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2,2,2-Trichloro-4-methoxy-1,3,2-benzodioxaphosphole in the reactions with terminal acetylenes

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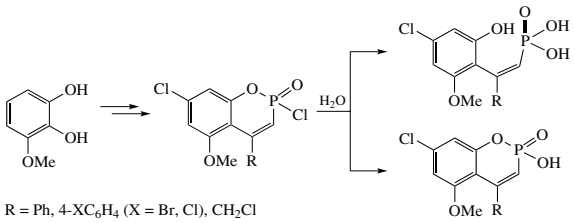
2,2,2-Trichloro-4-methoxy-1,3,2-benzodioxaphosphole reacts with arylacetylenes or 3-chloropropyne to give 2,7-dichloro-5-methoxy-4-aryl(haloalkyl)-1,2-benzoxaphosphinine 2-oxides. Hydrolysis of the latter leads to the opening of the oxaphosphinine ring and formation of (*E*)-2-(4-chloro-2-methoxy-6-hydroxyphenyl)ethenylphosphonic acid.



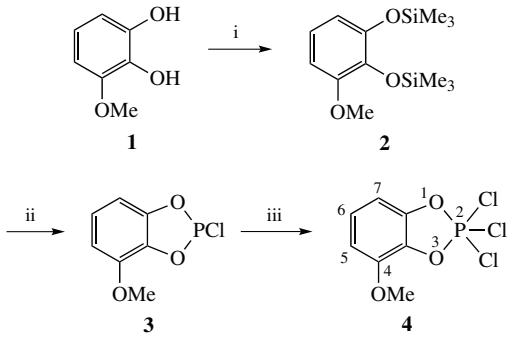
Keywords: dioxaphospholes, phosphoranes, arylacetylenes, cascade reactions, oxaphosphinines, ethenylphosphonic acids.

Polyoxyarenes are used in industry and laboratory practice[1] for the creation of functional materials[2] and biologically active compounds.[3] The presence of hydroxy and alkoxy groups provides a certain level of lipophilicity to natural substances.[4] Compounds bearing 1,2-dihydroxyarene (catechol) fragment are easily subjected to the phosphorylation with the formation of cyclic systems.[5] Stabilized trihalophosphoranes obtained from 1,2-dihydroxyarenes can be involved in the cascade reactions under the action of terminal acetylenes to form areno-1,2-oxaphosphophinines.[6] Earlier, we proposed convenient methods for the phosphorylation of trihydroxyarenes with P^{III} and P^{V} halides.[7]

As a result, we accessed functionalized derivatives containing phosphorus atoms in different valence-coordination states.[8] The reactions of phosphorylated pyrogallol and oxyhydroquinone derivatives with acetylenes are largely complicated by the disproportionation and ligand exchange processes involving the original phosphoranes. In the meantime, oxygen functional groups are convenient models for studying the effect of π-donor groups on the regiochemistry of the reaction of arenophospholes with acetylenes. The influence of acceptor and σ-donor substituents on the regiochemistry of the *ipso*-substitution of the oxygen atom in dioxaphospholene fragment and the halogenation of the latter in the course of reaction between dioxaphospholes and acetylenes were previously discussed for a wide range of substituted phospholes and acetylenes.[9] In this work we examined the influence of the π-donor methoxy group on the regiochemistry of the *ipso*-substitution of the oxygen atom and the halogenation of the benzo-1,2-oxaphosphinine fragment by the example of reaction between the phosphorylated 3-methoxycatechol derivative and acetylenes. Data on the phosphorylation of 3-methoxycatechol leading to oxazaspirophosphoranes and the effect of substituents on the intramolecular coordination are scarce.[10]

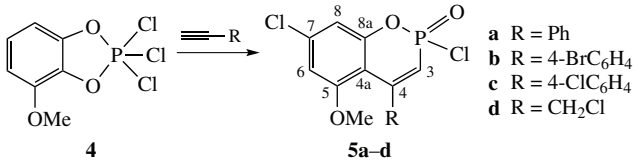
Phosphorylation of 3-methoxycatechol **1** was carried out *via* the preparation of intermediate disilyl ether **2** (Scheme 1). Reaction of compound **2** with phosphorus trichloride affords chlorophosphite **3** in quantitative yield.† Since ^{31}P NMR spectrum of the crude material contained one signal at 175.5 ppm, we subjected this material to further transformations without additional purification. To obtain chlorophosphorane **4**, an equimolar amount of phosphorus pentachloride was added to a solution of chlorophosphite **3** in dichloromethane. The completeness of the reaction was judged by the disappearance of the phosphorus pentachloride signal in the ^{31}P NMR spectrum. Phosphorane **4** is fairly stable and can be stored in an argon atmosphere without decomposition for a long time.

When analyzing the ^{13}C and ^{13}C-{^{1}H} NMR spectra of compounds **3** and **4**, one should note the dependence of the spin–spin coupling constants for the C^7 and C^4 atoms on the phosphorus atom coordination. Thus, there are the singlet and doublet ($^{3}J_{POCC^4}$ 2.3 Hz) in the ^{13}C-{^{1}H} NMR spectrum of $\sigma^3\lambda^3$-chlorophosphole **3** belonging to the C^7 and C^4 atoms, respectively. ^{13}C-{^{1}H} NMR spectrum of $\sigma^5\lambda^5$-trichlorophosphole **4** contains doublets having a larger value of the spin–spin coupling constant with phosphorus ($^{3}J_{POCC^4}$ = 19.4 Hz, $^{3}J_{POCC^7}$ = 17.6 Hz) that are related to the C^4



Scheme 1 *Reagents and conditions*: i, Me_3SiCl, NEt_3 (2 equiv. each), ii, PCl_3; iii, PCl_5, CH_2Cl_2.

† For the synthesis and characteristics of compounds obtained, see Online Supplementary Materials.



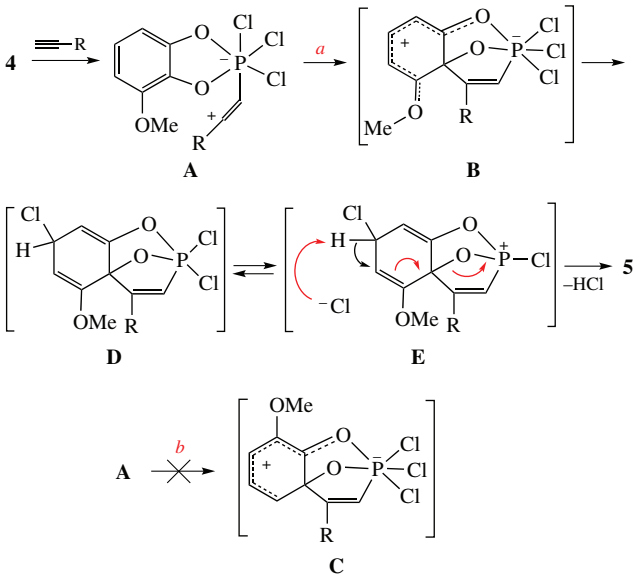
4 **5a–d**

Scheme 2

and C^7 atoms, respectively. This correlates with the data[11] that a lone electron pair of the P^{III} atom exhibits a strong shielding effect, which prevents the spin–spin coupling.

The reaction of phosphorane **4** with an excess of terminal acetylene proceeds under mild conditions with a sufficiently high rate and is accompanied by the changing in the color of the reaction mass from saturated yellow to pale brown.‡ Benzoxaphosphinines **5a–d** are the only organophosphorus products of the reaction (Scheme 2).

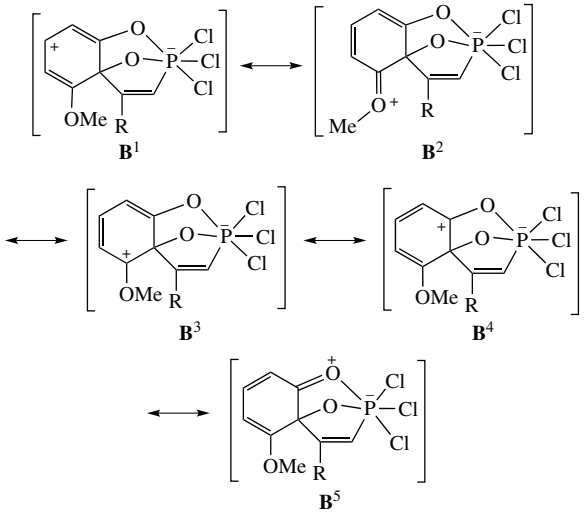
The ^{13}C, ^{1}H, ^{31}P NMR spectroscopic data allowed us to establish the regiochemistry of the processes of *ipso*-substitution of the oxygen atom and chlorination of the phenylene fragment in oxaphosphinines **5**. The regioselective migration of chlorine to the 7-position of the arenooxaphosphinine system occurred regardless of the nature of the acetylene and the substituent in



Scheme 3

‡ *Reaction of 2,2,2-trichloro-4-methoxy-1,3,2-benzodioxaphosphole* **4** *with acetylenes (general procedure).* To a solution of phosphorane **4** (12 mmol) in CH_2Cl_2 (10 ml), the corresponding acetylene (24 mmol) was added. The self-heating of the reaction mixture was observed. On the next day (in the case of arylacetylenes) or after 7 days (in the case of 3-chloropropyne) the mixture was evaporated *in vacuo* (15 Torr) to give 1,2-benzoxaphosphinine 2-oxides **5a–d** as glassy brown solids.

2,7-Dichloro-5-methoxy-4-phenyl-1,2-benzoxaphosphinine 2-oxide **5a** was prepared from phosphorane **4** (3.3 g, 12 mmol) and phenylacetylene (2.45 g, 2.2 ml, 24 mmol). ^1H NMR (400 MHz, $CDCl_3$), δ: 3.41 (s, 3H, OMe), 6.25 (d, 1H, C^3H, $^2J_{PCH}$ 26.4 Hz), 6.77 (d, 1H, C^8H, ^4J 1.3 Hz), 7.01 (d, 1H, C^6H, ^4J 1.3 Hz), 7.32–7.41 (m, 5H, Ph). ^{13}C NMR (100.6 MHz, $CDCl_3$), δ_C: 55.97 q (s) (OMe, $^1J_{HC}$ 145.6 Hz), 109.79 dd (s) (C^6, $^1J_{HC}$ 166.4 Hz, $^3J_{HC^8CC}$ 5.1 Hz), 110.83 m (d) (C^{4a}, $^3J_{PCCC}$ 17.9 Hz), 112.83 ddd (d) (C^8, $^1J_{HC}$ 172.5 Hz, $^3J_{POCC}$ 7.9 Hz, $^3J_{HC^6CC}$ 5.3 Hz), 115.49 dd (d) (C^3, $^1J_{HC}$ 172.1 Hz, $^1J_{PC}$ 157.5 Hz), 128.09 m (s) ($C^{11,13}$, $^1J_{HC}$ 161.1 Hz, $^3J_{HCCC}$ 6.8 Hz), 128.31 m (s) ($C^{10,14}$, $^1J_{HC}$ 161.3 Hz, $^3J_{HCCC}$ 7.2 Hz), 128.79 m (s) (C^{12}, $^1J_{HC}$ 161.3 Hz, $^3J_{HC^{11,14}CC}$ 7.2 Hz), 138.56 dd (s) (C^7, $^2J_{HC^8C}$5.1 Hz, $^2J_{HC^6C}$ 4.4 Hz), 140.42 m (d) (C^9, $^3J_{PCCC}$ 20.3 Hz, $^3J_{HCCC}$ 6.7 Hz, $^3J_{HCCC}$ 6.7 Hz), 151.87 dd (d) (C^{8a}, $^2J_{POC}$ 9.9 Hz, $^2J_{HC^8C}$ 4.3 Hz), 155.65 br. s (s) (C^4), 158.52 br. s (s) (C^5). $^{31}P/^{31}P$-{^1H} NMR (162 MHz, CH_2Cl_2), δ_P: 19.2 d (s) ($^2J_{PCH}$ 26.9 Hz).



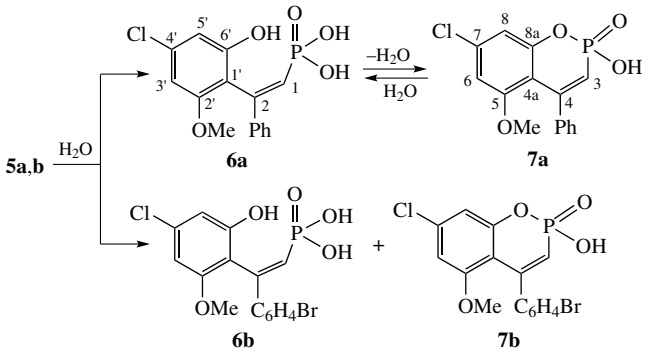
Scheme 4

aryl moiety. In addition, the *ipso*-substitution of the oxygen atom situated *ortho* to the methoxy group by terminal carbon atom of alkyne provides high regioselectivity of the process.

The results obtained can be accounted for the cascade process (Scheme 3). When phosphorane **4** reacts with terminal acetylene, σ-complex **A** is formed. A reorganization of the latter *via* the *ipso*-substitution of one of the oxygen atoms in the dioxaphosphole moiety is possible through pathways *a* and *b* with the formation of bicyclic intermediates **B** and **C**. In the case of intermediate **B** (Scheme 4), resonance forms $\mathbf{B}^1$–$\mathbf{B}^5$, including form $\mathbf{B}^2$ with a methoxy group, are involved in the delocalization of a positive charge. The latter form is energetically most preferable, which is the determining reason for the *ipso*-attack by a vinyl cation in the intramolecular pathway *a*. Further, a halogen attack is most likely to occur *meta* to the methoxy group (form $\mathbf{B}^1$), leading to bicyclic phosphorane **D**, which then transforms into final product **5** through the phosphonium intermediate **E**.

Since acid chlorides **5** are hydrolytically unstable, their hydrolysis was carried out for a more detailed characterization of the structure of the compounds obtained.§ In the presence of water, along with the cleavage of the P–Cl bond, a reversible opening of the oxaphosphinine cycle occurs, which depends on

§ *Hydrolysis of 2,7-dichloro-5-methoxy-4-phenyl-1,2-benzoxaphosphinine 2-oxide* **5a**. To a solution of compound **5a** in 1,4-dioxane (8 ml), water (3 ml) was added. On the next day, the resulting solution was evaporated *in vacuo* (15 Torr) to leave a dry residue. Benzene (10 ml) was added. The formation of (*E*)-2-(4-chloro-6-hydroxy-2-methoxyphenyl)-2-phenylethenylphosphonic acid **6a** as a light precipitate was observed. Product **6a** was filtered and dried *in vacuo* at 60 °C. Yield: 34% (1.39 g), mp 220 °C. ^1H NMR (400 MHz, DMSO-d_6), δ: 3.57 (s, OCH_2, dioxane), 3.60 (s, 3H, OMe), 6.34 (d, 1H, C^2H, $^2J_{PCH}$14.1 Hz), 6.52 (d, 1H, C^7H, ^4J 1.9 Hz), 6.53 (d, 1H, C^5H, ^4J 1.9 Hz), 7.25–7.31 (5H, Ph). ^{13}C NMR (100.6 MHz, DMSO-d_6), δ_C: 56.35 q (s) (OMe_3, $^1J_{HC}$ 144.5 Hz), 66.82 t (s) (OCH_2, dioxane, $^1J_{HC}$ 142.6 Hz), 103.37 dd (s) (C^7, $^1J_{HC}$ 166.0 Hz, $^3J_{HC^5CC}$ 5.0 Hz), 109.07 dd (s) (C^5, $^1J_{HC}$ 165.7 Hz, $^3J_{HC^7CC}$ 4.7 Hz), 114.83 dddd (d) (C^3, $^3J_{PCCC}$ 17.9 Hz, $^3J_{HC^1CC}$ 10.1 Hz, $^3J_{HC^5CC}$ 5.2 Hz, $^3J_{HC^7CC}$ 5.2 Hz), 121.66 dd (d) (C^1, $^1J_{PC}$ 187.1 Hz, $^1J_{HC}$ 149.2 Hz), 126.51 m (s) ($C^{11,13}$, $^1J_{HC}$ 159.2 Hz, $^3J_{HC^{13,11}CC}$ 7.1 Hz, $^2J_{HCC}$ 5.3 Hz), 128.76 m (s) ($C^{10,14}$, $^1J_{HC}$ 161.0–162.0 Hz), 128.76 m (s) (C^{12}, $^1J_{HC}$ 161.0–162.0 Hz), 133.10 dd (s) (C^6, $^2J_{HC^5C}$ 4.5 Hz, $^2J_{HC^7C}$ 4.5 Hz), 140.46 m (d) (C^9, $^3J_{PCCC}$ 21.6 Hz, $^3J_{HC^2CC}$ 14.2 Hz, $^3J_{HCCC}$ 6.7 Hz), 147.46 br. d (d) (C^8, $^4J_{PCCCC}$ 4.6 Hz), 156.46 br. s (s) (C^2), 158.95 br. s (s) (C^4). $^{31}P/^{31}P$-{^1H} NMR (162 MHz, DMSO-d_6), δ_P: 12.3 d (s) ($^2J_{PCH}$ 14.1 Hz). MS (MALDI-TOF), *m/z*: 341.00 (calc. for $C_{15}H_{14}ClO_5P$, *m/z*: 340.70). Found (%): C, 52.77; H, 3.33; Cl, 9.95; P, 8.78. Calc. for $C_{15}H_{14}ClO_5P$ (%): C, 52.88; H, 4.14; Cl, 10.41; P, 9.09.



Scheme 5

the nature of the *para*-substituent in the aryl fragment (Scheme 5). Thus, for 4-phenyl-substituted phosphinine **5a**, the hydrolysis is accompanied by a complete opening of the heterocycle to form vinyl phosphonate **6a**. Bromine-containing oxaphosphinine **5b** gives a mixture of acyclic (**6b**) and cyclic (**7b**) phosphonic acids under similar conditions. These compounds are easily distinguishable by the ^{31}P NMR spectra. Open-chain form **6b** manifests itself in low fields and has a smaller spin–spin coupling constant $^{2}J_{HP}$. The position of equilibrium significantly depends on the presence of water in the solvent. Gentle heating in dry solvents leads to the intramolecular dehydration and the formation of oxaphosphinines **7**, whereas the heterocycle can be re-opened on contact with excess water.

The structure of acid **6a** was confirmed by single crystal X-ray diffraction analysis (Figure 1).¶ Phosphonic acid **6a** crystallizes with half of a 1,4-dioxane molecule in the monoclinic space group $P2_1/c$. The $C^{9–14}$ phenyl ring is located approximately in the $P^1C^1C^2C^3C^9$ double bond plane, which contributes to the conjugation of the corresponding multiple bonds [the dihedral angle between the $C^{9–14}$ and $P^1C^1C^2C^3C^9$ planes is 5.6(1)°], while the $C^{3–8}$ phenyl substituent is noticeably deviated from this plane that makes conjugation between them impossible since the corresponding dihedral angle is 82.2(1)°. The main supramolecular motif is a two-dimensional bilayer oriented along the plane 0*bc*, which is formed *via* the ramified system of the intermolecular hydrogen bonds between the acid molecules: $O^1–H^1\cdots O^2$ [symmetry transformation: 1–*x*, *y*–1/2, 3/2–*z*; $O^1–H^1$ 0.76(3) Å, $O^1\cdots O^2$ 2.632(2) Å, $H^1\cdots O^2$ 1.88(3) Å, angle $O^1–H^1\cdots O^2$ 169(3)°] and $O^4–H^4\cdots O^2$ [symmetry transformation: 1–*x*, 1–*y*, 1–*z*; $O^4–H^4$ 0.84(3) Å, $O^4\cdots O^2$ 2.522(2) Å, $H^4\cdots O^2$ 1.69(3) Å, angle $O^4–H^4\cdots O^2$ 173(3)°]. 1,4-Dioxane is involved in the formation of the H-bond system also serving as an acceptor. Parameters of the H-bond $O^3–H^3\cdots O^{1s}$ are as follows: $O^3–H^3$ 0.80(3) Å, $O^3\cdots O^{1s}$ 2.592(3) Å, $H^3\cdots O^{1s}$ 1.81(3) Å, angle $O^3–H^3\cdots O^{1s}$ 165(3)°. Due to the dispersion interactions of peripheral fragments the bilayers are connected in the three-dimensional crystal structure with packing index of 65.8%.

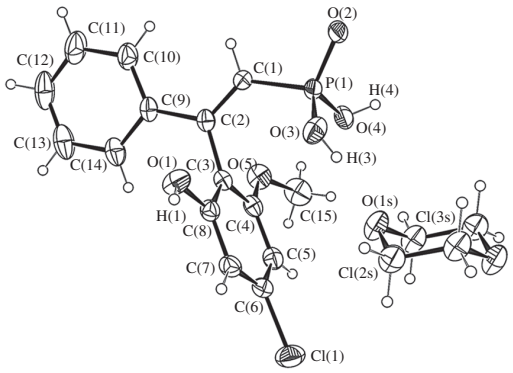


Figure 1 ORTEP diagram showing 30% probability anisotropic displacement ellipsoids of non-hydrogen atoms for a solvate of **6a** with 1,4-dioxane according to SC-XRD.

In summary, in 4-methoxy-2,2,2-trichloro-1,3,2-benzodioxaphosphole the effect of the π-donor methoxy group caused regioselective *ipso*-addition of terminal acetylenes at the carbon atom located *ortho* to the methoxy group in the dioxaphosphole fragment followed by *para*-chlorination of the arylene moiety. This transformation occurs regardless of the nature of the substituent in the acetylene molecule. The presence of the methoxy group in the arene moiety of oxaphosphinines makes the products hydrolytically instabile prone to easy opening of the heterocycle in aqueous solutions.

The synthetic part of this work was supported by the Russian Foundation for Basic Research (grant no. 19-03-00730). The spectral investigations of all synthesized compounds were supported by the Ministry of Education and Science of the Russian Federation, funded by a subsidy allocated to Kazan Federal University for the state assignment in the sphere of scientific activities (4.5151.2017/6.7). The authors gratefully acknowledge the CSF-SAC FRC KSC RAS for the diffraction data collection.

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.1016/j.mencom.2020.01.011.

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¶ *Crystal data for* **6a**. X-ray diffraction data for the single crystals of **6a** as a solvate with 1,4-dioxane were collected in an ω-scan mode on a Bruker SMART Apex II CCD diffractometer using graphite monochromated MoKα (0.71073 Å) radiation at 273(2) K. Data were corrected for absorption based on the Laue symmetry using equivalent reflections and for systematic errors. The structure was solved by the direct methods using SHELXT-2018/2[12] and refined by the full-matrix least-squares on F^2 using SHELXL-2018/3.[13] Non-hydrogen atoms were refined anisotropically. The positions of the hydrogen atoms H(1), H(3) and H(4) of the hydroxyl groups were determined by difference Fourier maps, and these atoms were refined isotropically. The other hydrogen atoms were inserted at the calculated positions and refined as riding atoms.

The crystals of **6a** were prepared by crystallization from a solution of 1,4-dioxane and water at room temperature. $C_{15}H_{14}ClO_5P\cdot0.5(C_4H_8O_2)$, M = 384.73, monoclinic, $P2_1/c$ (no. 14), a = 12.114(6), b = 9.461(5) and c = 16.426(8) Å, β = 106.176(7)°, V = 1808.1(16) Å^3, Z = 4, Z' = 1, d_{calc} = 1.413 g cm^{-3}, μ = 0.330 mm^{-1}, $F(000)$ = 800, $T_{max/min}$ = 0.6462/0.5784; 12774 reflections were collected (1.750° ⩽ θ ⩽ 25.249°; index ranges: –14 ⩽ h ⩽ 14, –11 ⩽ k ⩽ 11, –19 ⩽ l ⩽ 19), 3269 of which were unique, R_{int} = 0.0356, R_σ = 0.0312; completeness to θ of 25.242° 99.9%. The refinement of 239 parameters with no restraints converged to R_1 = 0.0411, wR_2 = 0.0978 for 2463 reflections with $I > 2\sigma(I)$ and R_1 = 0.0572, wR_2 = 0.1056 for all data with S = 1.049 and residual electron density, $\pi_{max/min}$ = 0.229 and –0.263 e Å^{-3}.

CCDC 1935982 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk.

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