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PHOSPHORYLNITRILE OXIDES.
6.* 1,3-CYCLOADDITION WITH AZOMETHINES AND NITRILES

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The 1,3-dipolar cycloaddition of diisopropoxyphosphorylnitrile oxide to Schiff bases gives 3-diisopropoxyphosphoryl-4-R-5-phenyl-1,2,4-oxadiazolines. The cycloaddition of phosphorylated nitrile oxides at the azomethine bond of hydrazones does not proceed due to the low dipolarophilic activity of the CH=N bond and high tendency of phosphorylnitrile oxides to undergo dimerization and polymerization. The reaction of nitrile oxides with 2-hydroxybenzylidenaniline leads to the corresponding ester of diisopropoxyphosphorylformhydroxamic acid. The nitrile group undergoes cycloaddition only when activated. The reaction of a solution of phosphorylnitrile oxides in benzene with tetracyanoethylene gives the bisadduct at the two C=N bonds without affecting the C=C bond.

The 1,3-dipolar cycloaddition of aromatic and aliphatic nitrile oxides to carbon–carbon multiple bonds has been discussed extensively [2, 3]. On the other hand, the reaction of nitrile oxides with dipolarophiles containing carbon–heteroatom multiple bonds has not been studied as thoroughly although this is a convenient pathway for the synthesis of several heterocyclic compounds such as oxadiazolines and oxadiazoles.

In previous work [4-8], we have reported that diisopropoxylphosphorylnitrile oxide (I) obtained from the oxime of diisopropoxyphosphorylcarbonyl halides readily undergoes 1,3-dipolar cycloaddition with alkenes and alkynes to give the corresponding phosphorylated isoxazolines and isoxazoles.

Extension of the cycloaddition reaction to dipolarophiles containing carbon–heteroatom multiple bonds, in particular, azomethine and nitrile bonds, would be expected to give phosphorylated oxadiazolines and oxadiazoles with the phosphoryl group at $C_{(3)}$ in the heterocycle. No example of such a reaction was found in the literature. The reactivity of heterodipolarophiles corresponds to the generalizations formulated for cycloaddition involving addends with unsaturated carbon–carbon bonds [3]. The dipolarophilic activity of the C=N bond is comparable to the activity of the C=C bond with the same substitution [3]. Competition between C=C and C=N multiple bonds was observed in the cycloaddition of nitrile oxides to several unsaturated heterocyclic compounds and compounds capable of tautomerization. For example, the use of β-diketone monoimines as the dipolarophile gives both monoadducts formed from the ketoenamine tautomer and bisadducts obtained from the enolimine form [9]. The dipolar cycloaddition of benzonitrile oxides to 1,4-diazepins takes place exclusively at the $C_{(7)}=N_{(1)}$ bond of the diazepin [10]. Hence, the cycloaddition of phosphorylnitrile oxides to an azomethine bond should lead to the corresponding phosphorylated heterocycle.

Phosphorylated nitrile oxides are more reactive than aromatic nitrile oxides. However, the enhanced activity of the phosphorylated analogs in cycloaddition reactions is accompanied by considerable facilitation of oligomerization and dimerization side-reactions. Thus, the reaction conditions, concentration of the addends, and rate of introduction of the phosphorylnitrile oxide should be varied to enhance the yield of the required cycloadduct depending on the activity of the dipolarophile [8]. We might expect that the presence of a phosphoryl substituent in the dipole would also affect the cycloaddition in going to cycloaddition reactions involving dipolarophiles containing an azomethine bond.

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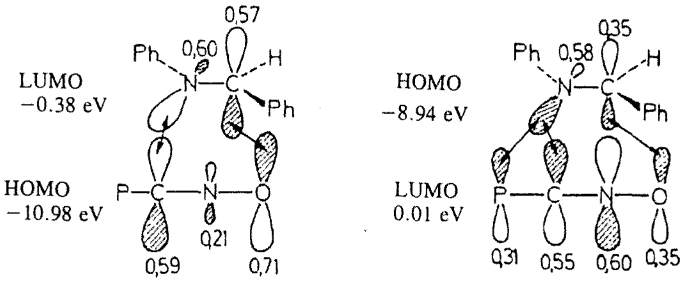
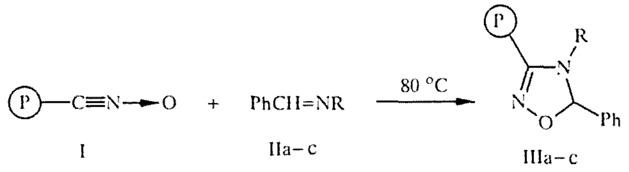


Fig. 1. Scheme of the interacting frontier molecular orbitals of model dimethoxyphosphorylnitrile oxide and benzylidenaniline (MNDO calculation).

Indeed, benzylidenaniline (IIa), which was selected as the simplest dipolarophile containing a CH=N bond, under conditions optimal for cycloaddition involving alkenes and alkynes, does not give the expected Δ^2-1,2,4-oxadiazoline in the reaction with phosphorylnitrile oxide I obtained *in situ*. Imine IIa does not react with this phosphorylnitrile oxide below 0°C, while this reaction system at room temperature gives a mixture of products that could not be separated into pure compounds. Apparently, the stepwise mechanism for the polymerization of the phosphorylnitrile oxide involving formation of highly reactive intermediates facilitates their reaction with benzylidenaniline and the products of this reaction may be polymer systems involving this aniline derivative. Maintenance of a low concentration of this phosphorylnitrile oxide also is an insufficient condition for the formation of the desired cycloadduct. In this case, maintenance of a constant low concentration of both addends when they are present in equimolar amounts may prove useful for obtaining Δ^2-1,2,4-oxadiazolines.

In order to check this hypothesis, benzene solutions of the reagents [oxime of (diisopropoxyphosphoryl)carbonyl chloride and triethylamine with benzylidenaniline] were simultaneously added dropwise to benzene at reflux. The elevated temperature of the reaction mixture is necessary for additional activation. The reaction mixture obtained after introduction of the reagents and removal of the solvent was subjected to preparative column chromatography. A sample of 3-(diisopropoxyphosphoryl)-4,5-diphenyl-1,2,4-oxadiazoline (IIIa) was obtained in 22% yield.



Ⓟ=(i-PrO)$_2$P(O); II, IIIa R=Ph; II, IIIb R=4-BrC$_6$H$_4$; II, IIIc R=C$_4$H$_9$

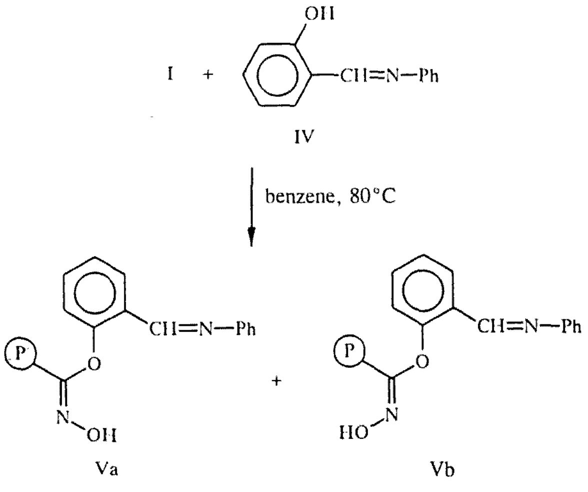
The reactions of benzylidene-4-bromoaniline (IIb) and benzylidenebutylamine (IIc) were carried out under analogous conditions. Preparative column chromatography gave 3-(diisopropoxyphosphoryl)-4-(4-bromophenyl)-5-phenyl-1,2,4-oxadiazoline (IIIb) in 24% yield and 3-(diisopropoxyphosphoryl)-4-butyl-5-phenyl-1,2,4-oxadiazoline (IIIc) in 15% yield.

The structures of cycloadducts IIIa-IIIc were supported by ^{1}H, ^{13}C, and ^{31}P NMR and IR spectral data. The cycloaddition of nitrile oxides to azomethines is regiospecific. The PMR singlet at 6.37 (IIIa), 6.25, (IIIb), and 6.09 ppm (IIIc) corresponds to the single proton at $C_{(5)}$ in the 1,2,4-oxadiazoline ring [11]. We should note that the methylene protons at the nitrogen atom in the butyl group in IIIc appear as two multiplets (3.29 and 3.03 ppm). The ^{13}C NMR spectrum of oxadiazoline IIIa has a doublet for $C_{(3)}$ bound to a phosphorus nucleus at 149.5 ppm and $^1J_{PC}$ 232.0 Hz and a signal related to the methine group carbon of the heterocycle, $C_{(5)}$, at 99.0 ppm with $^1J_{CH}$ 174.0 Hz in addition to the carbon nuclei of the phenyl rings (125.9-138.9 ppm) and isopropoxyl groups (73.2, 24.7, and 23.9 ppm).

We have already noted that phosphorylnitrile oxide I undergoes cycloaddition with the azomethine bond under conditions featuring low addend concentration and elevated temperature. Benzonitrile oxide, which is less reactive than phosphorylnitrile oxide [12, 13], reacts with benzylidenaniline at room temperature over 7 h to give the corresponding 3,4,5-triphenyl-1,2,4-oxadiazoline in 23% yield [11] (the synthesis of this product in 71% yield has been reported by Huisgen et al. [14]). The reduction in temperature and increase in the reaction time were possible due to the high stability of benzonitrile oxide, i.e., the low tendency of this compound to dimerize to the furoxane [3, 12, 13]. In the general case, an increase in the stability of the nitrile oxide and, as a consequence, increase the reaction time for the cycloaddition to carbon–heteroatom multiple bonds permit us to carry out reactions with extremely inert dipolarophiles. Thus, treatment of phenyl isocyanate, which displays very low reactivity of the cumulated C=N bond, with stable mesitonitrile oxide over 15 months leads to the corresponding adduct in 76% yield [14]. Some functional groups containing the C=N bond such as, for example, oxime and hydrazone groups, are reactive relative to nitrile oxides [3] although acid catalysis is required in some cases in order to obtain satisfactory yields. We were unable to carry out the reaction of phosphorylnitrile oxide I with such dipolarophiles as the dimethylhydrazone of benzaldehyde and 1-(3-nitrophenyl)-3-benzylidenehydrazone. The insufficient reactivity of the C=N bond in these dipolarophiles apparently cannot compete with the high tendency of oxide I to undergo dimerization and oligomerization.

As noted above, dipolarophiles with an azomethine bond undergo cycloaddition to nitrile oxides to give a single regioisomer [2, 3, 10, 11]. The observed regiospecificity of the cycloaddition is readily interpreted by analysis of the interacting frontier molecular orbitals. Figure 1 shows that the coefficients of the LUMO-AO at the reaction sites of benzylidenaniline are comparable in magnitude but the HOMO AO coefficient at the nitrogen atom is almost twice as large as for the carbon AO coefficient. Analogous behavior is found for most azomethine compounds. On the other hand, an opposite tendency for the reaction sites is found for nitrile oxides, especially, for phosphorylnitrile oxide, i.e., the LUMO AO coefficient is greatest for the nitrile oxide carbon atom [13]. Since the orientation of the addends in the reaction proceeds according to the principle of greatest overlap of atomic orbitals participating in new bond formation, the nonuniformity of the composition of the LUMO of the dipole and HOMO of the dipolarophile at the reaction sites should be considered a reason for the regiospecificity of the observed reactions.

In an attempt to effect the cycloaddition of 2-hydroxybenzylidenaniline (IV) under conditions analogous to those described above, we isolated a compound whose spectral data differed significantly from the corresponding data for adducts IIIa-IIIc. Thus, the PMR spectrum of this compound lacks the signal characteristic for the proton at $C_{(5)}$ of the oxadiazoline ring. On the other hand, the spectrum has a signal at 8.57 ppm close to the chemical shift of the azomethine proton of starting 2-hydroxybenzylidenaniline (8.39 ppm) and lacks the signal for the phenolic OH group proton (broad signal at 12.84 ppm). In addition, this spectrum has characteristic signals for the protons of the isopropoxyl and phenyl groups. The ^{13}C NMR spectrum features a signal at 162.9 ppm with $^1J_{CH}$ 161.0 Hz, i.e., the downfield signal related to the carbon of the CH=N group, instead of the signal of the $C_{(5)}$ atom of the oxadiazoline ring at 99.0 ppm with $^1J_{CH}$ 174.0 Hz. These data as well as the finding of two signals of equal intensity in the ^{31}P NMR spectrum at −1.83 and −3.14 ppm indicate that the product of this reaction is the 2-oxybenzylidenanil ester of (diisopropoxyphosphoryl)formhydroxamic acid (V) as a 1:1 mixture of E and Z isomers. The formation of the Z forms is probably a consequence of thermal isomerization of the E isomer Va formed during the reaction.



Ⓟ=$(i\text{-PrO})_2P(O)$

Thus, the nucleophilic activity of the hydroxyl group oxygen in 2-hydroxybenzylidenaniline is greater than the dipolarophilic activity of the CH=N bond, which leads to a 1,3-adduct with phosphorylnitrile oxide I as the result of nucleophilic addition.

The C≡N bond is a weaker dipolarophile than the C=C and C=N bonds [3]. Unactivated aliphatic nitriles react with nitrile oxides only in the presence of a catalyst such as BF_3 etherate [15] or when these reagents are present in large

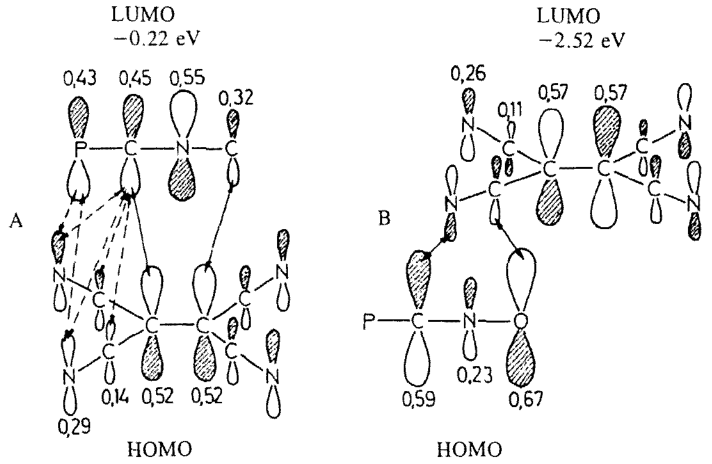
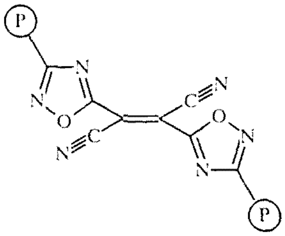


Fig. 2. Scheme for interaction of the frontier molecular orbitals of dimethoxyphosphorylnitrile oxide and tetracyanoethylene (AM1 calculation). A) unrealized type of cycloaddition, B) experimentally confirmed cycloaddition. ↔ represent orbital interaction facilitating bond formation and ←-→ represent orbital interactions hindering bond formation.

excess [16]. However, an attempt to carry out the cycloaddition of phosphorylnitrile oxide I with excess acetonitrile at the boiling point of acetonitrile proved unsuccessful.

Unsaturated nitriles such as acrylonitrile or propiolonitrile react with nitrile oxides to give exclusively cyano-substituted heterocycles [3]. Similarly, the cycloaddition of phosphorylnitrile oxide I to 3-nitrilecyclopropenes proceeds only at the cyclopropene double bond [7]. When the $C=C$ bond is deactivated, the $C\equiv N$ bond may undergo cycloaddition. Thus, some ethylenenitriles such as tetracyanoethylene add nitrile oxides to give 1,2,4-oxadiazoles [17]. Two nitrile groups of tetracyanoethylene may enter the reaction without affecting the ethylene fragment. The bisadduct obtained is capable of forming an unstable donor−acceptor complex with tetracyanoethylene [17].

In light of the electron-donor capacity of phosphorylnitrile oxides [13], the absolute inertness of the $C\equiv N$ bond in acetonitrile may be attributed to the high energy of the LUMO (1.66 eV from an AM1 calculation). Hence, we would expect that lowering the LUMO by the action of electron-withdrawing substituents would considerably activate the $C\equiv N$ bond. This indeed occurs in the case of tetracyanoethylene, in which the LUMO is 4.18 eV lower than for acetonitrile, such that the nitrile bond may react with a dipole. We correspondingly carried out the cycloaddition of phosphorylnitrile oxide I to tetracyanoethylene. Since tetracyanoethylene reacts with triethylamine, the preparation of diisopropoxyphosphorylnitrile oxide from the oxime of (diisopropoxyphosphoryl)carbonyl chloride *in situ* becomes impossible. Thus, a previously prepared benzene solution of phosphorylnitrile oxide I at close to 0°C was mixed with a three-fold excess of tetracyanoethylene also in benzene solution and the reaction mixture was heated at reflux for 6 h. Preparative column chromatography on silica gel gave bisadduct VI as colorless crystals and a complex of VI with tetracyanoethylene as an oil. The dark brown complex isolated is stable only in benzene or CCl_4 solution and dissociates into the bisadduct and tetracyanoethylene upon removal of the solvent.



VI

Thus, the reaction of phosphorylnitrile oxide I with tetracyanoethylene, similar to aromatic nitrile oxides [17], does not involve the $C=C$ bond. This occurs even though the coefficients of the atomic orbitals on the ethylene bond carbons are much greater than the coefficients on the atoms of the nitrile group. The observed selectivity is likely a consequence of secondary orbital interactions arising upon initial orientation of the addends (Fig. 2). These interactions hinder the formation of new isoxazoline bonds due to incompatability of the parity of the atomic orbitals of the phosphoryl group (LUMO) and nitrile group atoms (HOMO). The repulsion of these groups is also enhanced due to the symmetrical structure of tetracyanoethylene and the simultaneous participation of $C \equiv N$ bonds in secondary orbital interactions.

Analogous interactions arise in the case of aromatic nitrile oxides, for which incompatability of the parity of the atomic orbitals of the phenyl ring and nitrile groups is found.

EXPERIMENTAL

The ^{13}C NMR spectra were taken on a Bruker WM-250 Fourier spectrometer. The ^1H NMR spectra were taken on Varian T-60, Bruker WP-80, and Bruker WM-250 spectrometers. The ^{31}P NMR spectra were taken on a Bruker WP-80 spectrometer. The ^{13}C and ^1H NMR chemical shifts were determined relative to TMS. The phosphorus chemical shifts were determined relative to H_3PO_4 as an external standard. The IR spectra were taken on UR-20 and Bruker IFS-113V spectrometers with data accumulation and computer analysis.

The monitoring of the reaction course and purity of the products obtained were carried out by thin-layer chromatography on Silufol UV-254 and Alufol plates. Preparative chromatography was carried out on Merck-9385 silica gel (40-63 μm particle size) or Brockmann II alumina. The ratio of the adsorbent to compound ranged from 50:1 to 100:1. A mixture of ethyl acetate and hexane was used for elution. The quantum chemical calculations were carried out using the MOPAC 3.1 program.

Synthesis of the oxime of (diisopropoxyphosphoryl)carbonyl chloride was described in our previous work [18, 19].

Synthesis of 3-(Diisopropoxyphosphoryl)-4-R-5-phenyl-1,2,4-oxadiazolines IIIa-IIIc (general method). A solution of the corresponding benzylidenaniline (2.05-4.10 mmoles) and triethylamine (2.05-4.10 mmoles) in absolute benzene and a solution of the oxime of (diisopropoxyphosphoryl)carbonyl chloride (2.05-4.10 mmoles) in absolute benzene were added simultaneously dropwise to absolute benzene heated to reflux. The mixture was maintained at this temperature for 1 h and then cooled to 20°C. Preparative flash chromatography was carried out on silica gel or alumina.

3-(Diisopropoxyphosphoryl)-4,5-diphenyl-1,2,4-oxadiazoline (IIIa, $C_{20}H_{25}N_2O_4P$) was obtained from a solution of 0.50 g (diisopropoxyphosphoryl)carbonyl chloride oxime in 25 ml benzene and a solution of 0.29 ml triethylamine and 0.42 g benzylidenaniline in 25 ml benzene. The two solutions were added dropwise to 25 ml benzene at reflux. Preparative flash chromatography was carried out on alumina to give 0.20 g (22%) IIIa with a pronounced almond aroma. n_D^{20} 1.5108. PMR spectrum in $CDCl_3$: 7.24, 7.03 (10H, m, Ph), 6.37 [1H, s, $H_{(5)}$], 4.65 [2H, m, $\underline{CH}(CH_3)_2$], 1.28 ppm [12H, m, $\underline{CH}(CH_3)_2$]. ^{31}P NMR spectrum in CCl_4: 0.40 ppm (s). ^{13}C NMR spectrum in CCl_4: 149.5 [$C_{(3)}$, $^1J_{PC}$ 232.0], 138.9, 138.2, 130.1, 129.3, 128.1, 126.7, 125.9 (Ph), 99.0 [$C_{(5)}$, $^1J_{CH}$ 174.0], 73.2 [$\underline{C}H(CH_3)_2$), $^1J_{CH}$ 162.4], 24.7, 23.9 ppm [$CH(\underline{C}H_3)_2$, $^1J_{CH}$ 129.9]. IR spectrum: 3100-3000, 2990, 2940, 2880, 1740, 1710, 1650, 1605, 1555, 1505, 1468, 1390, 1280, 1210, 1180, 1150, 1110, 1060-970, 945, 840, 770 cm^{-1}.

3-(Diisopropoxyphosphoryl)-4-(4-bromophenyl)-5-phenyl-1,2,4-oxadiazoline (IIIb, $C_{20}H_{24}N_2O_4PBr$) was obtained from a solution of 1.00 g (diisopropoxyphosphoryl)carbonyl chloride oxime in 25 ml benzene and solution of 0.57 ml triethylamine and 1.07 g benzylidene-4-bromoaniline in 25 ml benzene. The two solutions were added dropwise into 25 ml benzene at reflux. Preparative flash chromatography was carried out on silica gel to give 0.46 g (24%) IIIb, n_D^{20} 1.5625. PMR spectrum in $CDCl_3$: 7.20-6.70 (9H, m, Ph), 6.25 [1H, s, $H_{(5)}$], 4.40 [2H, m, $\underline{CH}(CH_3)_2$], 0.80 ppm [12H, m, $CH(\underline{CH_3})_2$]. ^{31}P NMR spectrum in CCl_4: −3.00 ppm (s). IR spectrum: 3100-2800, 1600, 1550, 1500, 1470, 1385, 1280, 1250, 1185, 1150, 1110, 1080, 1060, 1030-1000, 945, 910, 830, 770, 705, 650, 600, 570 cm^{-1}.

3-(Diisopropoxyphosphoryl)-4-butyl-5-phenyl-1,2,4-oxadiazoline (IIIc, $C_{18}H_{29}N_2O_4P$) was obtained from a solution of 1.00 g (diisopropoxyphosphoryl)carbonyl chloride oxime in 25 ml benzene and a solution of 0.57 ml triethylamine and 0.66 g benzylidenebutylamine in 25 ml benzene. The two solutions were added dropwise to 25 ml benzene at reflux. Preparative flash chromatography was carried out on silica gel to give 0.23 g (15%) IIIc. PMR spectrum in $CDCl_3$: 7.34-7.30 (5H, m, Ph), 6.09 [1H, s, $H_{(5)}$], 4.84-4.65 [2H, m, $\underline{CH}(CH_3)_2$], 3.38-3.20 (1H, m, CH_2N), 3.10-2.95 (1H, m, CH_2N), 1.36, 135, 1.33, 1.32 [12H, d.d, $CH(CH_3)_2$], 1.19-1.17 (4H, m, $CH_2\underline{CH_2CH_2}CH_3$), 0.82-0.71 m.d (3H, m, $CH_2CH_2CH_2\underline{CH_3}$). ^{31}P NMR spectrum in CCl_4: −2.00 ppm (s).

2-Oxybenzylidenanil ester of (diisopropoxyphosphoryl)formhydroxamic acid (Va, E and Vb, Z isomers, $C_{20}H_{25}N_2O_5P$) was obtained according to the above procedure from a solution of 0.5 g (diisopropoxyphosphoryl)carbonyl chloride oxime in 25 ml benzene and a solution of 0.29 ml triethylamine and 0.40 g 2-hydroxybenzylidenaniline in 25 ml benzene. The two solutions were added dropwise to 25 ml benzene at reflux. Preparative flash chromatography on alumina gave 0.32 g (39%) of a 1:1 mixture of Va and Vb. PMR spectrum in $CDCl_3$: 8.57 (1H, s, CH=N), 7.45-6.55 (9H, m, Ph), 4.87 [2H, m, $\underline{CH}(CH_3)_2$], 1.25 ppm [12H, m, $CH(\underline{CH_3})_2$]. ^{31}P NMR spectrum in CCl_4: −1.33 (s), −3.14 ppm (s). ^{13}C NMR spectrum in CCl_4: 169.6, 161.0, 148.4, 132.7, 132.7, 132.3, 129.1, 126.5, 121.0, 118.6, 116.8 (Ph), 162.9 (CH=N, $^1J_{CH}$ 161.0), 73.7, [$\underline{CH}(CH_3)_2$], 23.8, 23.5, 23.2 ppm [$CH(\underline{CH_3})_2$]. IR spectrum: 3500-3000, 2990, 2935, 2880, 2600-2400, 1715, 1600, 1510, 1490, 1460, 1380, 1255, 1185, 1150, 1105, 1060-970, 959, 795, 765, 695, 610, 570 cm^{-1}.

1,2-Bis[3-(diisopropoxyphosphoryl)-1,2,4-oxadiazol-5-yl]-1,2-dicyanoethylene (VI, $C_{20}H_{28}N_6O_8P_2$) and Its Tetracyanoethylene π-Complex (VII, $C_{26}H_{28}N_{10}O_8P_2$). A solution of 2.25 g (10.38 mmoles) diisopropoxyphosphorylnitrile oxide [8] in 60 ml benzene at 0°C was mixed with a solution of 3.98 g (31.08 mmoles) tetracyanoethylene in 60 ml benzene. After 30 min, the reaction mixture was heated to reflux. The mixture was heated at reflux for 6 h. Preparative flash column chromatography was carried out on silica gel to give 0.04 g VI as colorless crystals with mp 185°C and 0.92 g bisadduct VII as a donor–acceptor complex. Product VI: ^{31}P NMR spectrum in $CDCl_3$: −3.37 ppm (s). Product VII: ^{31}P NMR spectrum in $CDCl_3$: −3.37 ppm (s).

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