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New Phosphorus-Containing Quinone Derivatives II: Tri- and Tetraphosphorylated Quinone Derivatives

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ABSTRACT: *A new synthetic approach to aromatic diols substituted with two, three, and four (similar or different) phosphorus groups via the Michael-type addition of the P—H bond containing reagents to p–benzoquinone derivatives, reoxidation, and subsequent addition of another phosphorus unit is presented. A variety of new phosphorus-containing p–hydroquinone derivatives were synthesized and fully characterized.* © 2013 Wiley Periodicals, Inc. Heteroatom Chem. 24:252–262, 2013; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.21089

INTRODUCTION

The reaction behavior of phosphorus reagents toward unsaturated systems has been thoroughly investigated throughout the past decades [1–4]. As such, a great variety of recent studies focus on the phospha-Michael reaction [4–11]. Specifically, addition reactions of phosphorus-containing compounds to quinones have been investigated thoroughly [12–14] as a result of the significance of quinone derivatives as dyes and biologically active compounds [15]. Recently, phosphorus-carrying hydroquinones also gained importance as flame-retardant additives or comonomers for polymers, especially epoxy resins and polyesters [16–23].

In general, the majority of phosphorus-containing compounds react with, i.e. *p*-benzoquinone via/via a Michael-type addition, resulting in *C*-phosphorylated *p*-hydroquinone derivatives, whereas some reagents also undergo *O*-phosphorylation [12]. Phosphine- and phosphine oxide-type compounds perform mono-*C*-addition in most cases, with the exception of primary phosphines (H_2P-R), which undergo a redox reaction with *p*–benzoquinone, resulting in *p*–hydroquinone and RPH-HPR [24]. Phosphite-type reagents perform mono- and di–*C*- addition as well as *O*-phosphorylation, depending on the conditions [12]. As a special case, the formation of tetraphosphorylated quinone derivates was reported for the reaction of chloranil with certain phosphites of secondary alcohols [25] whereas in the case of most phosphorus reagents chloranil undergoes

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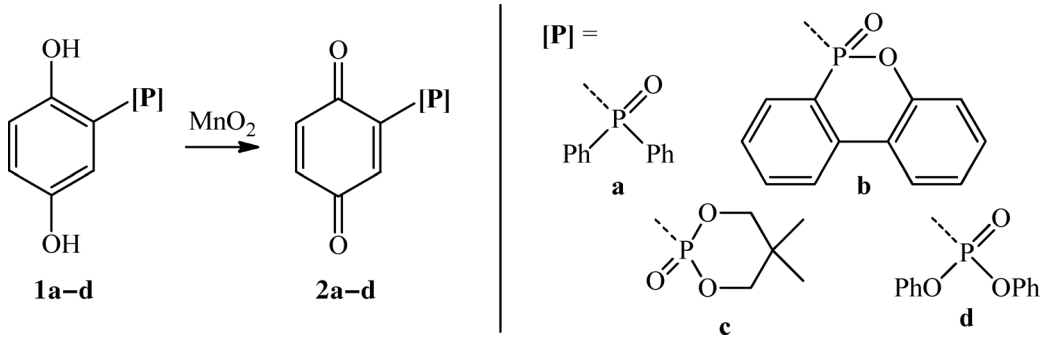


SCHEME 1 Oxidation of monosubstituted phosphorus-containing *p*-hydroquinone derivatives DPhPO-HQ (**1a**), DOPO-HQ (**1b**), DDPO-HQ (**1c**), and $(PhO)_2PO$-HQ (**1d**) with activated MnO_2. Compounds **2a, c, and d** were not isolated as original substances.

mono-*C*-phosphorylation [26] or *O*-phosphorylation [27]. For phosphate-type compounds, no reaction with *p*-benzoquinone takes place [13].

In summary, the existing work focuses on quinones carrying one, two, or in rare cases four similar phosphorus substituents.

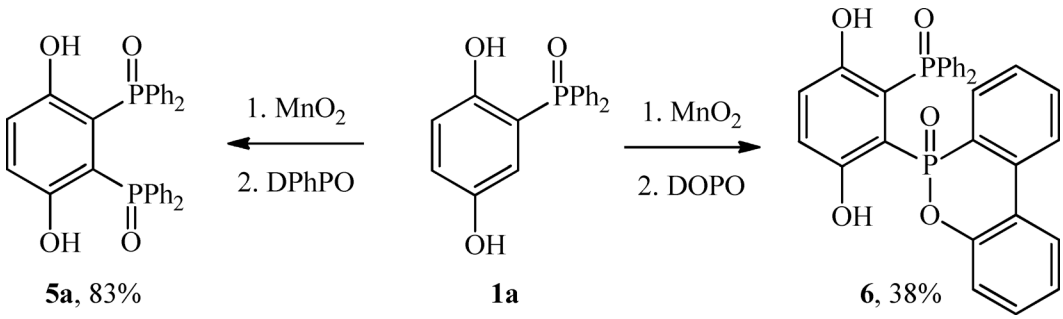
In our previous study, we presented a novel synthetic approach to twofold phosphorylated quinone derivatives that is not limited to identical P-units [28]. A phosphorus carrying *p*–benzoquinone derivative was synthesized by oxidation of DOPO-HQ, a common flame retardant [17, 18, 22, 23], which is easily synthesized via the Michael-type addition of *p*–benzoquinone and 6-oxido-6*H*-dibenzo[*c,e*][1,2]oxaphosphinine (DOPO; a widely used flame retardant by itself and building block for advanced flame retardants [29–32]). Treatment of DOPO-HQ with activated manganese dioxide, a quick and easy laboratory-scale procedure known to oxidize a large variety of hydroxy compounds to ketones [33], provided the correspondent compound DOPO-BQ in good yield and purity. Several new *p*–hydroquinone derivatives carrying two different phosphorus groups were synthesized by addition of a second [P]-H reactant to DOPO-BQ.

In this study, the concept described above was at first applied to further starting materials, as the addition products of *p*–benzoquinone with diphenylphosphine oxide (DPhPO) [20, 23, 34], diphenyl phosphite, and 5,5-dimethyl-2-oxido-1,3,2-dioxaphosphinan (DDPO; another phosphite-type reactant) [21, 35]. Subsequently, the obtained twofold phosphorylated quinone derivatives were further processed in the same manner to open a novel pathway to new tri- and tetraphosphorylated quinone derivatives that is not limited to identical P-units.

RESULTS AND DISCUSSION

The *p*-hydroquinone derivatives DPhPO-HQ (**1a**), DOPO-HQ (**1b**), DDPO-HQ (**1c**), and $(PhO)_2PO$-BQ (**1d**), monosubstituted with phosphorus groups of different chemical environments regarding the phosphorus atom, all readily underwent oxidation as indicated by the intense reddish colored solutions after a few minutes of treatment at room temperature (Scheme 1). As different methods of oxidation, **1b** was treated with aqueous H_2O_2 and with *tert*-butyl hydroperoxide in toluene [36], respectively. However, in both cases no reaction was observed after 24 h of treatment at room temperature.

While DOPO-BQ (**2b**) was obtained as a bright orange solid as shown in our previous work [28], DPhPO-BQ (**2a**), DDPO-BQ (**2c**), and $(PhO)_2PO$-BQ (**2d**) could not be isolated as original substances due to their higher solubility (attempts to precipitate them were not successful; also drying in melt could not be attempted due to the limited thermal stability of the *p*-benzoquinone derivates). Hence, secondary products of the latter were prepared by processing them in situ with a [P]-H reactant (Scheme 2). Treatment of DPhPO-HQ (**1a**) with activated MnO_2 and subsequent addition of diphenylphosphine oxide yielded DPhPO-2-HQ (**5a**). A similar synthesis for **5a** using Ag_2O as an oxidant is described in [37] but could not be reproduced. X-Ray analysis of **5a** (Fig. 1) revealed the addition of the second phosphorus group to take place in ortho position as was expected [28] (it should be noted that the para-substituted isomer of **5a** was recently described by Howell and Carter via lithium diisopropylamide catalyzed rearrangement of the correspondent *O*-phosphorylated *p*-hydroquinone derivative [38]). The synthesis of **5a** was carried out in three different solvents for comparison. While the properties of the product did not alter, the yield was



SCHEME 2 Preparation of DPhPO-2-HQ (**5a**) and DPhPO-HQ-DOPO (**6**) from DPhPO-HQ (**1a**).

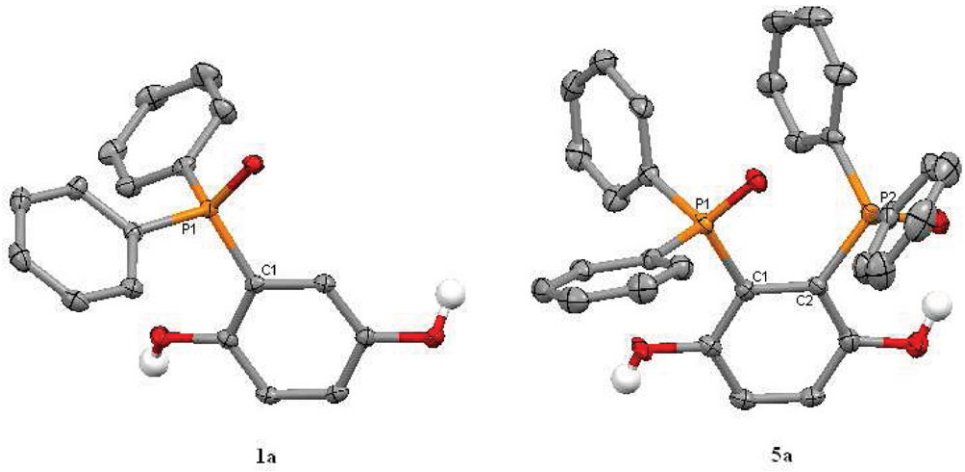
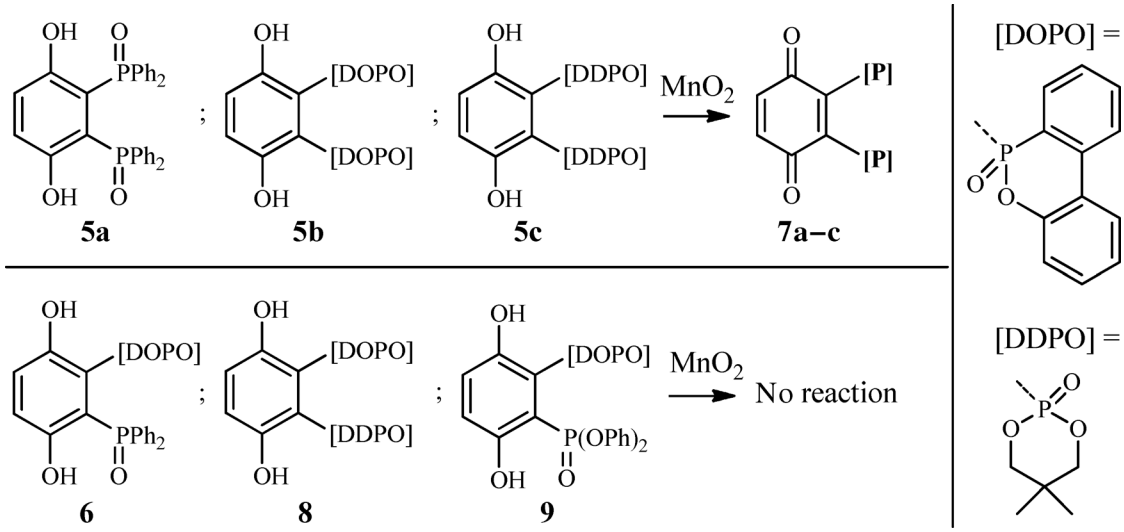


FIGURE 1 Structure of DPhPO-HQ (**1a**) and DPhPO-2-HQ (**5a**), validated by X-ray analysis (**5a** shown as one of two conformations. Hydrogens are omitted except on heteroatoms for reasons of clarity). In compound **5a**, one hydroxyl group forms in intramolecular hydrogen bond with the neighboring P═O unit, while 2 out of 4 phenyl units exhibit intramolecular π–stacking.

significantly different: Although only 31% yield was obtained processing the raw materials in toluene and 46% in chloroform, respectively, the reaction in 1,4-dioxane gave 83% yield. This can be attributed to the different polarities of the solvents and the solubility of the intermediate DPhPO-BQ (**2a**), respectively (vs. the absorption of the latter to the activated MnO_2). Furthermore, DPhPO-HQ-DOPO (**6**) was obtained exemplarily from DPhPO-HQ (**1a**) and DOPO (Scheme 2), as already previously synthesized vice versa from the isolated compound DOPO-BQ (**2b**) and diphenylphosphine oxide [28]. Using the in situ procedure, the yield of **6** was slightly lower (38%) as compared to the two-step procedure (45% starting from DOPO-HQ (**1b**)).

Compounds **5a** and a variety of other twofold phosphorylated *p*-hydroquinone derivates presented in our previous work were investigated regarding a new reoxidation with manganese dioxide (Scheme 3). A brisk reaction after few minutes of treatment with activated MnO_2 was exhibited by DPhPO-2-HQ (**5a**), DOPO-2-HQ (**5b**), and DDPO-2-HQ (**5c**). DDPO-2-BQ (**7c,** X-ray structure in Fig. 2) was obtained as a bright orange solid; the correspondent compounds **7b** and **7c** could not be isolated as original substances due to the aforementioned reasons. For DPhPO-HQ-DOPO (**6**), DOPO-HQ-DDPO (**8**), and DOPO-HQ-$(PhO)_2$PO (**9**), no reaction with manganese dioxide was observed, even after prolonged processing time (24 h at room temperature). In summary, in all cases studied, all derivatives carrying two similar phosphorus groups (regardless of their chemical environment) readily underwent reoxidation whereas all compounds with different phosphorus substituents did not. Besides, it is worth mentioning that the P—C bond in DDPO-2-BQ (**7c**) is significantly longer than in the non-oxidized form **5c** (Table 1).

Trifunctional phosphorus containing quinone derivatives was synthesized from DDPO-2–BQ (**7c**). The reaction of **7c** with diphenylphosphine oxide yielded DDPO-2-HQ-DPhPO (**10**; Scheme 4) as a 2,3,5-triphosphorylated *p*-hydroquinone derivative, as was expected (see X-ray structure in Fig. 2). For



SCHEME 3 Reaction behavior of several disubstituted phosphorus-containing *p*-hydroquinone derivates toward activated manganese dioxide. The synthesis of **5b**, **5c**, **6**, **8**, and **9** was presented in our previous work [28]. Compounds **7a** and **7b** were not isolated as original substances.

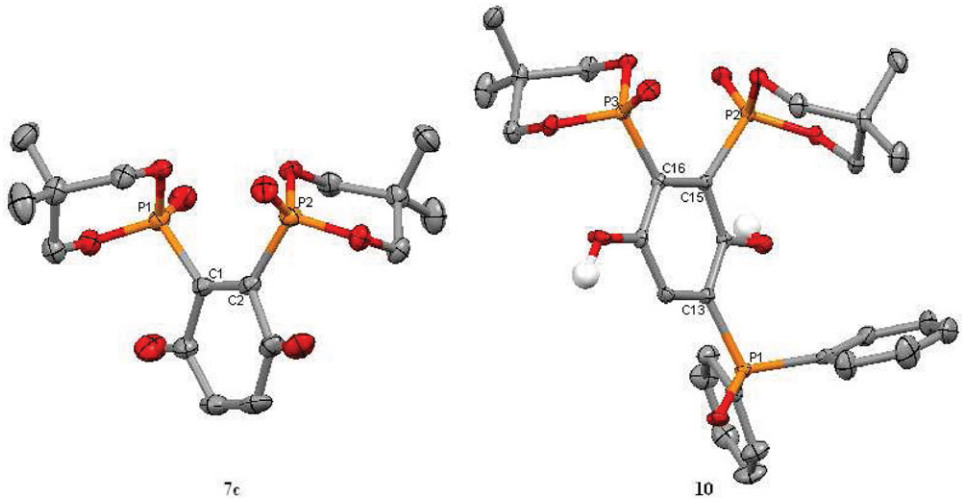


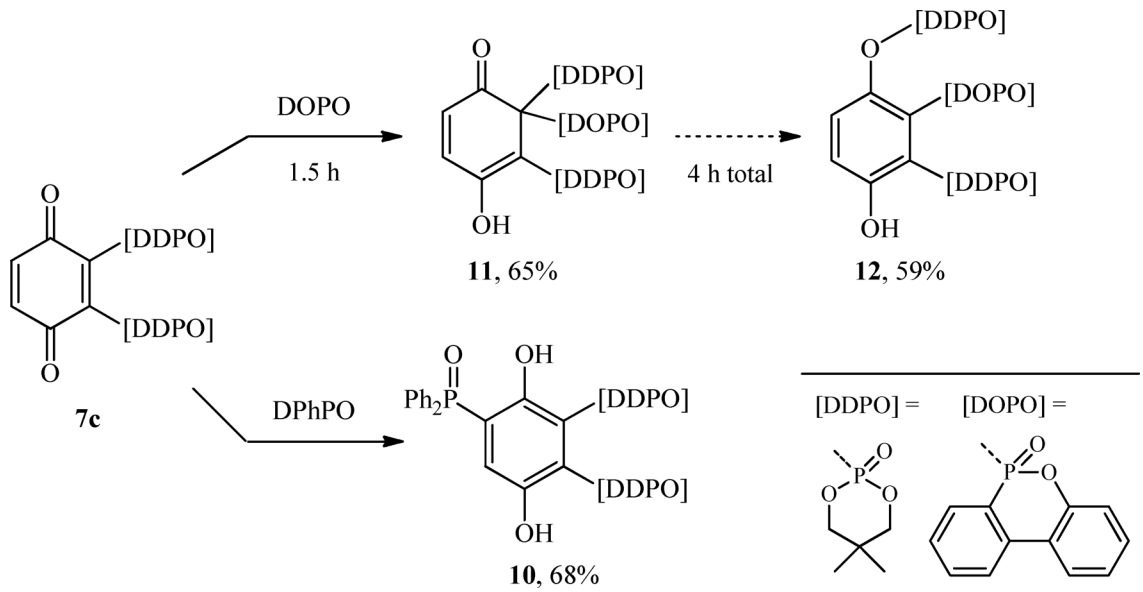
FIGURE 2 Structure of DDPO-2-BQ (**7c**) and DDPO-2-HQ-DPhPO (**10**), validated by X-ray analysis (hydrogens are omitted except on heteroatoms for reasons of clarity. **7c** shown as one of two conformations).

diphenyl phosphite, no similar product was formed due to the limited reactivity of phosphite-type reactants; prolonged processing led to decomposition (according to ^{31}P NMR). In the case of the phosphinate-type [P]-H-reactant, DOPO, an addition reaction with **7c** took place, however, exhibited a rather unexpected regiochemistry, yielding DDPO-2-HQ-DOPO (**11**) as a 2,2,3-triphosphorylated quinone derivative (Scheme 4). An explanation can be provided by consideration of the mesomeric structures for an *ortho*-diphosphorylated *p*-benzoquinone derivative. As shown in Scheme 5, a negative charge can be localized not only at the C=O units, but at the P=O units as well, resembled by an additional mesomeric structure with a localized positive charge at ortho-position. In the case of a monophosphorylated derivative, the nucleophilic addition of a second [P]-H unit therefore leads to 2,3-substituted derivatives with a high selectivity, as evident from our previous work [28]. In the case of an *ortho*-diphosphorylated *p*-benzoquinone derivative, however, a localization of a positive charge at the ortho position is much less favored due to the second P=O unit that is already deprived of electron density, especially in the case of a phosphite-type substituent as in DDPO-2-BQ (**7c**). Hence, in the reaction with a third [P]-H compound, the selectivity toward the 2,2,3- and the 2,3,5-triphosphorylated product is more dependent

TABLE 1 Lengths of P—C and P—O bonds between quinone and phosphorus units.

Compound	*P–C (DPhPO Unit)*	*P–C (DDPO Unit)*
DPhPO-HQ (**1a**)	1.801	–
DPhPO-2-HQ (**5a**)	1.814–1.825	–
DDPO-2-HQ (**5c**)[a]	–	1.804–1.805
DDPO-2-BQ (**7c**)	–	1.819–1.834
DDPO-2-HQ-DPhPO (**10**)	1.816	1.811–1.821
DDPO-2-HQ-DOPO (**11**)	[b]	1.811 (vicinal) 1.857 (geminal)
DDPO-2-HQ-DOPO-rear (**12**)[c]	[d]	1.81 (P–C) 1.601 (P–O)
DPhPO-3-HQ (**13**)	1.822–1.847 (vicinal) 1.808 (opposing site)	–
DPhPO-4-HQ (**15**)	1.823–1.858	–

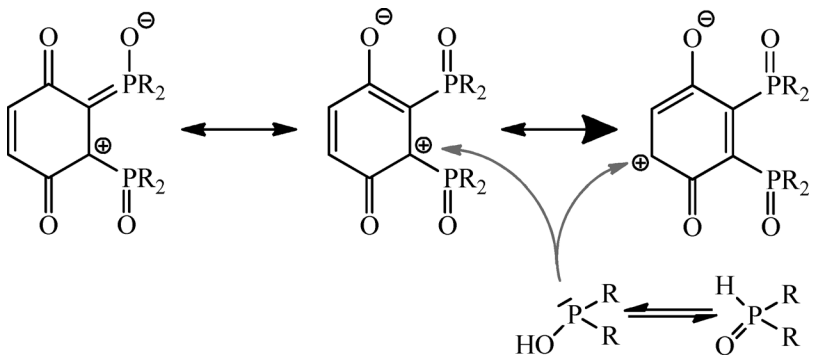
All values given in Å (standard deviation < 0.005).
[a]X-Ray analysis given in our previous work [28].
[b]P–C DOPO (geminal): 1.907.
[c]Standard deviation 0.01 is due to crystal quality.
[d]P–C DOPO (vicinal): 1.79.



SCHEME 4 Reaction of DDPO-2-BQ (**7c**) with DOPO and diphenylphosphine oxide.

on the [P]-H reactant and the stability of the potential products, respectively. Likewise, the solubility of the latter is of importance due to the reversibility of the phospha-Michael addition, influencing the equilibrium by precipitation of one component, as both **10** and **11** precipitated from the correspondent reaction mixture.

It should be noted that, regarding the C-4 position, **11** emerged distinctively in the enolic form as evident from ^{1}H NMR (singlet at 12.94 ppm, 1-OH), ^{13}C NMR (161.9 ppm, 1C–OH) and X-ray analysis (Fig. 3). Although a 1,4-diketo compound would be expected for reasons of keto-enol tautomerism, the enolic form is stabilized via an intramolecular hydrogen bond. As further expected, the P—C bond of the DDPO unit in the geminal position to the DOPO unit is significantly longer than the less sterically hindered one in the vicinal position (Table 1). Furthermore, prolonged processing of DDPO-2-BQ (**7c**) and DOPO led to a 1,3–rearrangement of one DDPO unit in **11**, yielding DDPO-2-HQ-DOPO-rear (**12**; Scheme 4 and X-ray structure shown in Fig. 3). It is worth mentioning that treatment of the first isolated intermediate **11** under same conditions (toluene, 4 h, 110°C) did not lead to rearrangement, and the latter was found to be unchanged. Treatment of **11** in the presence of a small amount of triethylamine led to formation of some **12**, but was accompanied by a large amount of decomposition products (as evident from ^{31}P NMR).



SCHEME 5 Mesomeric structures for [P]-2-BQ and nucleophilic attack of a third [P]-H reactant.

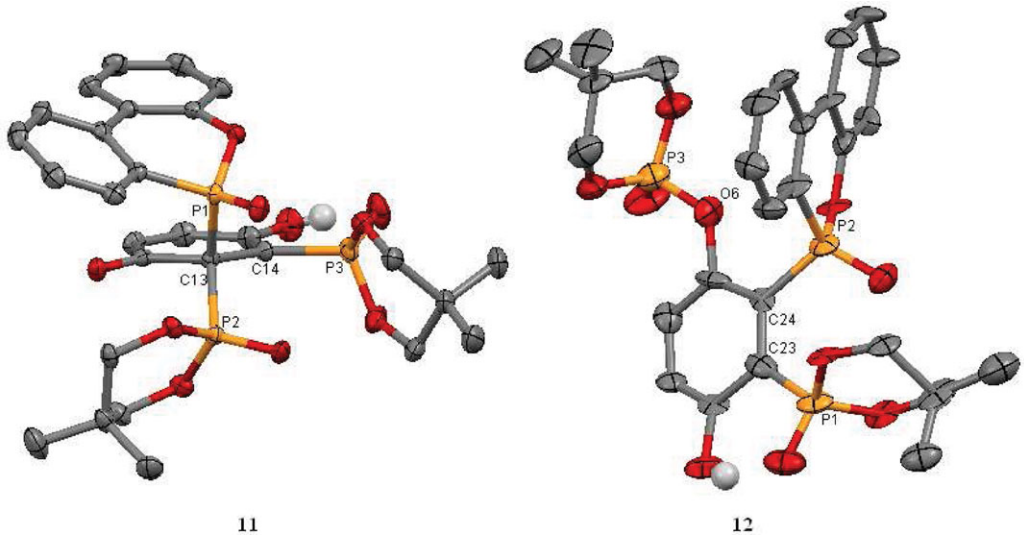
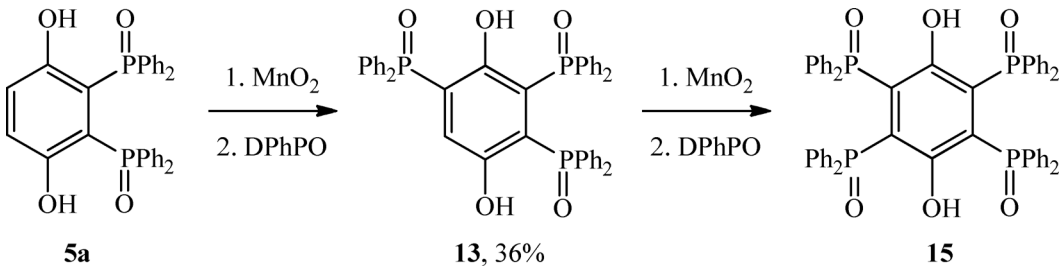


FIGURE 3 Structure of DDPO-2-HQ-DOPO (**11**) and DDPO-2-HQ-DOPO-rear (**12**), validated by X-ray analysis (hydrogens are omitted except on heteroatoms for reasons of clarity). In both cases, the hydroxyl group forms an intramolecular hydrogen bond. **11** exhibits intramolecular π-stacking.

Furthermore, DPhPO-3-HQ (**13**; Scheme 6, X-ray structure shown in Fig. 4) was synthesized from DPhPO–2–HQ (**5a**) and diphenylphosphine oxide in a similar manner as **5a**. While compound **13** was isolated via precipitation as a 2,3,5-triphosphorylated *p*-hydroquinone derivative exclusively, the relatively low yield of 36% indicates the formation of by-products.

Compound **13** readily underwent reoxidation with activated MnO_2. The resulting DPhPO-3-BQ (**14**) could again not be isolated as an original substance and was processed in situ with diphenylphosphine oxide, to synthesize DPhPO-4-HQ (**15**) as a 2,3,5,6–tetraphosphorylated *p*-hydroquinone derivative conclusively (Scheme 6). Although a mixture of products was obtained and DPhPO-4-HQ (**15**) could not be isolated as the original substance, the presence of **15** was confirmed by HRMS of the mixture (EI, calculated for $[^{12}C_{54}H_{42}O_6P_4]^+$ 910.1932, found 910.2025). The chemical shift of **15** in ^{31}P NMR was determined as 52.90 ppm (s) in $CDCl_3$, as evident from analysis of the mixture after complete removal of the solvent. Small crystals were obtained by recrystallization of the residue from ethyl acetate that allowed X-ray analysis of **15** (Fig. 4). The sole remaining by-product was identified as diphenylphosphinic acid Ph_2POOH by ^{31}P NMR. However, a complete separation of the latter and compound **15** was not possible due to the formation of a charge-transfer complex, as indicated by the fluorescent yellowish color of the solution or the residue, respectively.

The average P—C bond lengths between the quinone moiety and the phosphorus units in DPhPO-HQ (**1a**), DPhPO-2-HQ (**5a**), DPhPO-3-HQ (**13**), and DPhPO-4-HQ (**15**) increased with the number of phosphorus substituents, as was expected due to steric hindrance. Also, in DPhPO-3-HQ (**13**), the two



SCHEME 6 Synthesis of DPhPO-3-HQ (**13**) and DPhPO-4-HQ (**15**). Compound **15** was not isolated as the original substance.

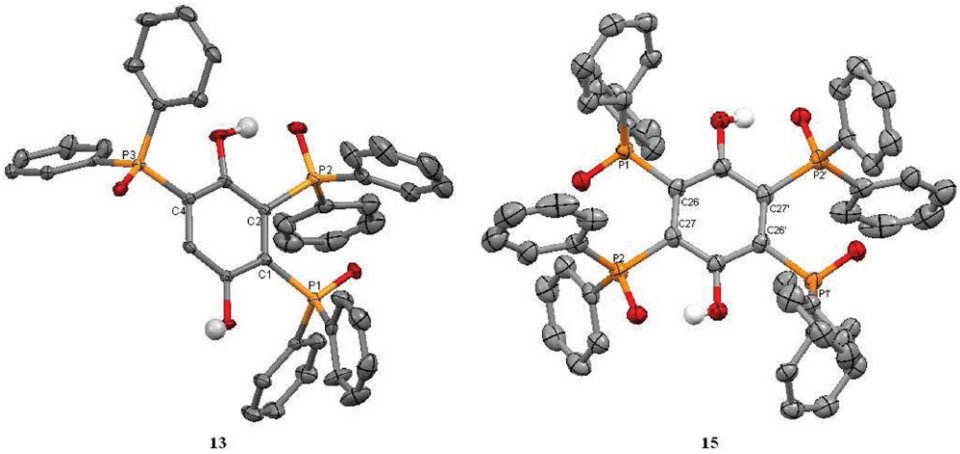


FIGURE 4 Structure of DPhPO-3-HQ (**13**) and DPhPO-4-HQ (**15**), validated by X-ray analysis (hydrogens are omitted except on heteroatoms for reasons of clarity). In **15**, both hydroxyl groups form an intramolecular hydrogen bond, whereas in **13** only the enclosed one does. For both compounds, no intramolecular π-stacking is evident.

vicinal P—C bonds were significantly longer than the opposing one (Table 1). In addition, X–ray analysis revealed the planarity of the *p*-hydroquinone unit in DPhPO-4-HQ (**15**) to be undeformed despite the presence of four bulky substituents, in consistence with the results reported for *p*-hydroquinone 2,3,5,6-tetrakis(diisopropyl phosphonate) [39].

Also, all *p*-benzoquinone derivatives not isolated as original substances (**2a**, **2c**, **2d**, **7a**, **7b**, and **14**) were characterized in situ via HRMS and ^{31}P NMR of their concentrated solutions (Table 2).

It should be noted that most of the presented derivatives emerge as a mixture of different diastereomers due to the bulkiness of the phosphorus substituents in sterically hindered positions and the chirality regarding the phosphorus atom in DOPO moieties, given that the formation of different diastereomers is a known effect for compounds carrying two or more DOPO substituents and was well described, i.e., in [40]. As evidence, the correspondent signals in ^{31}P NMR (measured proton-decoupled) appeared as two separated peaks, exhibiting a small difference in chemical shift. The phenomenon was observed in the case of the derivatives **5b** and **8–12** with a deviation in chemical shift of about 0.2 ppm and a ratio of always about 1:1, with the exception of **5b,** where the effect was most distinct with 1.15 ppm deviation and a ratio of 1:1.5, at which the two isomers were successfully separated as shown in our previous work [28]. For the derivatives **8–12**, no separation of isomers was attempted.

CONCLUSIONS

A new synthetic approach to di-, tri-, and tetrafunctional phosphorus-containing quinones and a variety of corresponding derivatives was presented. In contrast to previous methods, the new approach is not limited to similar phosphorus units and, therefore, offers a convenient pathway to tailor-made aromatic diols regarding the number and kind of phosphorus substituents for multiple purposes, for instance as comonomers, dyes, or ligands.

EXPERIMENTAL

Materials and Measurements

Unless stated otherwise, solvents and chemicals were obtained from commercial sources and used

TABLE 2 Chemical Shift in ^{31}P NMR in $CDCl_3$ and HRMS (EI) for Phosphorylated *p*-Benzoquinone Derivatives Not Isolated as Original Substances **2a, 2c, 2d**, **7a, 7b**. and **14**.

Compound	*^{31}P NMR ($CDCl_3$)*	*HRMS (EI)*
DPhPO-BQ (**2a**)	43.84 ppm (s)	calcd. $[^{12}C_{18}H_{13}O_3P]^+$ 308.0602, found 308.0563
DDPO-BQ (**2c**)	1.85 ppm (s)	calcd. $[^{12}C_{11}H_{13}O_5P]^+$ 256.0501, found 256.0507
$(PhO)_2PO$-BQ (**2d**)	1.82 ppm (s)	calcd. $[^{12}C_{18}H_{13}O_5P]^+$ 340.0501, found 340.0482
DPhPO-2-BQ (**7a**)	28.32 ppm (s)	calcd. $[^{12}C_{30}H_{22}O_4P_2]^+$ 508.0993, found 508.0952
DOPO-2-BQ (**7b**)	15.47 ppm (s)[a]	calcd. $[^{12}C_{30}H_{18}O_6P_2]^+$ 536.0579, found 536.0663
DPhPO-3-BQ (**14**)	28.15 ppm (s, 1 P), 27.76 ppm (s, 1 P), 21.81 ppm (s, 1 P)	calcd. $[^{12}C_{42}H_{31}O_5P_3]^+$ 708.1384, found 708.1339

[a]DOPO-2-HQ (**7b)** was processed as a mixture of both diastereomers (see [28]). However, only the less stable isomer underwent reoxidation within 30 min, exhibiting roughly 50% conversion as evident from ^{31}P NMR.

as received. DDPO was synthesized from neopentyl glycol and dimethyl phosphite according to the literature method [41]. Compounds **1c**, **1d**, **5b**, **5c**, **8**, and **9** were synthesized as shown in our previous work [28].

NMR spectra were recorded with a Bruker (Bellerica, MA) Analytical BZH 250/52 (250 MHz) and a Varian (Palo Alto, CA) Inova-400 (400 MHz). Chemical shifts are given as δ values with internal standard via a solvent. ^{31}P NMR spectra were measured proton decoupled, ^{13}C NMR proton decoupled and phosphorus coupled, and ^{1}H NMR phosphorus coupled. IR spectra were recorded with a Varian 660-IR (FT-IR). Melting points were measured with a Büchi (Flawil, Switzerland) B-545 (uncorrected). High-resolution mass spectra were obtained with a MicroMass (Beverly, MA) GCT (EI, 70 eV). Elementary analysis (CHNS) was performed with an Elementar (Hanau, Germany) VarioEL.

Crystallographic Data

Single crystal X-ray diffraction data were collected on a STOE IPDS2T diffractometer with graphite monochromated Mo Kα radiation (0.71073 Å) equipped with an Oxford Cryostream unit for low-temperature measurements ($T = 180$ K). Structure solution and refinement against F^2 were carried out using SHELXS and SHELXL software [42]. Refinement was performed with anisotropic temperature factors for all non-hydrogen atoms (disordered atoms were refined isotropically); hydrogen atoms were calculated on idealized positions. The crystallographic data of **1a**, **5a**, **7c, 10–13**, and **15** are summarized in Table 3 (continued) in the Supporting Information. Visualization for evaluation was performed with XPMA [43], and figures were created with Mercury [44].

CCDC numbers 905545–905552 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Synthesis

(2,5-Dihydroxyphenyl)diphenylphosphine Oxide (DPhPO-HQ, ***1a****).* Diphenylphosphine oxide (101.1 g, 0.50 mol) was dissolved in 300 mL of toluene in a three-necked flask equipped with a condenser, argon inlet, thermometer, and magnetic stirrer. *p*-Benzoquinone (54.1 g, 0.50 mol) was added in small portions at 75°C, and the mixture was refluxed at 110°C for 1 h. After cooling down, the product was filtered off at reduced pressure, washed with acetone, and air-dried. Light brown solid, yield: 132.94 g (86%). mp: 214°C. ^{31}P NMR (101 MHz, DMSO-d_6, ppm): 28.30 (s). ^{13}C NMR (63 MHz, DMSO-d_6, ppm): 152.5 (d, $J = 2.8$ Hz, 1 C-OH); 150.2 (d, $J = 14.2$ Hz, 1 C-OH); 133.5 (d, $J = 105.3$ Hz, 2C-P); 132.1 (s, 2C); 131.7 (d, $J = 10.2$ Hz, 4 C); 128.7 (d, $J = 12.1$ Hz, 4C); 121.8 (s, 1C); 118.9 (d, $J = 8.1$ Hz, 1C); 117.9 (d, $J = 8.6$ Hz, 1C); 116.4 (d, $J = 102.5$ Hz, 1C-P). ^{1}H NMR (250 MHz, DMSO-d_6, ppm): 9.75 (s, 1 H, OH); 9.10 (s, 1 H, OH); 7.70–7.45 (m, 10 H, —$P(O)Ph_2$); 6.94 (dd, $J = 13.92$ Hz, $J = 2.75$ Hz, 1H, C—H); 6.85 (dd, $J = 8.64$ Hz, $J = 2.63$ Hz, 1H, C—H); 6.70 (dd, $J = 8.52$ Hz, $J = 6.12$ Hz, 1H, C—H). IR (KBr) ν 3142 (br, O—H); 1606 (w); 1590 (w); 1512 (w) 1486 (w); 1431 (vs, P—Ph); 1359 (w); 1252 (m); 1230 (m); 1206 (m); 1130 (vs, P=O); 1088 (s); 1053 (s); 1027 (w); 998 (w); 875 (w); 826 (m); 752 (m); 734 (s); 716 (m); 691 (s); 584 (w); 569 (m); 534 (m); 520 (s); 489 (w); 450 (w). HRMS (EI) calcd. $[^{12}C_{18}H_{15}O_3P]^+$ 310.0759; found 310.0768. Anal calcd. for: $C_{18}H_{15}O_3P$: C 69.68, H 4.87; found: C 69.49, H 4.98.

(3,6-Dihydroxy-1, 2-phenylene)bis(diphenylphosphine Oxide) (DPhPO-2-HQ, ***5a****).* DPhPO–HQ (**1a**, 6.21 g, 20 mmol) and activated manganese dioxide (25.0 g) were suspended in 1,4-dioxane and vigorously stirred at room temperature for 1 h.

The mixture was filtered off, and the residue thoroughly washed with 1,4-dioxane. The combined liquid phases were filtered again using particularly fine-pored paper to separate remaining traces of MnO_2. Diphenylphosphine oxide (4.04 g, 20 mmol) was added, and the mixture was heated to 90°C for 2 h until the reddish color faded. The mixture was concentrated under reduced pressure. The product was collected by filtration at reduced pressure, washed with 1,4-dioxane, and dried at 0.2 mbar. White solid, yield: 8.48 g (83%). mp: 217–218°C. ^{31}P NMR (101 MHz, TFA-*d*, ppm): 56.28 (s). ^{13}C NMR (63 MHz, TFA-*d*, ppm): 155.9 (d, J = 16.8 Hz, 2C—OH); 135.1 (s, $4C_{Ph}$); 132.8 (d, J = 11.9 Hz, $8C_{Ph}$); 129.6 (d, J = 14.2 Hz, $8C_{Ph}$); 126.7 (d, J = 5.9, 2C—H); 126.2 (d, J = 114.9 Hz, 4P—C_{Ph}); 115.6 (dd, J = 104.9 Hz, J = 5.4 Hz, 2P—C). ^{1}H NMR (250 MHz, TFA-*d*, ppm): 8.94–8.78 (m, 12H, arom.); 8.72–8.62 (m, 8H, arom.); 8.34 (t, J = 3.11 Hz, 2H, C—H quinone). IR (KBr) ν 3058 (C—H aryl.); 1573 (w); 1461 (s); 1437 (s, P—Ph); 1267 (s); 1207 (w); 1151 (vs, P=O); 1120 (m); 1087 (m); 1062 (m); 932 (w); 833 (w); 731 (m); 702 (m); 689 (s); 588 (s); 573 (m); 548 (w); 527 (s); 503 (w). HRMS (EI) calcd. $[^{12}C_{30}H_{24}O_4P_2]^+$ 510.1150, found 510.1157. Anal calcd. for $C_{30}H_{24}O_4P_2$: C 70.59, H 4.74; found: C 70.20, H 4.86.

2,3-Bis (5,5-dimethyl-2-oxido-1,3,2-dioxaphosphinan-2-yl)cyclohexa-2,5-diene-1,4-dione (DDPO-2-BQ, ***7c****).* DDPO-2-HQ (**5c**, 4.06 g, 10 mmol) and activated manganese dioxide (32.0 g) were suspended in acetone and vigorously stirred at room temperature for 1 h. The mixture was filtered off, and the residue *thoroughly* washed with acetone. The solution was dried over $MgSO_4$ and filtered off. The product was obtained by complete removal of the solvent at reduced pressure and dried at 0.2 mbar. Bright orange solid, yield: 2.26 g (56%). mp: decomp. >190°C. ^{31}P NMR (101 MHz, $CDCl_3$, ppm): –1.71 (s). ^{13}C NMR (63 MHz, $CDCl_3$, ppm): 184.3 (t, J = 11.6 Hz, 2C=O); 144.2 (d, J = 167.8 Hz, 2C—P); 136.8 (t, J = 4.5 Hz, 2C, —CH=CH—); 78.1 (t, J = 3.8 Hz, 4—O—CH_2—); 32.0 (t, J = 3.4 Hz, $2C_{quart}$); 21.7 (s, 2—CH_3 ax.); 20.3 (s, 2—CH_3, equ.). ^{1}H NMR (250 MHz, $CDCl_3$, ppm): 6.92 (t, J = 3.11 Hz, 2H, —HC=CH—); 4.05–3.96 (m, 8H, —CH_2—O—); 1.31 (s, 6H, —CH_3 ax.); 0.83 (s, 6H, —CH_3 equ.). IR (KBr) ν 2974 (w, C—H alkyl); 2883 (w, C—H alkyl); 1657 (s, C=O); 1479 (m, P—C); 1290 (vs, P=O); 1060 (vs, P—OC_{alkyl}); 1007 (s, P—OC_{alkyl}); 986 (m); 864 (m); 778 (m); 488 (s). HRMS (EI) calcd. $[^{12}C_{16}H_{22}O_8P_2]^+$ 404.0790, found 404.0869. Anal calcd. for $C_{16}H_{22}O_8P_2$: C 47.53, H 5.48; found: C 47.59, H 5.69.

2,2′-(4-(Diphenylphosphoryl)-3,6-dihydroxy-1,2-phenylene)bis(5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide), (DDPO-2-HQ-DPhPO, ***10****).* In a flame-dried flask equipped with a condenser and an argon inlet, DDPO-2-BQ (**7c**; 404 mg, 1.0 mmol) and diphenylphosphine oxide (202 mg, 1.0 mmol) were refluxed in 20 mL of dry toluene for 1 h until the orange color of the mixture faded. The mixture was cooled with an ice bath for 30 min. The product was collected by filtration at reduced pressure, washed with cold toluene, and dried at 0.2 mbar. White solid, yield: 410 mg (68%). mp: 178°C. ^{31}P NMR (101 MHz, $CDCl_3$, ppm): 37.46 (s, 1 P DPhPO); 9.82 + 9.66 (isomers, 1P DDPO); 7.67 + 7.59 (isomers, 1P DDPO). ^{13}C NMR (101 MHz, $CDCl_3$, ppm): 158.3 (d, J = 17.2 Hz, $1C_{arom}$—OH); 154.6 (td, J = 15.8 Hz, J = 4.8 Hz, $1C_{arom}$—OH); 133.2 (d, J = 2.6 Hz, $2C_{Ph}$); 132.3 (d, J = 10.7 Hz, $4C_{Ph}$); 130.1 (d, J = 107.5 Hz, $2C_{Ph}$—P); 129.1 (d, J = 12.8 Hz, $4C_{Ph}$); 128.4 (t, J = 10.2 Hz, 1C); 122.0 (dd, J = 99.8 Hz, J = 9.6 Hz, 1C—P); 119.2 (dd, J = 182.9 Hz, J = 10.8 Hz, 1 C—P); 116.6 (dt, J_d = 178.6 Hz, J_t = 9.2 Hz, 1 C—P); 79.1 (d, J = 7.1 Hz, 2 —CH_2—O—) 78.5 (d, J = 7.0 Hz, 2 —CH_2—O—); 33.0 (d, 10.0 Hz, $1C_{quart}$); 32.7 (d, J = 8.4 Hz, $1C_{quart}$); 23.3 (s, 1 —CH_3 ax.); 23.1 (s, 1 —CH_3 ax.); 21.3 (s, 1 —CH_3 equ.); 21.2 (s, 1 —CH_3 equ.). ^{1}H NMR (250 MHz, $CDCl_3$, ppm): 11.48 (s, 1 H, Ar—OH); 10.43 (s, 1 H, Ar—OH); 7.71 (dd, J = 12.7 Hz, J = 7.3 Hz, 4 H, arom.); 7.61 (d, J = 7.1 Hz, 2H, arom.); 7.53 (dd, J = 7.2 Hz, J = 2.7 Hz, 4H, arom.); 7.19 (m, 1H, C—H quinone unit); 4.34 (dd, J = 41.3 Hz, J = 10.4 Hz, 4H, —CH_2—O—); 4.00 (dd, J = 10.5 Hz, J = 4.9 Hz, 2H, —CH_2—O—); 3.92 (t, J = 8.9 Hz, 2 H, —CH_2—O—); 1.44 (s, 3H, —CH_3 ax.); 1.40 (s, 3 H, —CH_3 ax.); 0.82 (s, 6H, CH_3 equ.). IR (KBr) ν 3061 (w, C—H aryl); 2965 (w, C—H alkyl); 2884 (w, C—H alkyl); 1580 (w); 1474 (w); 1439 (m; P—Ph); 1359 (m); 1284 (s, P=O); 1234 (s, P=O); 1208 (m); 1148 (m); 1122 (m); 1061 (vs, P—OC_{alkyl}); 1009 (s, P—OC_{alkyl}); 943 (w); 812 (s); 793 (m); 753 (w); 728 (w); 697 (m); 651 (m); 582 (s); 564 (m); 507 (s); 474 (w). HRMS (EI) calcd. $[^{12}C_{28}H_{33}O_9P_3]^+$ 606.1337, found 606.1343. Anal calcd. for $C_{28}H_{33}O_9P_3$: C 55.45, H 5.48; found: C 55.45, H 5.85.

5,6-Bis(5,5-dimethyl-2-oxido-1,3,2-dioxaphosphinan-2-yl)-4-hydroxy-6-(6-oxido-6H-dibenzo[c,e][1,2]oxaphosphinin-6-yl)cyclohexa-2,4-dienone (DDPO-2-HQ-DOPO, ***11****).* In a flame-dried flask equipped with a condenser and an argon inlet, DDPO-2-BQ (**7c**; 404 mg, 1.0 mmol) and DOPO (216 mg, 1.0 mmol) were refluxed in 20 mL of dry toluene for 1.5 h, not longer. After cooling down, the product was collected by filtration at reduced

pressure, washed with toluene, and dried at 0.2 mbar. Yellowish solid, yield: 403 mg (65%). mp: 197°C. ^{31}P NMR (101 MHz, $CDCl_3$, ppm): 32.41 + 32.35 (isomers, 1P DOPO); 13.10 (s, 1P DDPO vicinal), 1.70 + 1.64 + 1.59 (isomers, 1P DDPO geminal). ^{13}C NMR (63 MHz, $CDCl_3$, ppm): 188.1 (m, 1C═O); 161.9 (m, 1C—OH); 147.1 (d, J = 8.6 Hz, 1C); 140.4 (dt, J_d = 18.3 Hz; J_t = 3.0 Hz, 1C); 135.9 (d, J = 6.9 Hz, 1C_{quart}); 133.8 (d, J = 2.5 Hz, 1C); 132.9 (d, J = 8.7 Hz, 1C); 130.8 (s, 1C); 130.0 (d, J = 2.8 Hz, 1C); 128.5 (d, J = 50.9 Hz, 1C—P); 128.4 (d, J = 14.3 Hz, 1C); 125.5 (s, 1C); 125.2 (s, 1C); 124.8 (d, J = 42.6 Hz, 1C—P); 123.8 (d, J = 12.1 Hz, 1C_{quart}); 123.5 (d, J = 11.0 Hz, 1C); 122.5 (d, J = 6.5 Hz, 1C); 97.7 (dt, J_d = 200.4 Hz, J_t = 7.8 Hz, 1 P—C—P); 80.2 (d, J = 6.4 Hz, 1 —CH_2—); 79.9 (d, J = 8.4 Hz, 1 —CH_2—); 79.5 (d, J = 8.5 Hz, 1 —CH_2—); 79.1 (d, J = 7.3 Hz, 1 —CH_2—); 33.3 (d, J = 13.3 Hz, 1C_{quart}); 32.9 (d, J = 11.6 Hz, 1C_{quart}); 23.8 (s, 1 —CH_3 ax.); 22.7 (s, 1 —CH_3 ax.); 21.2 (s, 1 —CH_3 equ.); 20.4 (s, 1 —CH_3 equ.). ^{1}H NMR (250 MHz, $CDCl_3$, ppm): 12.94 (s, 1 H, —OH); 8.16 (dd, J = 13.40 Hz, J = 7.48 Hz, 1H, arom.); 7.72 (s, 2H, arom.); 7.59 (d, J = 7.15 Hz, 2H, arom.); 7.29 (s, 1H, arom.); 7.26 (t, J = 16.20 Hz, 2H, arom.); 7.17 (m, 1H, arom.); 6.39 (m, 1H, —CH—); 5.50 (d, J = 9.69, 1H, —CH═CH—); 5.33 (d, J = 9.83, 1H, —CH═CH—); 5.02 (d, J = 6.52 Hz, 1H, —CH_2—O—);4.45 (d, J = 9.32 Hz, —CH_2—O—); 4.10 (t, J = 14.80 Hz, 2H, —CH_2—O—); 3.99 (t, J = 11.36 Hz, 2H, —CH_2—O—); 3.78 (t, 1H, —CH_2—O—); 3.59 (d, J = 9.37 Hz, 1H, —CH_2—O—); 1.56 (s, 3H, —CH_3 ax.); 1.35 (s, 3H, —CH_3 ax.); 0.92 (s, 3H, —CH_3 equ.); 0.74 (s, 3H, —CH_3 equ.). IR (KBr) ν 3070 (w, C—H aryl); 2960 (w, C—H alkyl); 2932 (w, C—H alkyl); 1658 (m); 1638 (m); 1546 (m); 1475 (m); 1430 (m); 1283 (s, P═O); 1238 (m, P═O); 1199 (w); 1147 (m, P—OC_{aryl}); 1116 (w); 1071 (s, P—OC_{alkyl}); 1065 (s, P—OC_{alkyl}); 1032 (w); 1013 (m); 989 (w); 939 (w); 909 (m); 887 (w); 852 (m); 790 (w); 768 (m); 625 (m); 529 (m); 511 (m); 497 (m). HRMS (EI) calcd. $[^{12}C_{28}H_{31}O_{10}P_3]^+$ 620.1130, found 620.1190. Anal calcd. for $C_{28}H_{31}O_{10}P_3$: C 54.20, H 5.04; found: C 53.97, H 5.03.

2,2′-(3-Hydroxy-6-((6-oxido-6H-dibenzo[c,e][1,2]oxaphosphinin-6-yl)oxy)-1,2-phenylene)bis(5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide) (DDPO-2-HQ-DOPO-rear, ***12****).* In a flame-dried flask equipped with a condenser and an argon inlet, DDPO–2-BQ (**7c**, 808 mg, 2.0 mmol) and DOPO (432 mg, 2.0 mmol) were refluxed in 20 mL of dry toluene for 4 h. The mixture was cooled with an ice bath for 30 min. The product was collected by filtration at reduced pressure, washed with cold toluene, and dried at 0.2 mbar. White solid, yield: 731 mg (59%). mp: decomp. >212°C. ^{31}P NMR (101 MHz, $CDCl_3$, ppm): 19.21 + 19.07 (isomers, 1P DOPO); 14.04 + 13.90 (isomers, 1P—C DDPO); —14.95 (s, 1 P—O DDPO). ^{13}C NMR (63 MHz, $CDCl_3$, ppm): 162.0 (dd, J = 14.6 Hz, J = 7.2 Hz, 1C); 149.2 (d, J = 8.2 Hz, 1C); 147.0 (dd, J = 18.7 Hz, J = 5.3 Hz, 1C); 134.5 (d, J = 5.6 Hz, 1C); 132.8 (d, J = 2.6 Hz, 1C); 130.4 (s, 1C); 129.5 (d, J = 13.7 Hz, 1C); 128.6 (d, J = 15.3 Hz, 1C); 127.2 (d, J = 15.7 Hz, 1C); 125.9 (d, J = 134.0 Hz, 1C—P); 125.8 (d, J = 8.8 Hz, 1C); 124.7 (s, 1C); 124.4 (s, 1C); 123.2 (d, J = 10.1 Hz, 1C); 121.2 (d, J = 12.1 Hz, 1C); 120.7 (d, J = 6.7 Hz, 1C); 117.7 (dm, J_d = 183.0 Hz, 1C—P); 113.9 (dd, J = 195.9 Hz, J = 9.5 Hz, 1C—P); 79.8 (d, J = 7.5 Hz, 1C); 79.2 (d, J = 7.7 Hz, 1C); 77.9 (d, J = 7.2 Hz, 2C); 33.2 (d, J = 15.5 Hz, 1C); 31.4 (d, J = 6.1 Hz, 1C); 24.4 (s, 1C); 22.1 (s, 1C); 21.2 (s, 1C); 19.5 (s, 1C). ^{1}H NMR (250 MHz, $CDCl_3$, ppm): 12.66 (s, 1H, Aryl-OH); 8.09 (t, J = 7.10 Hz, 2H, arom.); 7.72 (m, 2H, arom); 7.48 (m, 2H, arom.); 7.39 (d, J = 6.97 Hz, 1H, arom.); 7.30 (d, J = 8.74 Hz, 2H, arom); 7.22 (m, 1H, arom.); 4.96 (dd, J = 9.42 Hz, J = 3.94, 1H, —CH_2—); 4.79 (dd, J = 9.33 Hz, J = 4.35 Hz, 1 H, —CH_2—); 4.01 (dq, J_q = 12.31 Hz, J_d = 2.4 Hz, 2 H, —CH_2—); 3.56 (m, 2 H, —CH_2—); (dd, J = 24.3 Hz, J = 10.9 Hz, 1 H, —CH_2—); 3.13 (d, J = 11.0 Hz, 1 H, —CH_2—); 1.64 (s, 3 H, —CH_3 ax.); 1.05 (s, 3 H, —CH_3 ax.); 0.83 (s, 3H, —CH_3 equ.); 0.48 (s, 3 H, —CH_3 equ.). IR (KBr) ν 3075 (w, C—H aryl); 2967 (w, C—H alkyl); 2885 (w, C—H alkyl); 1595 (w); 1584 (w); 1479 (m, P—C_{aryl}); 1432 (m, P—C_{aryl}); 1375 (w); 1316 (m); 1296 (m, P═O); 1255 (s, P═O); 1215 (m); 1170 (m, P—OC_{aryl}); 1119 (w); 1062 (s, P—OC_{alkyl}); 1009 (m, P—OC_{alkyl}); 991 (m); 969 (m); 940 (m); 909 (m); 836 (m); 813 (m); 792 (m); 757 (m); 734 (w); 715 (w); 632 (m); 524 (w); 508 (w); 492 (m). HRMS (EI) calcd. for $[^{12}C_{28}H_{31}O_{10}P_3]^+$ 620.1130, found 620.1126. Anal calcd. for $C_{42}H_{33}O_5P_3$: C 70.99, H 4.68; found: C 70.66, H 4.84.

(3,6-Dihydroxybenzene-1,2,4-triyl)tris(diphenylphosphine oxide) (DPhPO-3-HQ, ***13****).* DPhPO-2-HQ (**5a**, 5.10 g, 10 mmol), activated manganese dioxide (20.0 g), and diphenylphosphine oxide (2.02 g, 10 mmol) were processed in the manner described for **5a**. White solid, yield: 2.58 g (36%). mp: decomp. >220°C. ^{31}P NMR (101 MHz, $CDCl_3$, ppm): 52.86 (s, 1P); 38.97 (s, 1P); 32.80 (s, 1P). ^{13}C NMR (101 MHz, $CDCl_3$, ppm): 156.6 (m, 1C—OH); 152.5 (m, 1 C—OH); 131.3–123.2 (37C);* 118.1 (d, J = 116.0 Hz, 1C—P); 116.0 (d, J = 101.0 Hz, 1C—P); 115.9 (d, J = 100.1 Hz, 1C—P). ^{1}H NMR (250 MHz, $CDCl_3$, ppm): 13.52 (s, 1H, Aryl-OH); 11.09 (s, 1H, Aryl-OH); 7.83 (dd,

J = 12.8 Hz, J = 7.4 Hz, 3H arom.); 7.75–7.61 (m, 5H, arom.); 7.61–7.52 (m, 4H, arom.); 7.40–7.08 (m, 18H, arom.); 7.00 (dd, J = 14.2 Hz, J = 4.2 Hz, 1H, arom.). IR (KBr) ν 3054 (C—H aryl.); 1572 (w); 1483 (w); 1437 (s, P—Ph); 1353 (m); 1266 (w); 1196 (m); 1165 (s, P=O); 1122 (s); 1094 (m); 1042 (w); 998 (w); 910 (w); 801 (w); 745 (m); 729 (s); 701 (s); 687 (s); 655 (m); 618 (w); 590 (s); 563 (s); 531 (w); 517 (s). HRMS (EI) calcd. $[^{12}C_{42}H_{33}O_5P_3]^+$ 710.1541, found 710.1592. Anal calcd. for $C_{42}H_{33}O_5P_3$: C 70.99, H 4.68; found: C 70.66, H 4.84. *detailed assignment not possible due to limited resolution of the available NMR technology. The structure of **13** was confirmed by X-Ray analysis and HR-MS.

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