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α-Hydroxy- and (α-Acyloxyimino)benzylphosphonates

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Abstract—*N*-Hydroxyfluorobenzimidoylphosphonates and their *O*-acyl derivatives are synthesized. Reaction of phosphorylated oximes with sulfinyl chloride proceeds with rearrangement and leads to synthetically prospective *N*-sulfonylimidoylphosphonates. By the method of ^{19}F NMR are revealed values of σ-constants of *N*-hydroxy- and *N*-acyloxy substituted imidoylphosphonate groups.

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α-Hydroxyiminoalkylphosphonates is a prospective type of bifunctional organophosphorus compounds. They are used in the syntheses of amino- [1,2] and hydroxyaminophosphonic acids [3], have been proposed as phosphorylating agents via generation of metaphosphates [4], are potent metal-binding agents [4]. (α-Acyloxy imino)benzylphosphonates have been patented as herbicide antagonists [5]. Synthetic potential of C-phosphorylated oximes has not been sufficiently studied, the transformations described are connected mainly with *E/Z* isomerization, thermal fragmentation and dealkylation of the phosphorus-containing fragment [4]. Reaction at azomethine group are restricted to the reduction of C=N bond, due probably to insufficient electrophilicity of imine carbon atom. We suggested that introduction of substituents to the hydroxy group oxygen atom in *N*-hydroxyl-benzimidoylphosphonate could increase electrophilicity of azomethine bond and might reveal new pathways of reactions of these compounds.

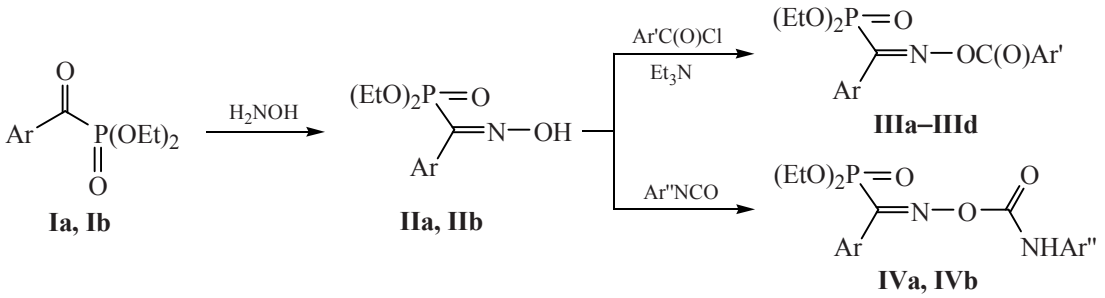
In this work we synthesized *N*-hydroxyfluorobenz-imidoylphosphonates and their *O*-acyl derivatives, analyzed electronic structure of *R*-oxyimidoylphos-phonate groups and studied some properties of the synthesized C-phosphorylated imines.

Reaction of fluorobenzoylphosphonates **Ia** and **Ib** with hydroxylamine hydrochloride in the presence of pyridine results in formation of C-phosphorylated oximes **IIa** and **IIb** formed as mixtures of *E/Z* isomers (*E/Z* ratio is 2.3 and 2.1, respectively) (Scheme 1). Assign-ment of the isomers is made on the basis of the data [4]. In ^{31}P, ^{19}F NMR spectra of compounds **II** the resonance of *E* isomers occur at weaker field ($\Delta\delta_P$ 3.4–3.5 ppm, $\Delta\delta_F$ 0.5–1.4 ppm) as compared with the *Z* isomers. The oximes obtained are readily acylating with acid chlorides or isocyanates. Thus, the reaction of compounds **II** with aromatic acid chlorides in the presence of a base leads to imines **III** and with 3,4-dichlorophenylisocyanate are obtained derivatives **IV** containing fragments of carbaminic and imidoylphos-phonic acids (Scheme 1).

Distinctly to the reactions in the scheme 1 leading to stable O-acylated derivatives, the reaction of phosphorylated oximes **II** with 4-tolylsulfinyl chloride proceeds with rearrangement of the primary products of O-sulfination (compare [6]) and leads to *N*-sulfonyl-imidoylphosphonates (**V**) (Scheme 2). This reaction is a new convenient method for the synthesis of C-phosphorylated *N*-sulfonylimines that are pros-pective reactive precursors of aminophosphonic acids [7, 8].

Magnetic shielding of fluorine nuclei in substituted fluorobenzenes depends on the electronic parameters of substituents at the benzene ring and is widely used for quantitative revealing the latter [9]. By measuring chem-ical shifts of fluorine nuclei relatively to internal fluoro-benzene in imidoylphosphonates **II–IV** dissolved in $CDCl_3$ and applying Taft's equations [10, 11] we for first time revealed inductive and resonance σ-constants of *N*-hydroxy-, *N*-acyloxyimidoylphosphonate {–C[P(O)·$(OEt)_2$]=NOX} and phosphorylformyl {–C[P(O)·$(OEt)_2$]=O} groups.

Scheme 1.



Ia, Ib **IIa, IIb** **IIIa–IIId** **IVa, IVb**

I, II: Ar = 4-FC_6H_4 (**a**), 3-FC_6H_4 (**b**); **III**: Ar' = 4-$O_2NC_6H_4$, Ar = 4-FC_6H_4 (**a**), 3-FC_6H_4 (**b**); Ar' = 4-ClC_6H_4, Ar = 4-FC_6H_4 (**c**), 3-FC_6H_4 (**d**); **IV**: Ar'' = 3,4-$Cl_2C_6H_3$, Ar = 4-FC_6H_4 (**a**), 3-FC_6H_4 (**b**).

Scheme 2.

Va, Vb

As follows from the obtained data (see table), *N*-hydroxyimidoylphosphonate group (no. 2) is a weak electron-acceptor (σ_p 0.15–0.25) even below *N*-benzyl (no. 9) analog by total electron-acceptor effect, that is reflected by the observed low reactivity of the phosphorylated oximes. Attaching of acyl (nos. 4–6) or carbamoyl (nos. 7, 8) group to the oxime O-atom increases acceptor property substantially: total electron-acceptor effect of these groups becomes comparable with that of such strong acceptors as *N*-sulfonyl (no. 10) or *N*-phosphorylimidoyl substituents (no. 11). Interesting to note that electronic effect

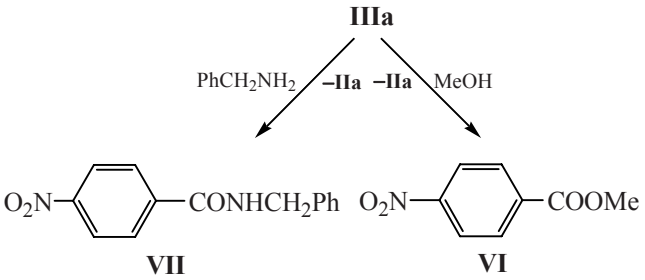
NMR chemical shifts of fluorosubstituted benzenes XC_6H_4F and σ-constants of N-substituted imidoylphosphonate groups

Run no.	X	$\delta^F(XC_6H_4F)$		σ_I	σ_R	σ_p
		3-F	4-F			
1	O=C[P(O)(OEt)$_2$]–	2.23	11.72	0.40	0.32	0.72
2	(*E*)-HON=C[P(O)(OEt)$_2$]–	0.69	2.74	0.18	0.07	0.25
3	(*Z*)-HON=C[P(O)(OEt)$_2$]–	0.21	1.35	0.11	0.04	0.15
4	(*E*)-4-ClC_6H_4COON=C[P(O)(OEt)$_2$]–	1.92	4.88	0.35	0.10	0.45
5	(*Z*)-4-ClC_6H_4COON=C[P(O)(OEt)$_2$]–	1.17	4.34	0.25	0.11	0.36
6	(*E*)-4-$O_2NC_6H_4$COON=C[P(O)(OEt)$_2$]–	2.18	5.45	0.39	0.11	0.50
7	(*E*)-3,4-$Cl_2C_6H_3$NHCOON=C[P(O)(OEt)$_2$]–	2.18	5.38	0.39	0.11	0.50
8	(*Z*)-3,4-$Cl_2C_6H_3$NHCOON=C[P(O)(OEt)$_2$]–	1.41	4.33	0.28	0.10	0.38
9	$PhCH_2$N=C[P(O)(OEt)$_2$]–	1.80	2.38	0.34	0.02	0.36 [12]
10	$PhSO_2$N=C[P(O)(OEt)$_2$]–	2.09	7.42	0.38	0.18	0.56 [13]
11	TsN=C[P(O)(OEt)$_2$]–	1.90	7.10	0.35	0.18	0.53
12	$(EtO)_2$P(O)N=C[P(O)(OPh)$_2$]–	2.05	7.42	0.37	0.18	0.55 [8]

depends on the geometry of imidoylphosphoryl group: in all cases the electron–acceptor ability is higher at *E* configuration of substituents at the azomethine bond. From the analysis of the data in the table follows that electron–acceptor property of oxo- or iminomethylphosphoryl group $(EtO)_2P(O)C(=X)–$ raises in the following series, depending on the unsaturated substituent X: NOH < NCH_2Ph < NOC(O)R < $NP(O)OEt)_2$ < NSO_2Ph < O.

The obtained values of σ-constants (see table) show that O-acylated imidoylphosphonates **III** are enough electrophilic systems. Actually, in distinct to the phosphorylated oximes **II** they react with O- and N-centered nucleophiles under mild conditions. Reactions with methanol or benzylamine proceed at the most electrophilic center, carbonyl group, and leads to formation of oxime **IIa** and methyl ester **VI** or benzylanide **VII**, respectively (Scheme 3). Thus, compounds **III** are soft acylating agents.

Scheme 3.



The *N*-sulfonylimidoylphosphonates (**V**) prepared from the oximes (Scheme 2) are even stronger electrophiles (see table, no. 11). Unlike the O-acylated compounds **III**, they react with O-, S- and P-centered nucleophiles at the C=N bond forming functional aminophosphonic acid derivatives [8, 13].

Thus, modification of the substituent at the oxygen atom of hydroxyimidoylphosphonate allows to increase substantially its electrophilicity and to change reactivity of the corresponding C-phosphorylated imines.

EXPERIMENTAL

The IR spectra were registered on an UR–20 spectrophotometer. The ^{1}H NMR spectra were recorded on a NMR spectrometer Varian VXR-300, operating frequency 299.95 MHz, the ^{19}F and ^{31}P NMR spectra on a Varian Gemini–200 instrument, operating frequencies 188.28 and 81.03 MHz, respectively. Chemical shifts are given relatively to internal references, TMS (^{1}H), $CFCl_3$ or PhF (^{19}F), and external 85% H_3PO_4 (^{31}P). All reactions were carried out under anhydrous conditions, under argon atmosphere.

***N*-Hydroxyimidoylphosphonates (II).** A mixture of 4 mmol of ketophosphonate **I**, 5.2 mmol of hydroxylamine hydrochloride and 5.6 mmol of pyridine in 2 ml of anhydrous ethanol was stirred at room temperature for 36 h. The solvent was evaporated and residue was washed with water, extracted with ether, and dried over $MgSO_4$. After evaporation of solvent was isolated compound **II**.

Diethyl *N*-hydroxy-4-fluorobenzimidoylphosphonate (IIa). Yield 72%, oil. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.21 (*E*) and 1.22 (*Z*), overlapping triplets (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.0–4.2 m (4H, CH_2), 7.0 m (2H, Ar), 7.6 m (2H, Ar), 10.56 br (*E*), 11.81 br (*Z*). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –112.89 (*E*), –113.37 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 9.4 (*E*), 6.0 (*Z*), *E*/*Z* ~ 2.1. Found, %: N 4.86; P 10.94. $C_{11}H_{15}FNO_4P$. Calculated, %: N 5.09; P 11.25.

Diethyl *N*-hydroxy-3-fluorobenzimidoylphosphonate (IIb). Yield 68%, oil. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.21 (*E*) and 1.22 (*Z*), overlapping triplets (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.0–4.2 m (4H, CH_2), 7.0 m (1H, Ar), 7.2 m (3H, Ar), 10.9 br (*E*), 12.1 br (*Z*). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –112.89 (*E*), –113.37 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 8.5 (*E*), 5.0 (*Z*). *E*/*Z* ~ 2.2. Found, %: N 4.78; P 10.80. $C_{11}H_{15}FNO_4P$. Calculated, %: N 5.09; P 11.25.

***N*-Acyloxy imidoylphosphonates (III)**. To a solution of 1 mmol of oxime **II** and 1.2 mmol of triethylamine in 3 ml of benzene was added at stirring 1 mmol of respective acid chloride. The mixture was stirred at 60°C for 3 h. Residue was filtered off and filtrate was washed with water and dried ($MgSO_4$). After evaporation of the solvent the residue was wear out with petroleum ether.

Diethyl *N*-(4-nitrobenzoyloxy)-4-fluorobenzimidoylphosphonate (IIIa). Yield 59%, mp 73–75°C. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.31 t (*Z*) and 1.41 t (*E*) (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.2 m (*Z*), 4.3 m (*E*), (4H, CH_2), 7.1–7.3 m (2H, FAr), 7.69 d.d (*J* 8.4 and 5.9 Hz) (*E*), 7.79 d.d (*J* 8.4 and 5.4 Hz) (*Z*) (2H, FAr), 8.00 d (*J* 9.3 Hz) (*E*) and 8.38 d (*J* 9 Hz) (*Z*) (2H, O_2NAr), 8.28 d (*J* 9.3 Hz) (*E*) and 8.49 d (*J* 9 Hz) (*Z*) (2H, O_2NAr). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –108.21 (*E*), –108.74 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 5.9 (*E*), 1.5 (*Z*), *E*/*Z* ~ 5.9. Found,

%: C 51.36; H 4.42; N 6.51; P 7.45. $C_{18}H_{18}FN_2O_7P$. Calculated, %: C 50.95; H 4.28; N 6.60; P 7.30.

Diethyl *N*-(4-nitrobenzoyloxy)-3-fluorobenzimidoylphosphonate (IIIb). Yield 64%, mp 89–90°C. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.31 t (*Z*) and 1.40 t (*E*) (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.2 m (*Z*), 4.3 m (*E*), (4H, CH_2), 7.2–7.6 m (4H, FAr), 7.99 d (*J* 9 Hz) (*E*) and 8.38 d (*J* 9 Hz) (*Z*) (2H, O_2NAr), 8.27 d (*J* 9 Hz) (*E*) and 8.50 d (*J* 9 Hz) (*Z*) (2H, O_2NAr). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –111.41 (*E*), –112.23 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 4.7 (*E*), 1.4 (*Z*), *E*/*Z* ~ 10. Found, %: N 6.43; P 7.52. $C_{18}H_{18}FN_2O_7P$. Calculated, %: N 6.60; P 7.30.

Diethyl *N*-(4-chlorobenzoyloxy)-4-fluorobenzimidoylphosphonate (IIIc). Yield 73%, oil. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.27 (*Z*) and 1.35 (*E*) (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.1 m (*Z*), 4.29 quintet, *J* 7 Hz (*E*), (4H, CH_2), 7.1–7.2 m (2H, FAr), 7.37 d (*J* 8.3 Hz) (*E*) and 7.48 d (*J* 8.3 Hz) (*Z*) (2H, ClAr), 7.6–7.7 m (2H, FAr), 7.73 d (*J* 8.3 Hz) (*E*) and 8.16 d (*J* 8.3 Hz) (*Z*) (2H, ClAr). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –108.74 (*E*), –109.30 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 5.2 (*E*), 0.7 (*Z*), *E*/*Z* ~ 10. Found, %: Cl 8.43; N 3.24; P 7.67. $C_{18}H_{18}ClFNO_5P$. Calculated, %: Cl 8.57; N 3.39; P 7.49.

Diethyl *N*-(4-chlorobenzoyloxy)-3-fluorobenzimidoylphosphonate (IIId). Yield 76%, oil. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.22 (*Z*) and 1.31 (*E*) (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.1 m (*Z*), 4.25 quintet, *J* 7 Hz (*E*), (4H, CH_2), 7.0–7.5 m (4H, FAr), 7.33 d (*J* 8.5 Hz) (*E*) and 7.95 d (*J* 8.1 Hz) (*Z*) (2H, ClAr), 7.68 d (*J* 8.1 Hz) (*E*) and 8.16 d (*J* 8.5 Hz) (*Z*) (2H, ClAr). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –111.75 (*E*), –112.50 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 5.4 (*E*), 0.8 (*Z*), *E*/*Z* ~ 2. Found, %: Cl 8.72; N 3.47; P 7.27. $C_{18}H_{18}ClFNO_5P$. Calculated, %: Cl 8.57; N 3.39; P 7.49.

***N*-Carbamoyloxyimidoylphosphonates (IV)**. Equimolar mixture of oxime **II** and 3,4-dichlorophenylisocyanate was stirred at 60°C for 6 h. Solvent was then evaporated and residue ws wear with hexane.

Diethyl *N*-(3,4-dichlorophenylcarbamoyloxy)-4-fluorobenzimidoylphosphonate (IVa). Yield 84%, mp 74–76°C. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.26 (*Z*) and 1.29 (*E*), overlapping triplets (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.0–4.2 m (4H, CH_2), 7.0–7.6 m (7H, Ar), 8.0 br (*E*) and 8.3 br (*Z*) (1H, NH). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –108.23 (*E*), –109.27 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 5.7 (*E*), 0.6 (*Z*), *E*/*Z* ~ 3.2. Found, %: Cl 15.59; P 6.72. $C_{18}H_{18}Cl_2FN_2O_5P$. Calculated, %: Cl 15.31; P 6.69.

Diethyl *N*-(3,4-dichlorophenylcarbamoyloxy)-3-fluorobenzimidoylphosphonate (IVb). Yield 48%, mp 87–89°C. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.26 (*Z*) and 1.28 (*E*), overlapping triplets (6H, CH_3, $^3J_{HH}$ 7 Hz), 4.0–4.2 m (4H, CH_2), 7.0–7.4 m (6H, Ar), 7.45 d (*Z*) and 7.61 d (*E*) (1H, $Cl_2C_6H_3$), 8.1 br (*Z*) and 8.2 br (*E*) (1H, NH). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –111.44 (*E*), –112.21 (*Z*). The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 5.3 (*E*), 0.1 (*Z*), *E*/*Z* ~ 5.9. Found, %: N 5.94; P 6.63. $C_{18}H_{18}Cl_2FN_2O_5P$. Calculated, %: N 6.05; P 6.69.

Reaction of *p*-nitrobenzoyloxyimidoylphosphonate (IIIa) with methanol (Scheme 3). A solution of compound **IIIa** (1 mmol) in 1 ml of methanol was refluxed for 6 h. Methanol was evaporated. ^{1}H and ^{31}P NMR analysis showed quantitative formation of oxime **IIa** and methyl *p*-nitrobenzoate identified by comparison with authentic samples.

Reaction of *p*-nitrobenzoyloxyimidoylphosphonate (IIIa) with benzylamine (Scheme 3). To a solution of 0.07 g (0.16 mmol) of imidoylphosphonate **IIIa** in 0.5 ml of benzene was added at 5°C 0.017 g (0.16 mmol) of benzylamine. After 2-h reflux the solvent was evaporated and residue was washed with petroleum ether. The residue by the data of ^{1}H and ^{31}P NMR spectroscopy contained *p*-nitrobenzylamide and oxime **IIa** that were identified by comparison with authentic samples.

Imidoylphosphonates (V). To a solution of 1 mmol of oxime **II** and 1.1 mmol of triethylamine in 3 ml of ether was added at stirring and cooling with ice 1 mmol of 4-tolylsulfinyl chloride. The temperature was allowed to reach room one and the mixture was left overnight. The precipitate was filtered off, the filtrate was evaporated and the residue was washed with petroleum ether.

Diethyl *N*-(4-tolylsulfonyl)-4-fluorobenzimidoylphosphonate (Va). Yield 68%, oil. The ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.21 m (6H, CH_3), 2.44 s (3H, $CH_3C_6H_4$), 4.02–4.18 m (4H, CH_2), 7.15 d.d (2H, Ar, $^3J_{HH}$ 8 Hz, $^3J_{HF}$ 8 Hz), 7.31 d (2H, Ar, $^3J_{HH}$ 8 Hz), 7.80 d (2H, Ar, $^3J_{HH}$ 8 Hz), 7.83–7.87 m (2H, Ar). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –106.5. The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 2.5.

Diethyl *N*-(4-tolylsulfonyl)-3-fluorobenzimidoylphosphonate (Vb). Yield 60%, oil. The ^{1}H NMR

spectrum ($CDCl_3$), δ, ppm: 1.21 m (6H, CH_3), 2.43 s (3H, $CH_3C_6H_4$), 4.03–4.19 m (4H, CH_2), 7.21 m (1H, Ar), 7.30 d (2H, Ar, $^3J_{HH}$ 8 Hz), 7.41–7.57 m (3H, Ar), 7.77 d (2H, Ar, $^3J_{HH}$ 8 Hz). The ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –111.7. The ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 2.6.

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