Phosphorylformonitrile oxides as spin traps for carbon-centered free radicals: co-formation with radicals in the generation of nitrile oxides from *C*-phosphorylformohydroximoyl halides in the presence of alcohols or ethers

V. I. Morozov, B. I. Buzykin, and A. V. Il´yasov*

A. E. Arbuzov Institute of Organic and Physical Chemistry,
Kazan Research Center of the Russian Academy of Sciences,
8 ul. Akad. Arbuzova, 420088 Kazan, Russian Federation.
Fax: +7 (843 2) 73 2253. E-mail: buz@iopc.knc.ru

One-electron oxidation of the oximes $R_2P(=O)C(=NOH)X$ (X = Cl or Br) generates the nitrile oxides $R_2P(=O)C^+=NO^-$, which serve as spin traps for unstable carbon-centered radicals. The latter are generated upon addition of PbO_2 to a mixture of formohydroximoyl halide with an alcohol or an ether of the general formula $R^1OCHR^2R^3$ under the action of atomic chlorine (bromine) released during the generation of nitrile oxide. This gives rise to new, more persistent *C*-phosphoryliminoxyls $R_2P(=O)C(=NO^{\bullet})C(OR^1)R^2R^3$ (R^1, R^2, R^3 = H, Alk). When primary alcohols ($R^1 = R^2 = H$) are used, acyl radicals generated at the initial step of the reaction are also trapped by nitrile oxides to give *C*-acyl-*C*-phosphoryl iminoxyl radicals $R_2P(O)C(=NO^{\bullet})C(=O)R^3$. Hyperfine coupling constants for more than 20 *C*-phosphoryliminoxyls existing in solutions as mixtures of *Z*- and *E*-isomers were determined. The effect of the structure of the primary radical (length of the carbon chain, degree of branching, the presence of a ring, and its size) on the radiospectroscopic characteristics of new *C*-phosphoryliminoxyl radicals was studied.

Key words: electron spin resonance, spin traps, iminoxyls, *C*-phosphorylated iminoxyl radicals, *C*-phosphorylated nitrile oxides, *C*-phosphorylated oximes, hydroximoyl halides, geometrical isomerism of iminoxyls, oxidation of oximes.

Electron spin resonance (ESR) spectroscopy is an efficient method of studying free radicals in solutions. However, the short lifetimes (10^{-5}—10^{-10} s) of radicals prevent them from being detected directly. This obstacle has been surmounted by adding a specially selected diamagnetic reagent (spin trap) to a short-lived radical to form a new, more stable radical (so-called spin adduct). Analysis of the spectroscopic characteristics of spin adducts provides information on both the amount and structures of primary radicals.

Current spin traps are representatives of more than 15 classes containing the corresponding functions (nitroso-, nitro, imino, azo, oxo, thioxo, nitrone, nitrile oxide, and other groups).[1—3] Among them, nitroso compounds (R—N=O) and nitrones (R´—CH=N^+(—O^-)R), including phosphorylated ones, are most commonly used.[3,4] The resulting spin adducts are nitroxyl radicals R—N(—$O^{\bullet}$)—R″. Among other potential spin traps, nitrile oxides R—C≡N^+—O^-, which are classified as 1,3-dipolar reagents, seem to be of the greatest interest.[5,6] Their spin adducts are iminoxyl radicals. It is known that carbon-centered radicals mainly attack the azomethine C atom (also in nitrile oxides[1—3]), while adducts *via* the imine N atom of imines are detected only in rare cases.[7]

Comparison of nitrones and nitrile oxides as spin traps reveals the superiority of the latter in many cases. First, the presence of *Z*- and *E*-isomers of iminoxyls extends information taken from ESR spectra; second, dimerization of iminoxyls sometimes makes it possible to increase the radical lifetime and the experimental temperature, which is important in the study of viscous solutions and biological fluids; third, the a_N values of iminoxyl radicals are higher than those of nitrones; fourth, the ESR spectra of iminoxyls show narrower lines (~0.2 Oe) and the anisotropic contribution to their broadening is smaller, which is also significant in the study of radicals with high molecular masses characteristic of biological units.

Iminoxyl radicals of the general formula R(R´)C=N—$O^{\bullet}$ constitute a class of sufficiently long-lived σ-radicals. Like the starting oximes, iminoxyls are

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 2, pp. 336—341, February, 2005.

1066-5285/05/5402-0342

known to exist in solutions as two geometrical isomers. The energy barrier between their *Z*- and *E*-isomers is smaller than the barrier between the isomers of the corresponding oximes; for this reason, in the oxidation of oximes, an equilibrium mixture of the *Z*- and *E*-isomers of iminoxyl radicals is never dependent on the isomeric composition of the starting oxime (one of its isomers or their mixture of any composition).[2,3,8]

The HFC constants of oximes with N- and P-containing substituents are highly stereospecific because of the magnetic properties of the nuclei of these elements, which allows reliable determination of the ratio of the *Z*- and *E*-isomers of *C*-phosphoryliminoxyls.[3]

Taking into account that *C*-phosphorylated nitrile oxides have been studied well enough[6,9–11] and that some of them are stable under normal conditions,[11–14] they may be considered to be promising for use as spin traps. Our source of *C*-phosphoryl nitrile oxides was *C*-phosphorylformohydroximoyl halides $Y_2P(=O)C(X)=NOH$ containing alkoxy, dialkylamino, or morpholinyl fragments at the phosphoryl group (Y = MeO, EtO, PrO, Pr^{i}O, Et_2N, or $O(CH_2CH_2)_2N$; X = Cl, Br, or I).[15,16]

Here, we published the first results of the investigation of spin trapping with the use of *C*-(diisopropoxyphosphoryl)formohydroximoyl chloride (**1a**). Oxime **1a** and its bromine analog (bromide **1b**) exist in the crystal[16] and in solutions as the $Z_{C=N},E_{N-O}$-isomer (in terms of the Geneva nomenclature, this is the *anti*-isomer with respect to the P=O group). This isomer is also more stable for *C*-phosphorylated amide oximes synthesized from oxime **1a** and containing dialkylamino groups or morpholinyl, piperidinyl, or benzotriazolyl residues in the amide fragment.[17]

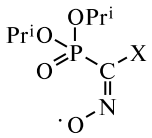
Unstable carbon-centered radicals were generated from aliphatic and cyclic alcohols and from dialkyl and cyclic ethers. The weakest C—H bond in alcohols and ethers is known[18] to be at the α-C atom. To reveal electronic and structural effects, we used linear, branched, and cyclic compounds containing methine or methylene protons activated by the neighboring hydroxy or alkoxy group, namely, alcohols **2a—j** and ethers **2k—n**.

Experimental

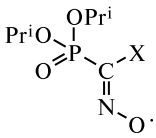
C-(Diisopropoxyphosphoryl)formohydroximoyl chloride (**1a**) was prepared as colorless crystals according to known procedures,[15,16] m.p. 68—69 °C (Boetius hot stage). Alcohols **2a—j** and ethers **2k—n** were purified according to known procedures.[19,20]

Spin adducts were synthesized as follows: a tube containing compound **1a** in CH_2Cl_2 ($5\cdot10^{-2}$—$5\cdot10^{-3}$ mol L^{-1}) was charged with an alcohol or ether of interest (1—10 vol.%) and then PbO_2 was added (10 to 20-fold excess with respect to the starting oxime **1a**). ESR spectra were recorded on a Radiopan SE/X-2544 spectrometer at 293 K. For rapid reactions, measurements were performed at low temperatures (down to 203 K). The ESR parameters of the detected iminoxyl radicals are given in Tables 1 (HFC constants with the N and P nuclei) and 2 (HFC constants with the protons). Their ESR spectra are shown in Figs 1 and 2.

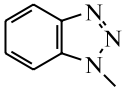
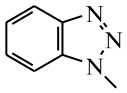
Table 1. HFC constants with the N (a_N/Oe) and P atoms (a_P/Oe) for the *syn*- and *anti*-isomers of *C*-phosphoryliminoxyl radicals



syn-Isomer



anti-Isomer

Radical	X	*syn*-Isomer[a]		*anti*-Isomer[a]	
		a_N	a_P	a_N	a_P
4 [b]	Cl	—	—	31.6	1.5
5	$(Pr^iO)_2P=O$	32.8	55.5	32.8	14.75
7a	$HOCH_2$—	30.60	56.20	32.10	9.62
7b—f	HOCH(R)—	~30.5	~55	~32	~10.6
7g	HOCH(Bu^i)—	~30.3	~56.5	~32.0	~10.3
7h—j	$HOC(R_2)$—	~30.5	~58.0	~32.0	~13.0
7k—n	R´OCH(R)—	~31.2	~54.5	~31.5	~10.2
10a—f	RC(=O)—	~30.0	~54.0	~31.5	~9.4
11		28.7	39.8	32.3	0.4
12 [c]		28.2	29.5	32.7	0.6

[a] The isomers are designated according to the Geneva nomenclature with respect to the phosphoryl group because of changing priority of the substituents X.
[b] $a_{^{13}C}$ = 52.0 Oe.
[c] (Dimorpholinophosphoryl)benzotriazolyliminoxyl radical.

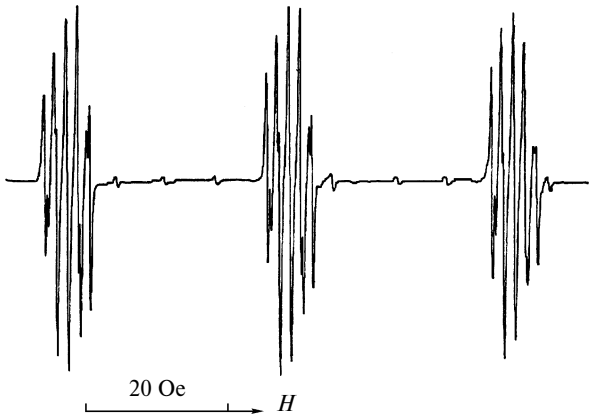
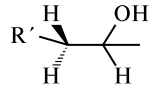
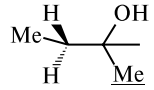
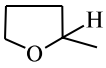
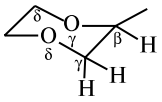


Fig. 1. ESR spectrum of a 0.01 *M* solution of iminoxyl radical **4** in CH_2Cl_2.

Table 2. HFC constants of the protons of the substituent R (a_H/Oe) in radicals **7a—n** and **10b—g** ((Pr^{i}O)$_2$P(=O)—C(R)=N—O$^\bullet$)

Radical[a]	R[b]	a_H^β			a_H^γ			a_H^δ	
		n	*Z*-isomer[c]	*E*-isomer[d]	*n*	*Z*-isomer[c]	*E*-isomer[d]	*n*	*E*-isomer[d]
7a	$HOCH_2$—	2	1.1	0.4	—	—	—	—	—
7b	MeCH(OH)—	1	1.2	—	3	—	1.1	—	—
7c—f[e]		1	1.2	3.2	1[f]	—	1.2—1.3	—	—
		—	—	—	1[f]	—	0.6—1.0	—	—
7g	$Me_2CHCH_2CH(OH)$—	1	1.1	3.2	1	—	1.1	—	—
7h	$Me_2C(OH)$—	—	—	—	6	—	0.7	—	—
7i		—	—	—	1[g]	—	1.8	—	—
		—	—	—	3	—	0.8	—	—
7j		—	—	—	2	0.5	—	—	—
7k	$MeCH_2OCH(Me)$—	1	1.2	4.5	3	—	1.0	2	0.3
7l	$MeOCH_2CH(OMe)$—	1	1.1	3.7	2	—	0.8	—	—
7m		1	1.1	3.0	2	—	1.1	—	—
7n		1	—	2.1	2	1.0	—	—	—
		—	—	—	1[g]	—	0.5	—	—
10b	MeC(=O)—	—	—	—	3	0.4	—	—	—
10c	$MeCH_2C(=O)$—	—	—	—	2	0.5	0.3	3	0.4
10d,e[h]	$R'CH_2CH_2C(=O)$—	—	—	—	2	0.5	0.3	2	0.4
10f	$Me_2CHC(=O)$—	—	—	—	1	0.8	0.9	6	0.2
10g	$Me_2CHCH_2C(=O)$—	—	—	—	2	0.5	0.4	1	0.5

[a] In radicals **7** and **10**, the phosphoryl group is a substituent of priority.
[b] The C atoms in the carbon chain R, to which the respective protons are attached (*n* is the number of the protons), are designated in the usual order: $^\bullet ON{=}C_\alpha{-}C_\beta{-}C_\gamma{-}C_\delta{-}$ (R is $C_\beta{-}C_\gamma{-}C_\delta$).
[c] *syn*-Isomer.
[d] *anti*-Isomer.
[e] R′ = Me (**c**), Et (**d**), Pr (**e**), and Pr^i (**f**).
[f] The enantiotopic protons.
[g] One of the enantiotopic protons.
[h] R′ = Me (**d**) and Et (**e**).

Results and Discussion

Like other hydroximoyl halides,[5,6] *C*-phosphoryl-formohydroximoyl halides in the presence of bases release HCl without cleavage of the C—P bond[9—11,15—17] to give sufficiently stable *C*-phosphoryl nitrile oxides.[9,10] For instance, *C*-(diisopropoxyphosphoryl)formonitrile oxide (**3**) obtained from oximes **1a,b** and triethylamine (Scheme 1) is persistent at 10 °C,[9—11] while *C*-(dimorpholinophosphoryl)formonitrile oxide persists for a long period of time (> 3 years) even at 20 to 25 °C.[12—14]

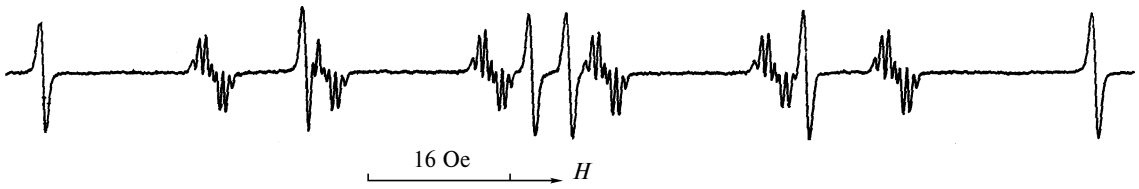
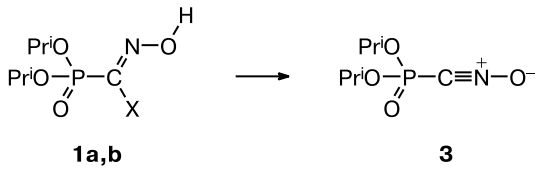


Fig. 2. ESR spectrum of radical **7i** in CH_2Cl_2—butan-2-ol (10 vol.%).

Scheme 1



1a,b **3**

X = Cl (**a**), Br (**b**)

We assumed that *C*-phosphorylformonitrile oxides would also be generated in the oxidation of *C*-phosphorylformohydroximoyl halides by analogy with the formation of nitrile imines from hydrazones of acyl halides (hydrazonoyl halides).[21] In this case, *C*-phosphorylformohydroximoyl halides (**1a,b** *etc.*) would happily combine the properties of spin trap precursors (as sources of nitrile oxide) and of initiators of radical processes (through generation of a halogen atom upon oxidation). For instance, a Cl atom generated in a reaction mixture gives rise to carbon-centered radicals that react with a spin trap. In our experiments, such radicals were generated from the following alcohols and ethers: methanol (**2a**), ethanol (**2b**), propanol (**2c**), butanol (**2d**), pentanol (**2e**), 2-methylpropanol (**2f**), 3-methylbutanol (**2g**), propan-2-ol (**2h**), butan-2-ol (**2i**), cyclohexanol (**2j**), diethyl ether (**2k**), ethylene glycol dimethyl ether (**2l**), tetrahydrofuran (**2m**), and dioxane (**2n**).

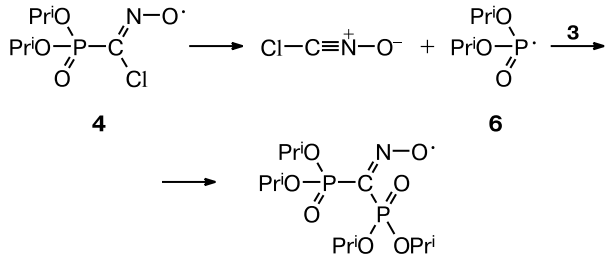
The one-electron oxidation of oxime **1a** in solvents that are inert to homolytic processes yields iminoxyl **4**; its ESR parameters (see Table 1, Fig. 1) are typical of iminoxyl radicals (usually, $a_N \approx 32.0$ Oe, $a_{Cl} \approx 1.5$ Oe).[3] It should be noted that the ESR spectra of radical **4** show signals only for the *Z*-isomer (*anti*-isomer with respect to the phosphoryl group). Such a pattern is very uncommon for iminoxyl radicals. For radical **4**, the HFC constant with the nucleus of the P atom in the β-position relative to the radical center is low ($a_P \approx 1.5$ Oe), though it is usually 6 to 14 Oe. At the same time, the HFC constant with the ^{13}C nucleus is 52 Oe, which is double the corresponding value for other iminoxyl radicals. In our case, it approaches the HFC constants with the ^{13}C nucleus, which are characteristic of carbon-centered radicals of the type $R^1R^2R^3C^{\bullet}$, including cycloalkyl radicals (30—100 Oe) and vinyl radicals $R_2C{=}(R)C^{\bullet}$ (100—150 Oe).[22] This fact suggests that iminoxyl **4** can exist in the ground state as resonance structures with an unpaired electron at the C atom of the imine group rather than those with an unpaired electron localized only at the σ-orbital of the N—O bond, which is orthogonal to the π-orbital of the C=N bond, as believed hitherto.[3]

The stability of the *Z*-isomer of radical **4** generated from compound **1a** agrees with the known data[23] on the exclusive formation of the *Z*-isomers of arylchloro iminoxyls ArC(Cl)=NO$^{\bullet}$ from oximes of aroyl chlorides ArC(Cl)=NOH.

The low HFC constants with the P atom were reported earlier[10,17] for phosphorylformoiminoxyl radicals in which substituents at the imine C atom exhibit σ,π-donating properties: dialkylamino groups and morpholinyl, piperidinyl, or benzotriazolyl fragments (see Table 1, compounds **11** and **12**).

Reactions in solvents inert to homolytic processes (*e.g.*, in benzene, CH_2Cl_2, CCl_4, *etc.*) yield, apart from iminoxyls of the type **4**, di(phosphoryl) iminoxyl radicals, *e.g.*, *C*,*C*-bis(diisopropoxyphosphoryl)formoiminoxyl (**5**). The formation of such radicals could be attributed to possible fragmentation of iminoxyl **4** releasing not only the Cl atom (to give phosphoryl nitrile oxide **3**) but also phosphoryl radical **6**, which reacts with the spin trap (Scheme 2).

Scheme 2



4 **6** **5**

However, since cleavage of the C—P bond in reactions of the starting oxime **1a** has not been noted earlier,[9,15] radicals **5** must have formed in other ways, *e.g.*, through the formation of dialkyl phosphonates $HP(=O)(OR)_2$, which are very easy to oxidize homolytically into dialkoxyphosphoryl radicals like radical **6**.

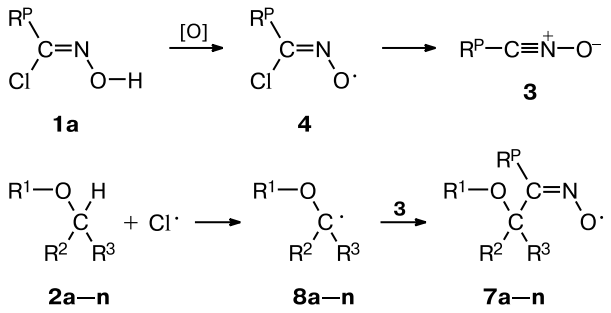
Addition of alcohols (**2a—j**) or ethers (**2k—n**) to the reaction mixture afforded new phosphorylated iminoxyls **7a—n** (ESR data), which are adducts of carbon-centered free radicals **8a—n** with the *in situ* generated spin trap (**3**). The occurring processes can be represented by Scheme 3.

The ESR spectrum of radical **7i** in CH_2Cl_2—butan-2-ol (10 vol. %) is shown in Fig. 2 as an example.

In the case of alcohols **2b—g** containing the α-methylene group, primary radicals **8b—g** further oxidized to acyl radicals **9b—g** (Scheme 4), which added *in situ* to phosphorylformonitrile oxide **3** to give *C*-acylated phosphorylformiminoxyl radicals **10b—g** (see Table 2). With methanol, no corresponding radical **10a** (R = H) was detected.

It should be emphasized that the lengthening of the alkyl chain in the attached fragments of radicals **7b—e** and in the acyl fragments of radicals **10b—e** by more than

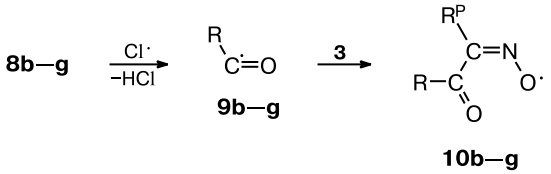
Scheme 3



$R^P = (Pr^iO)_2P(=O)$—

$R^1 = R^2 = H$,
R^3 = H (**a**), Me (**b**), Et (**c**), Pr (**d**), Bu (**e**), Pr^i (**f**), Bu^i (**g**);
R^1 = H, R^2 = Me, R^3 = Me (**h**), Et (**i**); R^1 = H, $R^2 + R^3 = (CH_2)_5$ (**j**);
R^1 = Et, R^2 = H, R^3 = Me (**k**); R^1 = Me, R^2 = H, $R^3 = CH_2OMe$ (**l**);
$R^1 + R^2 = (CH_2)_3$, R^2 = H (**m**); $R^1 + R^2 = (CH_2)_2OCH_2$, R^3 = H (**n**)

Scheme 4



$R^P = (Pr^iO)_2P(=O)$—

R = Me (**b**), Et (**c**), Pr (**d**), Bu (**e**), Pr^i (**f**), Bu^i (**g**)

two CH_2 groups no longer affects the radical center (*cf.* the parameters of radicals **7b**—**e** or **10b**—**e** in Table 2). Branching of the alkyl chain (**7f**) is also ineffective. That is why the radiospectroscopic characteristics of radicals **7c**—**f** (**10d,e**) are approximately equal (see Table 2).

Analysis of the data in Table 2 revealed some interesting facts: (1) the difference in the HFC constants a_H^β for the β-protons in the *Z*- and *E*-isomers of radicals **7**; (2) the constancy of the a_H^β values (1.1—1.2 Oe) in the *Z*-isomers (*syn*-isomers with respect to the P=O group) of radicals **7a**—**n**, which can be used for analytical purposes in the study of related iminoxyls; (3) a wide range of the a_H^β values (0.4—4.5 Oe) in the *E*-isomers (*anti*-isomers with respect to the P=O group) of radicals **7**; (4) close HFC constants for the γ-protons in the *Z*-isomers of cyclic iminoxyl radicals **7j,n** (probably, because of conformation equilibrium); (5) the diastereotopy of the methylene protons of radicals **7c**—**f,i,n** for the γ-protons in the *E*-isomer, which is due to the presence of the asymmetric oxygen-containing group. Detection of only one axial proton for radicals **7i,n** is explained by conformation equilibrium.

Presumably, use of alcohols or ethers containing optically active fragments can allow construction of molecular systems acting either as a "key" (according to Fisher) for natural receptors or as a "lock" for supply of radicals of certain configurations to the reactive site. Practical application of this approach will make it possible to assess the usefulness of nitrile oxides as spin traps for natural radicals (in particular, those generated from hydrocarbons).

We are grateful to M. P. Sokolov for samples of oxime **1a** and to A. A. Vafina for her assistance in our ESR studies.

This work was financially supported by the Foundation of Research and Development Work of the Tatarstan Republic and the Tatarstan Republic Academy of Sciences (Joint Grant 07-7.2-76/2002).

References

1. V. E. Zubarev, *Metod spinovykh lovushek. Primenenie v khimii, biologii i meditsine* [*Spin Trap Method. Applications in Chemistry, Biology, and Medicine*], MGU, Moscow, 1985, 178 pp. (in Russian).
2. V. E. Zubarev, *Spinovyi zakhvat v khimii i biologii* [*Spin Trapping in Chemistry and Biology*], in *Nitroksil´nye radikaly. Sintez, khimiya, prilozhenie* [*Nitroxyl Radicals. Synthesis, Chemistry, Applications*], Eds E. G. Rozantsev and R. I. Zhdanov, Nauka, Moscow, 1987, 144 (in Russian).
3. A. V. Il´yasov, I. D. Morozova, A. A. Vafina, and M. B. Zuev, *Spektry EPR i stereokhimiya fosforsoderzhashchikh svobodnykh radikalov* [*ESR Spectra and the Stereochemistry of Phosphorus-Containing Free Radicals*], Nauka, Moscow, 1985, 176 pp. (in Russian).
4. F. Chalier and P. Tordo, *J. Chem. Soc., Perkin Trans. 2*, 2002, 2110.
5. R. Huisgen, *Angew. Chem.*, 1963, **75**, 604; 742.
6. *1,3-Dipolar Cycloaddition Chemistry,* Vol. **1**, Ed. A. Padva, J. Wiley and Sons, New York, 1983, 817 pp.
7. G. K. Friested, *Tetrahedron*, 2001, **57**, 5461.
8. S. Berski, A. R. Jaszewski, and J. Jezierska, *Chem. Phys. Lett.*, 2001, **341**, 168.
9. B. I. Buzykin and M. P. Sokolov, *Zh. Obshch. Khim.*, 1992, **62**, 2266 [*Russ. J. Gen. Chem.*, 1992, **62** (Engl. Transl.)].
10. T. A. Zyablikova, B. I. Buzykin, A. S. Dokuchaev, M. P. Sokolov, R. M. Gainullin, and M. V. Petrov, in *Fiziko-khimicheskie metody issledovaniya struktury i dinamiki molekulyarnykh sistem (Materialy Vserossiiskogo soveshchaniya, Yal´chik, 1994)* [*Physicochemical Methods for the Study of the Structures and Dynamics of Molecular Systems (Proc. All-Russia Conf., Yalchik, 1994)*], Mariiskii Pedagogicheskii Inst., Ioshkar-Ola, 1994, 94 (in Russian).
11. V. A. Pavlov, N. V. Aristova, and B. I. Gorin, *Zh. Obshch. Khim.*, 1995, **65**, 1991 [*Russ. J. Gen. Chem.*, 1995, **65** (Engl. Transl.)].
12. A. S. Dokuchaev, B. I. Buzykin, M. P. Sokolov, and V. F. Sopin, *Izv. Akad. Nauk, Ser. Khim.*, 1994, 1245 [*Russ. Chem. Bull.*, 1994, **43**, 1180 (Engl. Transl.)].

13. T. A. Zyablikova, B. I. Buzykin, and M. P. Sokolov, *Zh. Obshch. Khim.*, 1995, **65**, 266 [*Russ. J. Gen. Chem.*, 1995, **65** (Engl. Transl.)].
14. B. I. Buzykin, T. A. Zyablikova, and M. P. Sokolov, *Abstrs, XIII Int. Conf. on Phosphorus Chemistry — ICPC*, Jerusalem, 1995, 222.
15. M. P. Sokolov, B. I. Buzykin, and V. A. Pavlov, *Zh. Obshch. Khim.*, 1990, **60**, 223 [*J. Gen. Chem. USSR*, 1990, **60** (Engl. Transl.)].
16. R. R. Shagidullin, V. A. Pavlov, B. I. Buzykin, N. V. Aristova, L. F. Chertanova, I. V. Vandyukova, A. Kh. Plyamovatyi, K. M. Enikeev, M. P. Sokolov, and V. V. Moskva, *Zh. Obshch. Khim.*, 1991, **61**, 1590 [*J. Gen. Chem. USSR*, 1991, **61** (Engl. Transl.)].
17. B. I. Buzykin, V. I. Morozov, M. P. Sokolov, T. A. Zyablikova, and A. V. Il´yasov, *Abstrs, 1 Vserossiiskaya konferentsiya po khimii geterotsiklov pamyati A. N. Kosta* [*1st All-Russia Conf. on Heterocyclic Chemistry in Memory of A. N. Kost*], Suzdal, 2000, 118 (in Russian).
18. S. Ya. Pshezhetskii, A. G. Kotov, V. K. Milinchuk, V. A. Roginskii, and V. I. Tupikov, *EPR svobodnykh radikalov v radiatsionnoi khimii* [*ESR of Free Radicals in Radiation Chemistry*], Khimiya, Moscow, 1972, 208 (in Russian).
19. *Organic Solvents*, Ed. A. Weissberger, Interscience, New York, 1955.
20. A. J. Gordon and R. Ford, *The Chemist´s Companion*, J. Wiley and Sons, New York, 1972.
21. M. K. Kadirov, A. V. Il´yasov, A. A. Vafina, Yu. P. Kitaev, B. I. Buzykin, and N. G. Gazetdinova, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1980, 1616 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1980, **29**, 1159 (Engl. Transl.)].
22. Landolt—Börnstein, *Numerical Data and Functional Relationships in Science and Technology. New Series, Group II. Vol. 17. Magnetic Properties of Free Radicals*, Springer-Verlag, Berlin, 1987, S. 584.
23. A. Alberti, G. Barbaro, A. Battaglia, and M. Guerra, *J. Org. Chem.*, 1981, **46**, 742.

Received September 30, 2003;
in revised form December 22, 2003