groups COMe, CO_2Me, NO_2, CN etc. [4–6]. The potential of PbO_2 is enhanced in the presence of acids stronger than CF_3CO_2H, namely, of HSO_3F and HF [8, 9]. The chemical [10] and electrochemical [11] oxidation of organic compounds in an anhydeous liquid hydrogen fluoride is of special importance for the preparation of fluorinated derivatives.

The target of the present study was investigation of reactions of diarylacetylenes with strong electron-withdrawing groups in the system HF–PbO_2.

Scheme 1 represents the oxidation of compounds **Ia–Id** with lead dioxide in anhydrous liquid hydrogen fluoride at –10÷–20°C for 0.5–3 h to give (*Z,Z*)-1,2,3,4-tetrakis(aryl)-1,4-difluorobuta-1,3-dienes **IIa–IId** through a stage of formation of cation radicals **A**a–**A**d. The oxidation under the same conditions (–20°C, 3 h) of 1,2-diphenylacetylene (tolan) (**Ie**) according to the data of ^{1}H and ^{19}F NMR spectra provided a mixture of polyfluorinated oligomeric products. The process carried out in the presence of a nucleophilic additive , sodium fluoride (molar ratio tolan:NaF:HF = 1:2:50) also resulted in oligomeric compounds. The analogous reaction of diarylacetylene **Id** in the system HF–NaF–PbO_2 did not increase the yield of the target product **IId** as compared with the oxidation in the system HF–PbO_2 (see EXPERIMENTAL).

Scheme 1.



Ar = C_6H_4R; R = 4-COMe (**a**), 4-CO_2Me (**b**), 3-CO_2Et (**c**), 4-NO_2 (**d**).

* For Communication XIV, see [1].

Table 1. Ratio of *s-cis*- and *s-trans*-isomers of compounds **IIa–IId** in $CDCl_3$ at 20°C

Compd. no.	Isomer content, %	
	s-cis	*s-trans*
IIa	74	26
IIb	73	27
IIc	75	25
IId	77	23

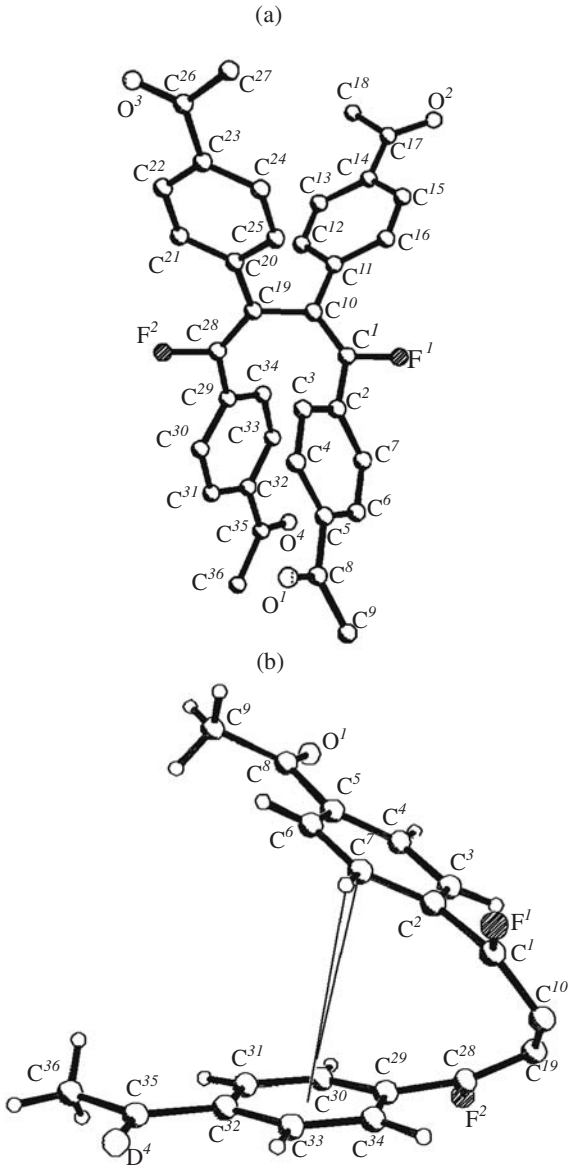
Table 2. ^{19}F NMR spectra and the ratio of *s-cis*- and *s-trans*-isomers in solution of (*Z*,*Z*)-1,2,3,4-tetrakis(4-methylcarbonylphenyl)-1,4-difluorobuta-1,3-diene (**IIa**) in $(CD_3)_2SO$ at various temperatures

T, °C	Isomer content, % (δ, ppm)	
	s-cis	*s-trans*
20	74 (–93.15)	26 (–145.38)
60	67 (–93.56)	33 (–145.98)
90	56 (–93.15)	44 (–145.38)

X-ray diffraction analysis revealed that compound **IIa** existed in the crystalline state solely in the *s-cis*-conformation (see the figure). However the ^1H and ^{19}F NMR spectra of solutions of compounds **IIa–IId** in $CDCl_3$ contain additional signals due to the presence in the solutions of dienes **IIa–IId** alongside the prevailing *s-cis*- conformers also *s-trans*- (minor) conformers [12] (Scheme 1). The ratio of *s-cis*- and *s-trans*-forms of compounds **IIa–IId** estimated from the areas of the corresponding signals in the ^1H and ^{19}F NMR spectra (Table 1) was virtually independent of the substituents.

In solutions the *s-cis*- and *s-trans*-isomers are involved in a reversible temperature equilibrium. As seen from Table 2, at growing temperature the fraction of the *s-trans*-conformer **IIa** increased.

The presence in solution of *s-cis*- and *s-trans*-conformers simultaneously is due to the hindered rotation around the central C–C bond of the butadiene system in compounds **IIa–IId** caused by the four streically bulky aryl substituents contained in the structure. Only the *s-cis*-form quantitatively crystallizes from solution which is probably stabilized by the π–π interaction [13] between aromatic systems in positions *1* and *4* of the butadiene system. The analysis of the structure of molecule **IIa** shows that the aryl rings C^{2-7} and C^{29-34} are partially located over each other (see the figure). The dihedral angle between the planes of these rings is 30.1 deg. The shortest distances between the center of C^{29-34} ring and atoms C^7, H^7 of the other aromatic ring are respectively



Molecular structure of (*Z*,*Z*)-1,2,3,4-tetrakis(4-methylcarbonylphenyl)-1,4-difluorobuta-1,3-diene (**IIa**) (a). A fragment of **IIa** molecule demonstrating the mutual position of aryl rings C^{2-7} and C^{29-34} (b). Main bond lengths in the butadiene fragment of structure **IIa**: F^1–C^1 1.365(2), F^2–C^{28} 1.360(2), C^1–C^{10} 1.329(2), C^{10}–C^{19} 1.493(2), C^{19}–C^{28} 1.335(2) Å.

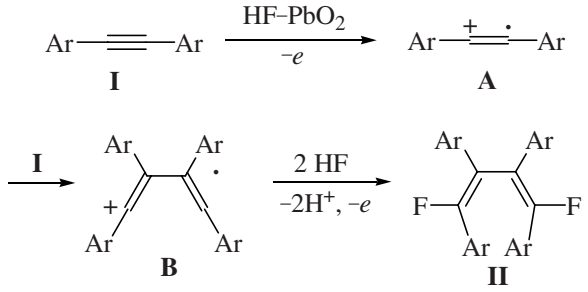
3.887 and 3.89 Å; according to [13] this fact may evidence an additional H-π interaction. This is indirectly confirmed by the deviation of the fluorine atoms from the corresponding planes of the aromatic rings. Torsion angles $F^1C^1C^2C^7$ and $F^1C^{28}C^{29}C^{34}$ are respectively –32.1 and 31.2 deg indicating the turn of the aryl fragments with respect to the C^1–C^2 and C^{28}–C^{29} bonds. Diene fragment $C^1C^{10}C^{19}C^{28}$ does not lie in a common plane due to the significant spatial dimensions of the four aryl

groups. Therefore the C–C distances within this fragment correspond to a system with an incomplete conjugation.

A large difference in the chemical shifts of fluorine signals (about 50 ppm) in the ^{19}F NMR spectra of *s-cis*- and *s-trans*-isomers **IIa–IId** stands out (see EXPERIMENTAL and Table 2). These findings are consistent with published data [14] demonstrating that depending on intramolecular surrounding of fluorine atoms in the terminal positions of various conjugated butadiene systems their signals appear in the ^{19}F NMR spectra in a wide range of chemical shifts (δ –92÷–143 ppm).

The presumable mechanism of formation of diene structure **II** (Scheme 2) involves the interaction of the primary cation radical **A** with the molecule of the initial substrate resulting in generation of a new carbon-carbon bond between the acetylene carbon atoms [1]. The intermediate cation radical of **B** type suffers further a single-electron oxidation and a reaction with HF molecules to give the final difluoride **II**. Therewith the overall pathway of initial compound **I** transformation into the product of the oxidative dimerization **II** includes a transfer of two electrons (Scheme 2) (cf. with data of [1]). Further oxidation of dienes **IIa–IId** in the system HF–PbO_2 is hampered by the deactivating effect of four aryl fragments with strong electron-withdrawing groups.

Scheme 2.



EXPERIMENTAL

^1H and ^{19}F NMR spectra were registered on a spectrometer Bruker AM-500 at operating frequencies 500 and 470.7 MHz respectively in solutions of $CDCl_3$, $(CD_3)_2SO$, or $(CD_3)_2CO$. As internal references in ^1H NMR spectra residual signals were used of $CHCl_3$ (δ 7.25 ppm), $(CD_3)(CD_2H)SO$ (δ 2.50 ppm), and $(CD_3)(CD_2H)CO$ (δ 2.05 ppm), in ^{19}F NMR spectrum, signal of $CFCl_3$ (δ 0.0 ppm). IR spectra were recorded on a spectrophotometer Specord 75IR from solutions of compounds in $CHCl_3$. Mass spectra were measured on MKh-1321 instrument, ionizing electrons energy 70 eV, direct admission in the ion source at the heater temperature 100–120°C.

X-ray study of the single crystal of compound **IIa** was carried out on an automatic diffractometer Smart APEX (graphite monochromator, MoK_α radiation, ω–θ scanning). A single crystal of compound **IIa** of dimensions 0.32×0.18×0.10 mm for X-ray study (see the figure) was obtained by keeping at room temperature for several days a solution of compound **IIa** in DMSO saturated at 50–60°C. Crystals of $C_{36}H_{28}F_2O_4$ at 293(2) K triclinic, *a* 10.7972(6), *b* 11.3357(7), *c* 12.1028(7) Å, α 90.056(1), β 102.155(1), γ 97.440(1) deg, *V* 1435.30(15) Å^3, *Z* 2, space group P-1, d_{calc} 1.302 g/cm^3, μ 0.093 mm^{-1}, $1.72 \le \theta \le 26.00°$, 12432 reflections were measured, among them 5615 independent reflections (R_{int} 0.0245), R_1 0.0460 [$I > 2\sigma(I)$], wR_2 0.1047 (for all reflections). The structure was solved by the direct method and refined by the least-squares method for F^2_{hkl} in anisotropic approximation for all nonhydrogen atoms. The hydrogen atoms were obtained from the difference Fourier synthesis and were refined isotropically. All calculations were carried out with the use of SHELXTL v. 6.10 software [15].

The preparation procedures and properties of 1,2-bis(4-methylcarbonylphenyl)acetylene (**Ia**), 1,2-bis(4-methoxycarbonylphenyl)acetylene (**Ib**), 1,2-bis(3-ethoxycarbonylphenyl)acetylene (**Ic**), 1,2-bis(4-nitrophenyl)acetylene (**Id**), and 1,2-diphenylacetylene (**Ie**) we published before [4, 5].

Diarylacetylenes Ia–Id oxidation in a system HF–PbO_2. General procedure**.** In 2.5–10 ml of anhydrous liquid hydrogen fluoride at –10÷–20°C was dissolved 0.2–0.75 mmol of acetylene compound **Ia–Id**, 0.2–0.75 mmol of PbO_2 was added, and the reaction mixture was stirred for 0.5–3 h at –10÷–20°C. On completion of the reaction the mixture was kept in an open vessel under a strong hood at 25°C to evaporate HF. The residue was treated with a saturated $NaHCO_3$ solution (100 ml) and extracted with chloroform (3×50 ml). The extract was washed with water solution of $NaHCO_3$ and with water, dried over Na_2SO_4, the solvent was distilled off under a vacuum of a water-jet pump. The solid residue was subjected to separation by preparative TLC on silica gel, eluent hexane–chloroform. Yield of compounds **IIa–IId** was estimated from the weight of fractions obtained by chromatography.

(*Z,Z*)-1,2,3,4-Tetrakis(4-methylcarbonylphenyl)-1,4-difluorobuta-1,3-diene (IIa) was obtained by

oxidation of 53 mg (0.2 mmol) of diarylacetylene **Ia** with 48 mg (0.2 mmol) of PbO_2 in 2.5 ml HF at –20°C within 0.5 h. Yield 20 mg (36%), mp 248–250°C.

Here and hereinafter the spectra of *s-cis*- and *s-trans*-forms were extracted from the spectra of mixtures.

s-cis-Form. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.55 s (6H, 2COMe), 2.56 s (6H, 2COMe), 7.24 d ($4H_{arom}$, *J* 8.4 Hz), 7.70 d ($4H_{arom}$, *J* 8.4 Hz), 7.73 ($4H_{arom}$, *J* 8.3 Hz), 7.90 ($4H_{arom}$, *J* 8.3 Hz). ^{1}H NMR spectrum [$(CD_3)_2SO$], δ, ppm: 2.54 s (6H, 2COMe), 2.55 s (6H, 2COMe), 7.22 d ($4H_{arom}$, *J* 8.3 Hz), 7.79 d ($4H_{arom}$, *J* 8.3 Hz), 7.85 ($4H_{arom}$, *J* 8.3 Hz), 7.94 ($4H_{arom}$, *J* 8.3 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ, ppm: –91.17 s.

s-trans-Form. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.58 s (6H, 2COMe), 2.61 s (6H, 2COMe), 7.59 d ($4H_{arom}$, *J* 8.5 Hz), 7.71 d ($4H_{arom}$, *J* 8.4 Hz), 7.94 d ($4H_{arom}$, *J* 8.5 Hz), 8.00 d ($4H_{arom}$, *J* 8.4 Hz). ^{1}H [$(CD_3)_2SO$], δ, ppm: 2.58 s (6H, 2COMe), 2.60 s (6H, 2COMe), 7.65 d ($4H_{arom}$, *J* 8.4 Hz), 7.73 d ($4H_{arom}$, *J* 8.6 Hz), 8.02 d ($4H_{arom}$, *J* 8.6 Hz), 8.05 d ($4H_{arom}$, *J* 8.4 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ, ppm: –145.92 s. Mass spectrum: *m/z* 562 $[M]^+$. Found, %: C 76.98; H 4.90. $C_{36}H_{28}F_2O_4$. Calculated, %: C 76.85; H 5.02. *M* 562.20.

(*Z,Z*)-1,2,3,4-Tetrakis(4-methoxycarbonylphenyl)-1,4-difluorobuta-1,3-diene (IIb) was obtained by oxidation of 136 mg (0.46 mmol) of diarylacetylene **Ib** with 111 mg (0.46 mmol) of PbO_2 in 5 ml HF at –20°C within 1 h. Yield 26 mg (18%), mp 198–201°C. IR spectrum, ν, cm^{-1} (conformers mixture):1710 (C=O), 1600 (C=C).

s-cis-Form. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.88 s (6H, $2CO_2Me$), 3.90 s (6H, $2CO_2Me$), 7.20 d ($4H_{arom}$, *J* 8.4 Hz), 7.70 d ($4H_{arom}$, *J* 8.2 Hz), 7.79 d ($4H_{arom}$, *J* 8.4 Hz), 7.98 d ($4H_{arom}$, *J* 8.2 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ, ppm: –91.30 s.

s-trans-Form. ^{1}H ($CDCl_3$), δ, ppm: 3.82 s (6H, $2CO_2Me$), 3.84 s (6H, $2CO_2Me$), 7.24 d ($4H_{arom}$, *J* 8.4 Hz), 7.66 d ($4H_{arom}$, *J* 8.4 Hz), 7.82 d ($4H_{arom}$, *J* 8.4 Hz), 7.92 d ($4H_{arom}$, *J* 8.4 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ, ppm: –141.23 s. Mass spectrum: *m/z* 626 $[M]^+$. Found, %: C 68.87; H 4.53. $C_{36}H_{28}F_2O_8$. Calculated, %: C 69.01; H 4.50. *M* 626.18.

1,4-Difluorobuta-(*Z,Z*)-1,2,3,4-tetrakis(3-ethoxycarbonylphenyl)-1,3-diene (IIc) was obtained by oxidation of 200 mg (0.62 mmol) of diarylacetylene **Ic** with 148 mg (0.62 mmol) of PbO_2 in 3 ml HF at –20°C within 3 h. Yield 21 mg (10%), oily substance. IR spectrum, ν, cm^{-1} (conformers mixture) 1710 (C=O), 1600 (C=C).

s-cis-Form. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.32 t (6H, 2Me, *J* 7.0 Hz), 1.37 t (6H, 2Me, *J* 7.1 Hz), 4.29 q (4H, $2CH_2$, *J* 7.0 Hz), 4.37 q (4H, $2CH_2$, *J* 7.1 Hz), 7.13–8.61 m ($16H_{arom}$).

s-trans-Form. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.33 t (6H, 2Me, *J* 7.0 Hz), 1.38 t (6H, 2Me, *J* 7.1 Hz), 4.30 q (4H, $2CH_2$, *J* 7.0 Hz), 4.38 q (4H, $2CH_2$, *J* 7.1 Hz), 7.13–8.61 m ($16H_{arom}$). Mass spectrum: *m/z* 682 $[M]^+$. Found, %: C 70.12; H 5.42. $C_{40}H_{36}F_2O_8$. Calculated, %: C 70.37; H 5.32. *M* 682.24.

(*Z,Z*)-1,2,3,4-Tetrakis(4-nitrophenyl)-1,4-difluorobuta-1,3-diene (IId) was obtained by oxidation of 200 mg (0.75 mmol) of diarylacetylene **Id** with 180 mg (0.75 mmol) of PbO_2 in 10 ml HF at –10°C within 3 h. Yield 45 mg (21%), mp 170–173°C. *s-cis*-Form. ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 7.56 d ($4H_{arom}$, *J* 8.7 Hz), 7.65 d ($4H_{arom}$, *J* 8.2 Hz), 8.00 d ($4H_{arom}$, *J* 8.7 Hz), 8.35 d ($4H_{arom}$, *J* 8.2 Hz). ^{19}F NMR spectrum [$(CD_3)_2CO$], δ, ppm: –91.54 s.

s-trans-Form. ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 7.42 d ($4H_{arom}$, *J* 8.7 Hz), 7.91 d ($4H_{arom}$, *J* 8.6 Hz), 8.10 d ($4H_{arom}$, *J* 8.7 Hz), 8.25 d ($4H_{arom}$, *J* 8.2 Hz). ^{19}F NMR spectrum [$(CD_3)_2CO$], δ, ppm: –145.95 s. Mass spectrum: *m/z* 574 $[M]^+$. Found, %: C 57.98; H 3.03; N 10.02. $C_{28}H_{16}F_2N_4O_8$. Calculated, %: C 58.54; H 2.81; N 9.75. *M* 574.09.

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