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Dimephosphone Analogs: II.[1] Dialkyl(diaryl)-(2-methyl-4-oxopent-2-yl)phosphine Oxide Oximes: Synthesis, Structure, and Generation of Iminoxyl Radicals

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Abstract—Dialkyl(diaryl)-(2-methyl-4-oxopent-2-yl)phosphine oxide oximes have been synthesized for the first time. According to the X-ray diffraction data, these compounds in crystal exist as a single *E* isomer. Their structure in solution and the *E*/*Z* isomer ratio were determined by ^{1}H and ^{13}C NMR spectroscopy.

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The drug Dimephosphone [2-(dimethoxyphosphoryl)-2-methylpentan-4-one] is a γ-phosphorylated ketone which attracts interest as precursor in the synthesis of new potentially biologically active compounds. Unlike most practically important organophosphorus compounds, Dimephosphone is low-toxic, it exhibits no anticholineesterase activity, and was recommended as antidote; it also represents a striking example of successful search for medicinal agents among organophosphorus compounds [2, 3]. Dimephosphone is used as antiacidotic agent in the treatment of acidoses of different origins and breathing disorders and as vasoactive drug in the treatment of cerebral circulation disorders. It displays membrane-stabilizing and anti-inflammatory activity, improves blood circulation, and normalizes cerebral metabolism. In addition, Dimephosphone exhibits cardiotropic, neurotropic, neuroprotective, antihypoxic, antiallergic, immunomodulating, anti-aggregation, antioxidant, radioprotective, and other useful properties [2, 3].

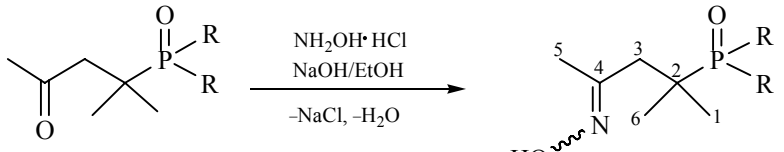
Aza analogs of Dimephosphone, in which the carbonyl group is replaced by a nitrogen containing fragment, also exhibit biological activity [4–7]. For example, Dimephosphone oxime possesses the same properties as the parent ketone [4], and nicotinoyl- and isonicotinoylhydrazones derived therefrom were found to show antitubercular and anti-inflammatory activity, being 13 times less toxic than Isoniazid [1, 5–7].

We previously developed a convenient procedure for the synthesis of other Dimephosphone analogs, dialkyl(diaryl)-(2-methyl-4-oxopent-2-yl)phosphine oxides, where the two alkyl (aryl) radicals are directly attached to the phosphorus atom [8–11]. The procedure utilizes accessible derivatives of phosphorus-containing heterocycles with an endocyclic P–O bond and Grignard reagents. For instance, 2-chloro-3,3,5-trimethyl-1,2λ^{5}-oxaphosphole 2-oxide, which can be readily prepared from simple and commercially accessible phosphorus trichloride and mesityl oxide or diacetone alcohol [12–15], is a convenient starting material for the preparation of dialkyl(diaryl)-(2-methyl-4-oxopent-2-yl)phosphine oxides (**I**). Such phosphine oxides containing a carbonyl group in the γ-position with respect to the phosphorus atom attract interest as efficient fire retardants for poly(vinyl chloride) compositions [8] and as extractants for the isolation of lanthanides from acid medium [16, 17]. Therefore, it seemed reasonable to study some their chemical properties in more detail.

In this article we report on the synthesis of dialkyl-(diaryl)(2-methyl-4-oxopent-2-yl)phosphine oxide

[1] For communication I, see [1].

Scheme 1.



Ia–If **IIa–IIg**

R = Et (**a**), Pr (**b**), Bu (**c**), C_6H_{13} (**d**), CH_2Ph (**e**), Ph (**f**), OMe (**g**).

oximes, their structure, and some chemical properties. It is known that oximes, including phosphorylated ones and Dimephosphone oxime, exhibit a broad spectrum of biological activity and act as antidotes toward organophosphorus chemical warfare agents [2, 4, 18–20]. Taking this into account, we have synthesized oximes **II** by reaction of γ-phosphoryl ketones **I** with hydroxylamine hydrochloride in ethanol at a reactant ratio of 1 : 1.5 (Scheme 1).

A small excess of $NH_2OH \cdot HCl$ was necessary to ensure complete conversion of ketones **I** into oximes **II**, since under the action of sodium hydroxide hydroxylamine free base was partly removed in the gaseous state from the reaction mixture. The optimal conditions included preliminary cooling of a mixture of phosphine oxide and hydroxylamine hydrochloride in ethanol using an ice bath and slow addition of ethanolic sodium hydroxide under vigorous stirring. All oximes **IIa**–**IIg** were isolated in quantitative yield.

The IR data showed that crystalline oximes **IIa**–**IIf**, unlike Dimephosphone oxime (**IIg**) [21, 22], were represented by a single isomer. However, the IR spectra were insufficient to unambiguously determine the isomer structure. By analogy with the data for Dimephosphone aryl- and aroylhydrazones [1, 5–7] and major isomer of Dimephosphone oxime [20, 21] oximes **IIa**–**IIg** can be assigned *E* configuration. This assumption was confirmed experimentally by analysis of their ^1H and ^{13}C NMR spectra, as well as by the X-ray diffraction data for dibutyl(2-methyl-4-oxopent-2-yl)phosphine oxide oxime (**IIc**).

Replacement of the carbonyl group in **Ia**–**Ig** by hydroxyimino in going to oximes **IIa**–**IIg** did not change the chemical shifts and multiplicities of signals in the ^1H and ^{13}C NMR spectra to an appreciable extent. The difference was that two sets of signals were observed in the NMR spectra of oximes **II** due to the presence of *E* and *Z* isomers. According to the ^1H and ^{13}C NMR data, oximes **II** in $CDCl_3$ solution (cf. our previous data for oximes **IIa** and **IIe** [11]) exist as equilibrium mixture of *E* and *Z* isomers. Afonin et al. [23, 24] showed that the methyl carbon atom in the *trans* position with respect to the lone electron pair on the nitrogen atom resonates in a stronger field as compared to the corresponding carbon atom in the *cis*. In the ^{13}C NMR spectrum of **IIa**, the major methyl carbon signal (C^5) appears as a quartet at δ_C 16.67 ppm ($^1J_{HC}$ = 127.6 Hz), which indicates *E* configuration of the major isomer; the minor C^5 signal belonging to the *Z* isomer is located at δ_C 23.0 ppm ($^1J_{HC}$ = 127.6 Hz). The C^3 signals in the proton-coupled ^{13}C NMR spectrum were triplets at δ_C 40.45 (major *E* isomer, $^1J_{HC}$ = 128.2 Hz) and 33.18 ppm (minor *Z* isomer, $^1J_{HC}$ = 128.8 Hz). In all cases, the C=N (C^4) carbon signal of the *Z* isomer was observed in a stronger field, and it displayed coupling with the phosphorus nucleus with a constant J_{CP} of ~11.2–11.6 Hz; the corresponding coupling constant for the *E* isomer was J_{CP} = 13.2–13.6 Hz. The ^{13}C signal intensities provide only a rough estimate of the isomer ratio.

For this purpose, we used the ^1H NMR spectra. In the ^1H NMR spectrum of **IIa**, the major 3-H signal (*E* isomer) appeared as a doublet in a stronger field, at δ 2.25 ppm ($^3J_{PH}$ = 7.7 Hz), and the 3-H proton of the *Z* isomer resonated in a weaker field (δ 2.42 ppm, d, $^3J_{PH}$ = 7.9). The signal intensity ratio was 10:1. Some parameters of the ^{13}C and ^1H NMR spectra of γ-phosphoryl oximes are presented in Table 1.

Figure 1 shows the molecular structure of oxime **IIc** in crystal according to the X-ray diffraction data. Oxime **IIc** has *E* configuration about the C=N bond. The phosphorus atom is characterized by a distorted tetrahedral configuration. The O^1, P^1, C^2, C^3, and C^4 atoms lie almost in one plane (**P**^{1}); the average deviation of atoms from that plane is 0.038(3) Å. Plane **P**^{1} is virtually almost orthogonal to the oxime fragment $C^3C^4C^5N^6O^7$ (**P**^{2}), where the average deviation of atoms from the mean-square plane is 0.017(3) Å. The dihedral angle between **P**^{1} and **P**^{2} is 80.9(2)°. Unlike

Table 1. Some parameters of the ^{13}C and ^{1}H NMR spectra of the *E* and *Z* isomers of γ-phosphoryl oximes **IIa**–**IIf**

Comp. no.	Chemical shifts, δ, δ_C, ppm				*E*/*Z* Isomer ratio
	C^3	H^3	C^4 δ ppm ($^3J_{PC}$, Hz)	C^5	
***Z*-IIa**	33.19	2.42	152.35 (11.5)	23.00	10 : 1
***E*-IIa**	40.45	2.25	152.77 (13.2)	16.67	
***Z*-IIb**	33.41	2.45	152.95 (11.7)	22.69	4 : 1
***E*-IIb**	40.06	2.34	154.46 (13.2)	16.98	
***Z*-IIc**	33.73	2.55	154.11 (11.9)	16.98	8 : 1
***E*-IIc**	40.78	2.39	154.38 (13.2)	23.16	
***Z*-IId**	33.60	2.60	153.44 (11.6)	16.91	5 : 1
***E*-IId**	40.72	2.43	153.81 (13.5)	23.23	
***Z*-IIe**	33.52	2.46	152.45 (11.3)	17.01	6 : 1
***E*-IIe**	40.62	2.25	152.74 (13.2)	22.27	
***Z*-IIf**	33.36	2.30	151.99 (13.1)	16.73	4 : 1
***E*-IIf**	40.43	2.51	152.31 (15.3)	22.88	

Dimephosphone 4-nitrophenylhydrazone [1], both $\mathbf{P^1}$ and $\mathbf{P^2}$ fragments in molecule **IIc**, as in Dimephosphone 2-nitrophenylhydrazone and aroylhydrazones [1] are turned apart with respect to the C^3–C^4 bond. Analogous molecular conformation is typical of bis(2-methoxyphenyl)(2-methyl-4-oxopent-2-yl)phosphine oxide, where the torsion angle $C^2C^3C^4C^5$ is –100.4(6)° [11].

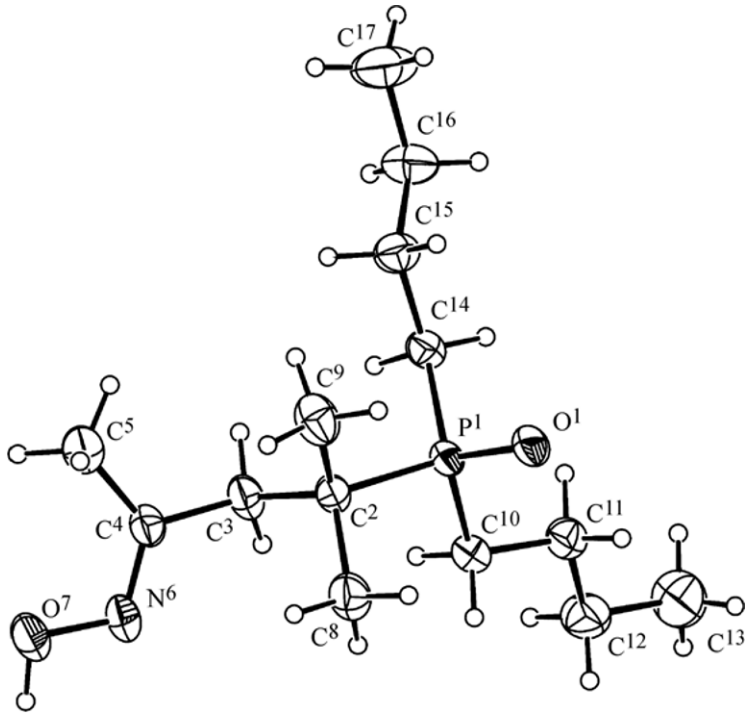


Fig. 1. Structure of the molecule of dibutyl(4-hydroxyimino-2-methylpent-2-yl)phosphine oxide (**IIc**) according to the X-ray diffraction data. Bond lengths, Å: P^1–O^1 1.494(2), P^1–C^2 1.845(3), P^1–C^{10} 1.802(3), P^1–C^{14} 1.802(3), O^7–N^6 1.401(4), O^7–H^7 0.93(5), N^6–C^4 1.297(4). Bond angles, deg: $O^1P^1C^2$ 110.5(1), $O^1P^1C^{10}$ 112.1(1), $C^2P^1C^{10}$ 107.4(1), $C^2P^1C^{14}$ 110.5(1), $C^{10}P^1C^{14}$ 103.4(2), $N^6O^7H^7$ 95(3), $O^7N^6C^4$ 110.2(3). Torsion angles, deg: $O^1P^1C^2C^3$ 175.2(2), $O^1P^1C^2C^9$ 51.6(2), $O^1P^1C^{10}C^{11}$ 55.2(3), $O^1P^1C^{14}C^{15}$ 62.3(3), $O^7N^6C^4C^5$ 3.6(4), $O^7N^6C^4C^3$ 179.5(2), $C^2C^3C^4C^5$ 84.8(4).

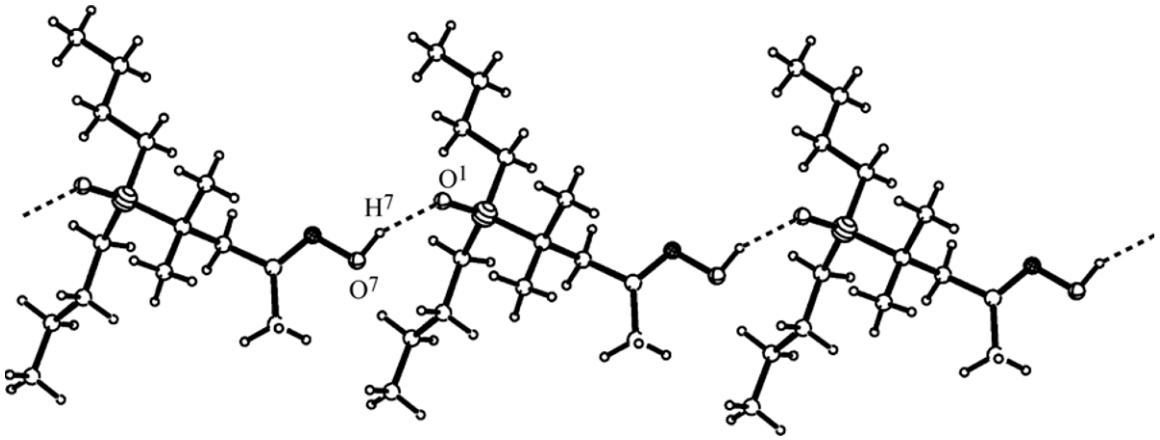


Fig. 2. Crystal packing of dibutyl(4-hydroxyimino-2-methylpent-2-yl)phosphine oxide (**IIc**).

Packing of molecules **IIc** in crystal is determined by intermolecular hydrogen bonds between the hydroxy proton and phosphoryl oxygen [O^7–$H^7\cdots O^1$, O^7–H^7 0.93(5), $H^7\cdots O^1$ 1.79(5), $O^7\cdots O^1$ 2.670(3) Å, $\angle O^7H^7O^1$ 156(4)°, symmetry operation 1 + *x*, *y*, *z*]. These hydrogen bonds give rise to chains along the 0*a* crystallographic axis (Fig. 2).

Oximes are precursors of iminoxyl radicals of the general formula $R^1R^2C{=}N{-}O^{\cdot}$. Due to magnetic properties of the phosphorus nucleus, iminoxyl radicals having phosphorus-containing substituents display high stereospecificity of the hyperfine coupling constants (HCC), which allows reliable determination of the ratio their *Z* and *E* isomers [25]. Iminoxyl radicals containing various phosphorus substituents in the α- and β-positions with respect to the radical center have been studied in sufficient detail [25–27], whereas the available data on their analogs with phosphorus substituents in the γ-positions are fairly limited. Taking into account that the stability and lifetime of iminoxyl radicals strongly depend on the nature of substituents in the alkylidene fragment, we tried to generate iminoxyl radicals by oxidation of oximes **II**.

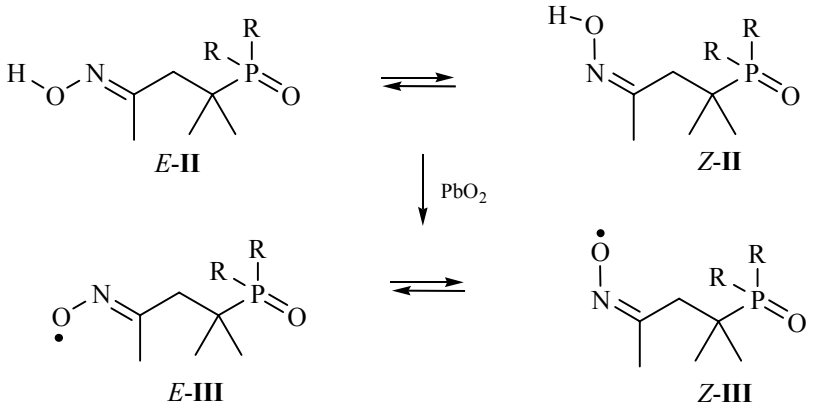
Like parent oximes, iminoxyl radicals in solution may exist as two geometric isomers. The energy barrier to *Z*/*E* isomerization of iminoxyls is lower than that for the corresponding oximes; therefore, the composition of the equilibrium mixture of *Z* and *E* iminoxyl isomers always differs from the isomer composition of the initial oxime [25].

Attempted generation of iminoxyl radicals via oxidation of oximes **II** with lead(IV) oxide in chloroform was unsuccessful. The results indicated more profound processes occurring in that solvent, which led to nitroxyl radicals whose detailed structure we failed to determine. Presumably, nitroxyl radicals are formed as a result of various transformations of initially formed iminoxyl radicals. It is well known that most iminoxyl radicals (unlike nitroxyl radicals) in solution readily undergo C–O, N–O, N–N, C–N, and C–C dimerization, and the resulting dimers are capable of reacting further [25–29]. In addition, intramolecular hydrogen shifts from methyl or methylene groups neighboring to the radical center are possible [28–30]. Therefore, we examined oxidation of oximes **II** with PbO_2 in methylene chloride and toluene, as well as electrochemical oxidation in acetonitrile in the presence of pyridine (MeCN–pyridine ratio 5.0 : 0.1; Scheme 2).

The solubility of oximes **II** in chloroform, methylene chloride, and acetonitrile is higher than in toluene. However, difficulties related to purification of chlorine-containing solvents and fast formation therein of impurities promoting side processes in the oxidation of oximes reduce their value as medium for studying such reactions. Toluene is the most appropriate solvent for ESR studies, but the solubility of γ-phosphoryl oximes **IIa**–**IIf** in that solvent is not always sufficient, and only a small part of generated iminoxyl radicals can be detected by ESR.

Furthermore, iminoxyl radicals with a phosphoryl fragment in the γ-position with respect to the radical center are less stable than their α- and β-phosphoryl analogs [25, 27]. In the oxidation of oxime **IIg** with PbO_2 or electrochemical oxidation, the formation of a mixture of iminoxyl (**IIIg**) and nitroxyl radicals (g = 2.0054, a_N = 9.5 Oe, a_H = 10.67 Oe) was observed in a

Scheme 2.



R = Et (**a**), Pr (**b**), Bu (**c**), C_6H_{13} (**d**), Bn (**e**), Ph (**f**), OCH_3 (**g**).

short time. Prolonged electrochemical oxidation in MeCN in the presence of *t*-BuONa gave rise to two nitroxyl radicals (g = 2.0054, a_N = 14.61 Oe, a_H = 20.03 Oe, a_H = 1.22 Oe; g = 2.0054, a_N = 13.76 Oe). Presumably, initially formed unstable γ-phosphoryl iminoxyl radicals **III** undergo various transformations.

According to the ESR data, γ-phosphoryl oximes **IIa**–**IIf**, as well as Dimephosphone oxime (**IIg**), in the examined solvents exist as two geometric isomers whose ratio is determined mainly by the solvent nature and, to a minor extent, by the nature of substituent in the phosphoryl fragment. The *E*/*Z* isomer ratio for radicals **IIIa**, **IIIb**, and **IIIg** was about 3 : 1). The ESR pattern obtained in the oxidation of oxime **IIe** did not allow us to reliably determine magnetic parameters. The results of our experiments on the generation of γ-phosphoryl iminoxyl radicals and their characteristics are given in Table 2 and Figs. 3 and 4.

The small difference in the magnetic parameters of iminoxyl radicals generated from oximes **IIa**, **IIb**, and **IIg** (Table 2) may be rationalized by the rigidity of the structural fragment linked to the phosphorus atom and remoteness of varied substituents from the radical center. Small differences in the electronic effects of the methoxy groups in **IIIg** and alkyl groups in **IIa**, **IIb** are leveled by the effect of the phosphoryl group.

Table 2. Parameters of the ESR spectra of the *E* and *Z* isomers of γ-phosphoryl iminoxyls **IIIa**, **IIIb**, and **IIIg** generated by electrochemical oxidation of oximes **IIa**, **IIb**, and **IIg** in acetonitrile in the presence of pyridine (acetonitrile–pyridine ratio 5.0 : 0.1)[a]

Comp. no.	Isomer	Isomer fraction, %	g-factor[b]	a_N	$a(CH_3)$	$a(CH_2)$	a_P	a_P
IIIa	Et	*E*	57 (~75)	2.0057 (2.0057)	30.626 (30.578)	1.655 (1.544)	1.469 (1.417)	0.908 (0.792)
		Z	43 (~25)	2.0058 (2.0058)	30.756 (30.513)	1.459 (1.482)	1.267 (1.055)	0.25[c] (>0.28[c])
IIIb	Pr	*E*	51 (~75)	2.0057 (2.0057)	30.678 (30.576)	1.674 (1.545)	1.450 (1.419)	0.901 (0.793)
		Z	49 (~25)	2.0058 (2.0058)	30.756 (30.515)	1.490 (1.480)	1.152 (1.057)	0.25[c] (>0.28[c])
IIIg	MeO	*E*	64 (~75)	2.0057 (2.0057)	30.634 (30.577)	1.711 (1.545)	1.440 (1.418)	0.937 (0.800)
		Z	36 (~25)	2.0058 (2.0058)	30.813 (30.514)	1.591 (1.481)	1.099 (1.056)	0.25[c] (>0.28[c])

[a] Parameters of the ESR spectra of iminoxyls **IIIa**, **IIIb**, and **IIIg** generated by oxidation of oximes **IIa**, **IIb**, and **IIg** with PbO_2 in toluene are given in parentheses. [b] Difference in the fifth decimal point. [c] Maximum possible value of the hyperfine coupling constant with the phosphorus nucleus, which is masked by the line width.

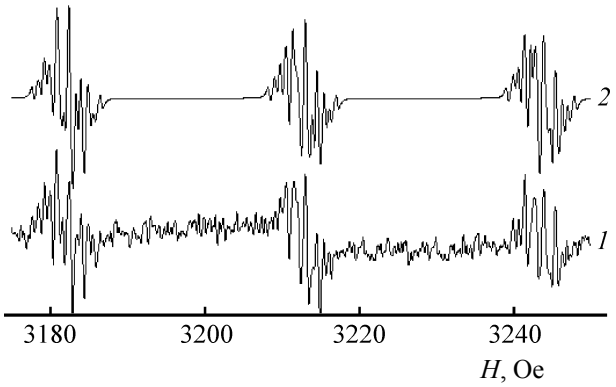


Fig. 3. (*1*) Experimental and (*2*) theoretical ESR spectra of iminoxyl radical **IIIa** generated by oxidation of oxime **IIa** with lead dioxide in toluene.

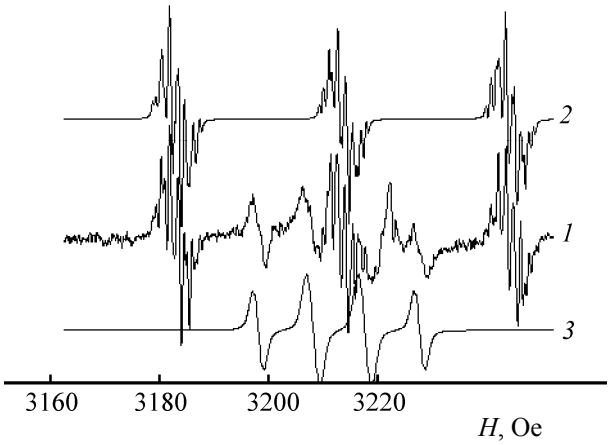


Fig. 4. (*1*) Experimental ESR spectrum of a mixture of radicals generated by electrochemical oxidation of Dimephosphone oxime (**IIg**) in acetonitrile in the presence of pyridine (acetonitrile–pyridine ratio 5.0 : 0.1), (*2*) theoretical ESR spectrum of iminoxyl radical **IIIa**, and (*3*) experimental ESR spectrum of nitroxyl radical generated from **IIIa**.

Hyperfine coupling with a phosphorus nucleus in the γ-position (so-called *W*-coupling) to the radical center in iminoxyl radicals has been reported [25, 27]. However, we observed almost no effect of the phosphorus atom in the ESR spectra of iminoxyl radicals generated by oxidation of oximes **IIa**, **IIb**, and **IIg**. A probable reason is their rigid structure due to the presence of two methyl groups in the α-position with respect to the phosphorus atom.

EXPERIMENTAL

The ^{1}H, ^{31}P, and ^{13}C NMR spectra were recorded on a Bruker Avance-400 spectrometer at 400, 160.9, and 100.6 MHz, respectively. The IR spectra were measured on a Bruker Vector-22 instrument from samples dispersed in mineral oil or pelletized with KBr. The melting points were determined on a Boetius melting point apparatus. The mass spectra (electron impact) were obtained on a Finnigan Trace MS GC/MS system; samples were injected as solutions in ethanol. The ESR spectra were recorded at room temperature on a Radiopan SE/X 2544 spectrometer equipped with an RCX 660 dielectric resonator (variable operating frequency, 9.1–9.8 GHz, klystron source, maximum output power 100 mW) at a constant microwave radiation frequency with variation of the magnetic field using external magnetic field modulation.

The X-ray diffraction data for a single crystal of oxime **IIc** were obtained at 293 K on a Bruker Smart APEX II CCD automatic diffractometer (graphite monochromator; λMoK_α radiation, λ 0.71073 Å; ω-scanning). A correction for absorption was applied semiempirically using SADABS program [31]. The structure was solved by the direct method using SIR program [32] and was refined in isotropic and anisotropic approximations with the aid of SHELXL–97 software package [33]. The position of the H^7 atom was determined from the difference Fourier maps and was refined in isotropic approximation. The other hydrogen atoms were placed into geometrically calculated positions and were included in the refinement procedure according to the riding model. All calculations were performed using WinGX [34] and APEX2 [35]. The molecular structure was plotted, and intermolecular interactions were analyzed, using PLATON [36] and ORTEP [37].

Colorless prisms, monoclinic crystal system; $C_{14}H_{30}NO_2P$; M 275.36; unit cell parameters: a = 9.1697(17), b = 20.772(4), c = 9.3557(18) Å; β = 109.636(2)°; V = 1678.4(5) Å^3; d_{calc} = 1.090 g/cm^3; Z = 4; space group $P2_1/n$; scan range 1.96° < θ < 26.00°; μ(MoK_α) = 1.61 cm^{-1}. Total of 3106 independent reflection intensities were measured, 2423 of which were characterized by $I \geq 2\sigma(I)$. Final divergence factors: R = 0.0617, R_w = 0.1442 for 2423 reflections with $F > 2\sigma(F^2)$.

Generation of iminoxyl radicals. A solution of oxime **IIa**, **IIb**, or **IId–IIg** in chloroform, methylene chloride, toluene, or acetonitrile with a concentration approaching saturation was placed into a 4-mm

(external diameter) ampule, and a required amount of lead dioxide was added. The ampule was shaken, cooled with liquid nitrogen, evacuated using a forevacuum pump, and defrosted. This procedure was repeated in triplicate to ensure complete deoxygenation, and the ampule was sealed. When chloroform was used as solvent (oxime concentration 1 mM), after addition of PbO_2, the mixture was purged with argon over a period of 10 min, and the ampule was sealed.

The ampule containing the reaction mixture was then placed into a 5-mm ER4118MD5-W1 dielectric cylindrical resonator, and samples were irradiated at a power of 2 mW; static magnetic field was modulated by alternating magnetic field with a frequency of 100 kHz and an amplitude of 1 G.

ESR studies in combination with electrolysis in situ were carried out using a setup consisting of a Radiopan SE/X-2544 radiospectrometer, a potentiostat, and a custom-made electrochemical cell. This setup allowed the electrolysis to be performed directly in the ESR probe. A platinum plate was used as working electrode, platinum wire was used as auxiliary electrode, and silver wire was a reference electrode. Solutions were deaerated by three freeze–evacuation–thaw cycles. The electrolysis was carried out in acetonitrile at 293–333 K with 0.1 M Et_4NClO_4 as supporting electrolyte. Organic solvents were dried and purified according to standard procedures. Tetrabutylammonium tetrafluoroborate and sodium *tert*-butoxide were used without additional purification.

Dimethyl (4-hydroxyimino-2-methylpent-2-yl)phosphonate (**IIg**) was synthesized according to the procedure described in [21].

General procedure for the synthesis of oximes IIa–IIf. A mixture of 0.0245 mol of phosphine oxide **Ia–If** and 3.8 g (0.0539 mol) of hydroxylamine hydrochloride in 10 mL of ethanol was cooled in an ice bath, and a solution of 2.2 g (0.0539 mol) of sodium hydroxide in 10 mL of ethanol was added dropwise under stirring. When the reaction was complete, the mixture was heated to the boiling point and cooled to room temperature, the solvent was removed, and the residue was dried at 60°C under reduced pressure (10 mm). The residue was dissolved in methylene chloride, the undissolved material was filtered off, the solvent was removed from the filtrate under reduced pressure (60°C, 10 mm), and the oily residue crystallized on storage. After washing with diethyl ether, a white crystalline substance was obtained.

Diethyl(4-hydroxyimino-2-methylpent-2-yl)-phosphine oxide (IIa). Yield 82%, mp 98°C; *Z*/*E* ratio 1 : 10. IR spectrum, ν, cm^{-1}: 468, 529, 603, 659, 709, 762, 789, 840, 883, 948, 972, 1023, 1135, 1168, 1240, 1276, 1324, 1370, 1390, 1411, 1466, 1648, 2164, 2883, 2943, 2972, 3068, 3200, 3385. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.01 d (6H, 1-H, 6-H, *E*, $^3J_{PH}$ = 14.2), 1.05 d (6H, 1-H, 6-H, *Z*, $^3J_{PH}$ = 14.1), 1.05 br.t (6H, 8-H, $^3J_{HH}$ = 15.2), 1.45–1.72 m (4H, 7-H, *AB* part of *ABMX*$_3$ spin system), 1.73 br.s (3H, 5-H, *E*), 1.74 br.s (3H, 5-H, *Z*), 2.25 d (2H, 3-H, *E*, $^3J_{PH}$ = 7.7), 2.42 d (2H, 3-H, *Z*, $^3J_{PH}$ = 7.9), 10.79 s (1H, OH). $^{13}C–\{^1H\}$ NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz) (hereinafter, the multiplicity of signals in the proton-coupled spectrum is given parentheses): 6.14 q.d. t (d) (C^8, *E*, $^1J_{HC}$ = 128.7, $^2J_{PC}$ = 5.4, $^2J_{HC} \approx$ 5.4), 6.16 br.q (d) (C^8, *Z*, $^1J_{HC} \approx$ 128.7, $^2J_{PC}$ = 5.3, $^2J_{HC} \approx$ 5.4), 16.31 br.d.t (d) (C^7, *Z*, $^1J_{HC} \approx$ 126.1, $^1J_{PC}$ = 62.0), 16.41 br.d.t (d) (C^7, *Z*, $^1J_{HC}$ = 126.1, $^1J_{PC}$ = 62.0), 16.67 br.q (s) (C^5, *E*, $^1J_{HC}$ = 127.6), 21.09 q.m (s) (C^1, C^6, *E*, $^1J_{HC}$ = 127.8), 21.94 q.m (s) (C^1, C^6, *Z*, $^1J_{HC}$ = 127.9), 23.00 br.q (s) (C^5, *Z*, $^1J_{HC}$ = 127.6), 33.19 br.t (s) (C^3, *Z*, $^1J_{HC}$ = 128.8), 35.75 br.d (d) (C^2, $^1J_{PC}$ = 64.2), 40.45 br.t (s) (C^3, *E*, $^1J_{HC}$ = 128.2), 152.35 m (d) (C^4, *Z*, $^3J_{PC}$ = 11.5), 152.77 m (d) (C^4, *E*, $^3J_{PC}$ = 13.2). $^{31}P–\{^1H\}$ NMR spectrum ($CDCl_3$), δ_P, ppm: 58.1 (*E*), 58.8 (*Z*). Found, %: C 54.63; H 10.47; N 6.34; P 13.95. $C_{10}H_{22}NO_2P$. Calculated, %: C 54.78; H 10.11; N 6.39; P 14.13.

(4-Hydroxyimino-2-methylpent-2-yl)dipropyl-phosphine oxide (IIb). Yield 95%, mp 105°C, *Z*/*E* ratio 1 : 4. IR spectrum, ν, cm^{-1}: 468, 523, 549, 602, 624, 714, 738, 762, 788, 844, 882, 904, 951, 975, 1027, 1135, 1165, 1239, 1321, 1370, 1388, 1408, 1463, 1649, 2871, 2930, 2964, 3057, 3156. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 0.85 t (6H, 9-H, $^3J_{HH}$ = 7.0), 0.86 t (6H, 9-H, $^3J_{HH}$ = 7.2), 1.02 d (6H, 1-H, 6-H, *E*, $^3J_{PH}$ = 14.6), 1.05 d (6H, 1-H, 6-H, *Z*, $^3J_{PH}$ = 14.9), 1.38–1.73 m (8H, 7-H, 8-H), 1.79 br.s (3H, 5-H, *E*) 1.81 s (3H, 5-H, *Z*), 2.34 d (2H, 3-H, *E*, $^3J_{PH}$ = 7.9), 2.45 d (2H, 3-H, *Z*, $^3J_{PH}$ = 8.1), 11.38 br.s (1H, OH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz): 15.60 t.d.m (d) (C^8, $^1J_{HC}$ = 128.3, $^2J_{PC}$ = 4.4), 15.79 q.d.m (d) (C^9, *E*, $^1J_{HC}$ = 125.0, $^3J_{PC}$ = 14.2), 15.82 q.d.m (d) (C^9, *Z*, $^1J_{HC}$ = 125.7, $^3J_{PC}$ = 14.1), 16.98 br.q (s) (C^5, *E*, $^1J_{HC} \approx$ 129.0), 21.01 q.m (s) (C^1, C^6, *E*, $^1J_{HC}$ = 128.0), 21.82 q.m (s) (C^1, C^6, *Z*, $^1J_{HC}$ = 128.0), 22.69 br.q (s) (C^5, *Z*, $^1J_{HC}$ = 128.2), 25.98 br.d.t (d) (C^7, *Z*, $^1J_{HC} \approx$ 127.7, $^1J_{PC}$ = 60.8), 26.09 br.d.t (d) (C^7, *Z*, $^1J_{HC}$ = 126.6, $^1J_{PC}$ = 60.8), 33.41 br.t (s) (C^3, *Z*, $^1J_{HC}$ = 129.3),

35.72 br.d (d) (C^2, $^1J_{PC}$ = 64.0), 40.06 br.t (s) (C^3, *E*, $^1J_{HC}$ = 129.4), 153.95 m (d) (C^4, *Z*, $^3J_{PC}$ = 11.7), 154.46 m (d) (C^4, *E*, $^3J_{PC}$ = 13.2). $^{31}P–\{^1H\}$ NMR spectrum ($CDCl_3$), δ_P, ppm: 59.1 (*E*), 59.8 (*Z*). Found, %: C 58.25; H 11.54; N 5.68; P 11.55. $C_{12}H_{26}NO_2P$. Calculated, %: C 58.28; H 10.60; N 5.66; P 11.52.

Dibutyl(4-hydroxyimino-2-methylpent-2-yl)-phosphine oxide (IIc). Yield 75%, mp 97°C, *Z*/*E* ratio 1 : 8. IR spectrum, ν, cm^{-1}: 439, 471, 497, 529, 584, 623, 684, 719, 742, 753, 795, 842, 875, 901, 952, 972, 997, 1019, 1050, 1063, 1112, 1123, 1164, 1193, 1210, 1222, 1235, 1285, 1303, 1329, 1377, 1419, 1464, 1641, 2856, 2922, 3139. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 0.87 t (6H, 10-H, $^3J_{HH}$ = 7.3), 1.12 d (6H, 1-H, 6-H, *E*, $^3J_{PH}$ = 14.5), 1.17 d (6H, 1-H, 6-H, *Z*, $^3J_{PH}$ = 14.6), 1.35 m (4H, 8-H), 1.45–1.67 m (4H, 9-H), 1.74 m (4H, 7-H, *AB* part of *ABMX*$_3$), 1.88 s (3H, 5-H) 2.39 d (2H, 3-H, *E*, $^3J_{PH}$ = 7.4) 2.55 d (2H, 3-H, *Z*, $^3J_{PH}$ = 7.7). ^{13}C NMR spectrum ($CDCl_3$, 32°C), δ_C, ppm (*J*, Hz): 13.65 q.t.t (s) (C^{10}, $^1J_{HC}$ = 124.9, $^3J_{HC}$ = 3.3–4.4), 16.98 br.q (s) (C^5, *E*, $^1J_{HC}$ = 125.0), 21.46 q.m (br.s) (C^1, C^6, $^1J_{HC}$ = 127.8, $^3J_{PC}$ = 4.5), 23.16 br.q (s) (C^5, *Z*, $^1J_{HC} \approx$ 125.0), 24.18 br.d.t (d) (C^7, *Z*, $^1J_{HC}$ = 123.6, $^1J_{PC}$ = 62.1), 24.28 br.d.t (d) (C^7, *E*, $^1J_{HC}$ = 123.6, $^1J_{PC}$ = 61.1), 24.34 t.d.m (d) (C^8, $^1J_{HC}$ = 124.9, $^2J_{PC}$ = 4.4), 24.61 t.d.m (d) (C^9, $^1J_{HC}$ = 129.5, $^3J_{PC}$ = 13.2), 33.73 t (s) (C^3, *Z*, $^1J_{HC}$ = 128.1), 36.17 d (d), (C^2, $^1J_{PC}$ = 64.4), 40.78 t (s) (C^3, *E*, $^1J_{HC}$ = 128.1), 154.11 m (d) (C^4, *Z*, $^3J_{PC}$ = 11.9), 154.38 m (d) (C^4, *E*, $^3J_{PC}$ = 13.2). $^{31}P–\{^1H\}$ NMR spectrum ($CDCl_3$), δ_P, ppm: 58.4 (*E*), 59.4 (*Z*). Found, %: C 61.33; H 11.17; N 5.12; P 11.41. $C_{14}H_{30}NO_2P$. Calculated, %: C 61.09; H 10.90; N 5.09; P 11.27.

Dihexyl(4-hydroxyimino-2-methylpent-2-yl)-phosphine oxide (IId). Yield 89%, yellow–brown crystals, mp 66–67°C, *Z*/*E* ratio 1 : 5. IR spectrum, ν, cm^{-1}: 472, 532, 601, 634, 669, 717, 791, 840, 884, 950, 973, 1003, 1023, 1137, 1168, 1208, 1240, 1261, 1296, 1321, 1369, 1388, 1411, 1467, 1541, 1647, 2341, 2360, 2871, 2929, 2958, 3062, 3177. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 0.88 t (6H, 12-H, $^3J_{HH}$ = 6.9), 1.17 d (6H, 1-H, 6-H, *E*, $^3J_{PH}$ = 14.7), 1.25 d (6H, 1-H, 6-H, *Z*, $^3J_{PH}$ = 15.6), 1.27 m (8H, 10-H, 11-H), 1.38 m (4H, 7-H), 1.51–1.83 m (8H, 8-H, 9-H), 1.92 s (3H, 5-H, *E*), 1.93 s (3H, 5-H, *Z*), 2.43 d (2H, 3-H, *E*, $^3J_{PH}$ = 7.5), 2.60 d (2H, 3-H, *Z*, $^3J_{PH}$ = 7.8), 9.13 br.s (1H, OH). ^{13}C NMR spectrum ($CDCl_3$, 32°C), δ_C, ppm (*J*, Hz): 13.96 br.q.t (s) (C^{12}, *E*, $^1J_{HC}$ = 124.5, $^3J_{HC}$ = 3.7), 15.19 br.q.t (s) (C^{12}, *Z*, $^1J_{NC} \approx$ 125.9, $^3J_{HC}$ = 4.0), 16.91 q.t (s) (C^5, *E*, $^1J_{HC}$ = 128.8), 21.34 q.m (br.s) (C^1, C^6, $^1J_{HC}$ = 127.7), 22.23 t.m (d) (C^8, *E*, $^1J_{HC} \approx$ 127.4, $^2J_{PC}$ = 4.5), 22.25 t.m (d) (C^8, *Z*, $^1J_{HC} \approx$ 127.4, $^2J_{PC}$ = 4.4), 22.44 t.m (s) (C^{11}, $^1J_{HC}$ = 125.1), 23.23 q.m (s) (C^5, *Z*, $^1J_{HC}$ = 127.5), 24.44 br.d.t (d) (C^7, *E*, $^1J_{HC} \approx$ 126.0, $^1J_{PC}$ = 60.9), 24.52 br.d.t (d) (C^7, *E*, $^1J_{HC}$ = 126.3, $^1J_{PC}$ = 60.9), 31.20 t.d.m (d) (C^9, $^1J_{HC} \approx$ 126.1, $^3J_{PC}$ = 12.9), 31.33 br.t.m (s) (C^{10}, $^1J_{HC} \approx$ 125.6), 33.60 br.t (s) (C^3, *Z*, $^1J_{HC}$ = 125.5), 36.09 br.d (d) (C^2, $^1J_{PC}$ = 64.1), 40.72 br.t (s) (C^3, *E*, $^1J_{HC}$ = 128.9), 153.44 m (d) (C^4, *Z*, $^3J_{PC}$ = 11.6), 153.81 m (d) (C^4, *E*, $^3J_{PC}$ = 13.5). $^{31}P–\{^1H\}$ NMR spectrum ($CDCl_3$), δ_P, ppm 56.6 (*E*), 56.8 (*Z*). Found, %: C 65.17; H 11.77; N 4.39; P 9.44. $C_{18}H_{38}NO_2P$. Calculated, %: C 65.26; H 11.48; N 4.23; P 9.37.

Dibenzyl(4-hydroxyimino-2-methylpent-2-yl)-phosphine oxide (IIe). Yield 77%, mp 145–147°C, *Z*/*E* ratio 1 : 6. IR spectrum, ν, cm^{-1}: 698, 834, 969, 1000, 1113, 1124, 1170, 1239, 1377, 1394, 1408, 1463, 1496, 1580, 2669, 2855, 2924, 3147. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 0.98 d (6H, 1-H, 6-H, *E*, $^3J_{PH}$ = 14.9), 1.01 d (6H, 1-H, 6-H, *Z*, $^3J_{PH}$ = 14.9), 1.70 s (3H, 5-H, *E*), 1.75 s (3H, 5-H, *Z*), 2.25 d (2H, 3-H, *E*, $^3J_{PH}$ = 8.3), 2.46 d (2H, 3-H, *Z*, $^3J_{PH}$ = 8.3), 3.02 m (2H, 7-H, *A* part of *ABX*, $^2J_{AB}$ = 14.8, $^2J_{AX}$ = 11.9 Hz), 3.28 m (2H, 7-H, *B* part of *ABX*, $^2J_{BA}$ = 14.8, $^2J_{BX}$ = 11.9 Hz), 7.15–7.25 m (10H, 9-H, 10-H, 11-H), 10.59 s (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm (*J*, Hz): 17.01 br.q (s) (C^5, *E*, $^1J_{HC}$ = 128.1), 21.61 q.m (s) (C^1, C^6, *E*, $^1J_{HC}$ = 127.9), 22.27 br.q (s) (C^5, *Z*, $^1J_{HC}$ = 127.9), 22.46 q.m (s) (C^1, C^6, *Z*, $^1J_{HC}$ = 128.0), 31.38 t.d (d) (C^7, *Z*, $^1J_{PC}$ = 56.2, $^1J_{HC}$ = 125.0), 31.65 t.d (d) (C^7, *E*, $^1J_{PC}$ = 56.3, $^1J_{HC}$ = 128.2), 33.52 br.t (s) (C^3, *Z*, $^1J_{HC}$ = 125.0), 36.81 br.d (d) (C^2, $^1J_{PC}$ = 64.2), 40.62 br.t (s) (C^3, *E*, $^1J_{HC}$ = 125.6), 152.45 m (d) (C^4, *Z*, $^3J_{RC}$ = 11.3), 152.74 m (d) (C^4, *E*, $^3J_{PC}$ = 13.2), 133.14 m (d) (C^8, $^2J_{PC}$ = 7.8), 130.28 d.m (d) (C^9, $^1J_{HC}$ = 160.3, $^3J_{PC}$ = 4.9), 128.37 d.m (d) (C^{10}, *Z*, $^1J_{HC}$ = 160.2, $^3J_{HC}$ = 4.3, $^4J_{PC}$ = 1.7), 128.42 d.m (d) (C^{10}, *Z*, $^1J_{HC}$ = 160.9, $^4J_{PC}$ = 1.7), 126.54 d.m (d) (C^{11}, $^1J_{HC}$ = 160.8, $^5J_{PC}$ = 2.1). $^{31}P–\{^1H\}$ NMR spectrum ($CDCl_3$), δ_P, ppm: 50.2 (*E*), 50.8 (*Z*). Found, %: C 70.11; H 7.85; N 4.12; P 8.78. $C_{20}H_{26}NO_2P$. Calculated, %: C 69.97; H 7.58; N 4.08; P 9.04.

(4-Hydroxyimino-2-methylpent-2-yl)diphenyl-phosphine oxide (IIf). Yield 98%, mp 207–208°C; published data [38]: mp 214–216°C; *Z*/*E* ratio 1 : 4. IR spectrum, ν, cm^{-1}: 420, 511, 541, 582, 715, 753, 780, 937, 996, 1022, 1075, 1093, 1109, 1139, 1174, 1339, 1360, 1383, 1416, 1436, 1468, 1723, 2887, 2919, 2970, 2990, 3055, 3412. ^1H NMR spectrum ($CDCl_3$),

δ, ppm (*J*, Hz): 1.10 d (6H, 1-H, 6-H, *E*, $^3J_{HH}$ = 15.8), 1.13 d (6H, 1-H, 6-H, *Z*, $^3J_{HH}$ = 15.8), 1.68 s (3H, 5-H, *E*), 1.74 s (3H, 5-H, *Z*), 2.30 d (2H, 3-H, $^3J_{PH}$ = 7.5), 2.51 d (2H, 3-H, $^3J_{PH}$ = 8.3), 7.50–7.64 m (7H, 8-H, 9-H, 10-H, OH), 7.96 m (4H, 7-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz): 16.73 q.t (s) (C^5, *E*, $^1J_{HC}$ = 127.0, $^3J_{HC}$ = 3.0), 21.62 q.m (s) (C^1, C^6, *E*, $^1J_{HC}$ = 127.9), 22.57 q.m (s) (C^1, C^6, *Z*, $^1J_{HC}$ = 128.1), 22.88 br.q (s) (C^5, *Z*, $^1J_{HC} \approx$ 125.6), 33.36 br.t (s) (C^3, *Z*, $^1J_{HC}$ = 126.4), 36.72 br.d (d) (C^2, *E*, $^1J_{PC}$ = 69.4), 36.99 br.d (d) (C^2, *E*, $^1J_{PC}$ = 69.2), 40.43 br.t (s) (C^3, *E*, $^1J_{HC}$ = 125.0), 128.72 d.d.d (d) (C^9, $^1J_{HC}$ = 162.3, $^3J_{HC}$ = 7.0, $^3J_{PC}$ = 10.7), 130.47 d.m (d) (C^7, *E*, $^1J_{PC}$ = 89.6, $^3J_{HC}$ = 7.0), 130.67 d.m (d) (C^7, *Z*, $^2J_{PC}$ = 89.4), 132.00 d.m (d) (C^{10}, $^1J_{HC}$ = 161.5, $^3J_{HC}$ = 6.7, $^4J_{PC}$ = 2.5), 132.22 d.m (d) (C^8, $^1J_{HC}$ = 161.2, $^3J_{HC}$ = 6.2, $^2J_{PC}$ = 8.0), 151.99 m (d) (C^4, *Z*, $^3J_{PC}$ = 13.1), 152.31 m (d) (C^4, *E*, $^3J_{PC}$ = 15.3). ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm 61.5 (*E*), 64.6 (*Z*). Found, %: C 68.63; H 7.13; N 4.51; P 9.71. $C_{18}H_{22}NO_2P$. Calculated, %: C 68.57; H 6.98; N 4.44; P 9.84.

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