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To the 85th Anniversary of birthday of late Yu.G. Gololobov

N-Cycloalkyl- and *N*-Arylimidoylphosphonates

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Abstract—Reaction of acetyl- and fluorobenzoylphosphonates with 2- and 3-trifluoromethyl-substituted cyclohexylamines or 2- and 3-trifluoromethylanilines has resulted in the formation of the corresponding iminophosphonates and/or in the cleavage of the C–P bond yielding the dialkyl phosphites. The σ constants of *N*-[(trifluoromethyl)cyclohexyl]- and *N*-[(trifluoromethyl)phenyl]-substituted imidoylphosphoryl group have been determined by means of ^{19}F NMR spectroscopy.

Keywords: imidoylphosphonate, oxophosphonate, cyclohexyl, trifluoromethyl, *E/Z* isomerism, σ constants

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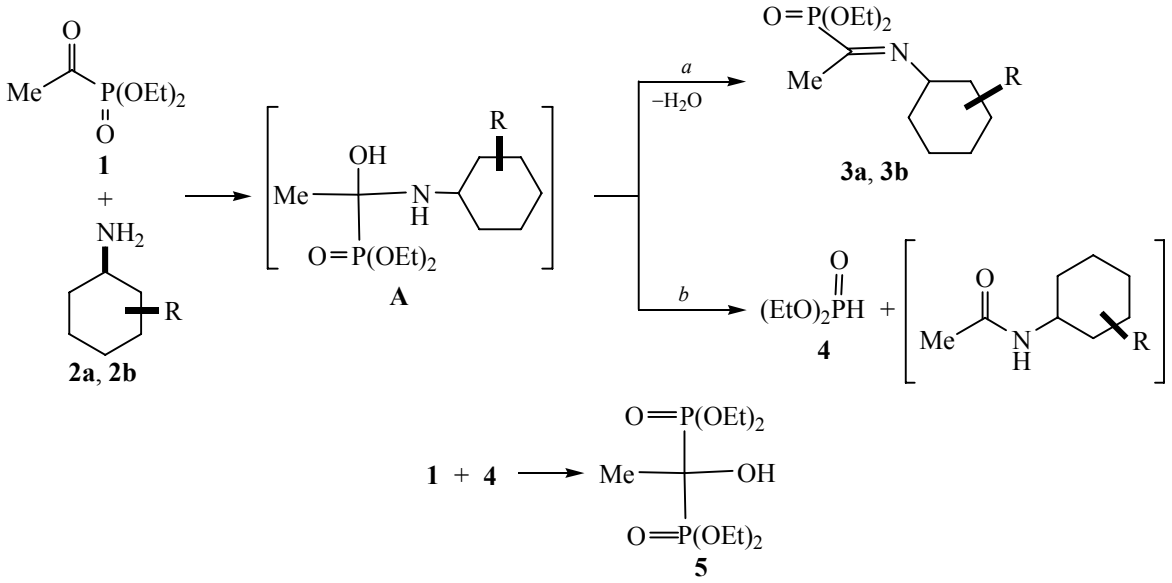
Imidoylphosphonates are recognized as new promising precursors of aminophosphonic acids. The presence of the "oxidized" fragment of α-aminophosphonic acid >P(O)C=N (capable of smooth reductive functionalization) in imidoylphosphonates opens versatile synthetic routes to new biologically important acyclic and heterocyclic derivatives containing aminophosphonate pharmacophore [1–5]. The emerging interest to the mentioned derivatives is due to the wide range of their practically important properties. In particular, they potentially can act as enzymes activity regulators or inhibitors of HIV protease; they have been applied as antibacterial, antiviral, and anticancer agents [6, 7]. We have earlier developed synthetic procedures for the preparation of NH-, *N*-alkyl-, *N*-aryl-, *N*-acyl-, *N*-sulfonyl-, and *N*-phosphoryliminophosphonates [1–5, 8, 10–12]. However, cycloalkyliminophosphonates have remained almost unknown so far [8, 9]. The presence of a cycloalkyl group is expected to improve physicochemical and biological properties of the discussed compounds. The overwhelming majority of the synthetic approaches to the imidoylphosphonates have been based on phosphorylation of imidoyl chlorides [8]. We have recently applied this route to prepare the first representatives of trifluoroacetyl- [9] and benzimidoylphosphonates [10] containing a cyclopropyl group at the nitrogen atom. The application of this method to obtain *N*-cycloalkyliminophosphonates has been limited by the poor availability of the starting imidoyl chlorides and their relatively low reactivity. On the other hand, the condensation of available α-oxophosphonates with amines appears as the most straightforward and the easiest route to the imidoylphosphonates; however, only a few examples of successful application of this method have been reported [11, 13, 14]. We have earlier demonstrated that the outcome of this reaction dramatically depends on the nature of the amine component: in the case of benzylamine, the C–P bond cleavage yielding the corresponding *N*-benzylamide is the primary pathway [11]. The less nucleophilic aminophosphonates readily react with oxophosphonates giving the target imidoylphosphonates in a high yield [11]. This work aimed to demonstrate the application of this approach by the study of the acetyl- and fluorobenzoylphosphonates reactions with isomeric trifluoromethylcyclohexylamines and trifluoromethylanilines of quite different nucleophilicity.

The outcome of the reaction of *O*,*O*-diethyl acetylphosphonate **1** with isomeric trifluoromethylcyclohexylamines appeared to depend on the amine structure. In particular, *cis*-2-trifluoromethylcyclohexylamine **2a** [15] reacted with phosphonate **1** under conditions described in [14] (Et_2O, AcOH, 20°C) to form exclusively iminophosphonate **3a**. In contrast, the reaction of the isomeric *cis*-3-trifluoromethyl-

Scheme 1.



R = 2-CF_3 (**a**), 3-CF_3 (**b**).

cyclohexylamine **2b** [15] under the same conditions was not selective: according to the ^{31}P NMR data, iminophosphonate **3b** was a minor product (<20%), the major oxygen-containing products being diethyl phosphite **4** and 1-hydroxyethylidene-1,1-bisphosphonate **5** (**3b** : **4** : **5** ≈ 1 : 1.5 : 3). The obtained results may be understood in the frame of Scheme 1.

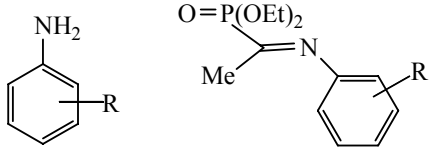
Intermediate **A** formed in the first stage could decompose via two competing pathways. With R = 3-CF_3 the elimination of dialkyl phosphite anion occurred (route *b*), owing to the influence of strongly electron-donor amino and hydroxy groups (cf. [11]). In the case of amine **2a**, the donor properties of amine substituent were significantly reduced due to the inductive effect of the CF_3 group, the C–P bond in the intermediate **A** was stronger, and the major pathway of its decay was dehydration (route *a*).

It could be expected that the far less nucleophilic trifluoromethylanilines **6a** and **6b** would mainly react via the path *a*. Indeed, the reaction of ketophosphonate **1** with anilines **6a** and **6b** initially occurred selectively to give the iminophosphonates **7a** and **7b**, but the reaction was however very slow: the conversion of 34% was attained after 2 days, other conditions being the same (Et_2O, AcOH, and 20°C) (Scheme 2).

Further keeping of the reaction mixture resulted in hydrolysis of the iminophosphonates **8a** and **8b**, and the formed diethyl phosphite **4** reacted with the starting compound **1** to give the hydroxybisphosphonate **5**. The interaction between compounds **1** and **6a** under the more severe conditions (refluxing in benzene in the presence of TsOH with simultaneous azeotropic distillation off of water) afforded the complete conversion within 48 h, the reaction mixture contained iminophosphonate **7a**, hydroxybisphosphonate **5**, and diethyl phosphite **4** (**7a** : **5** : **4** ≈ 10 : 6 : 1; ^{31}P NMR data).

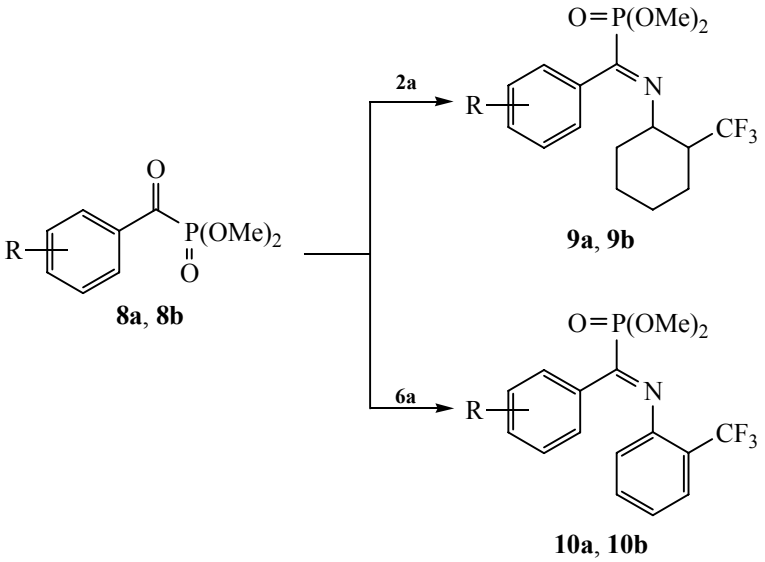
3- and 4-fluorobenzoylphosphonates **8a** and **8b** reacted with 2-trifluoromethylcyclohexylamine signifycantly faster. Other conditions being the same (benzene, TsOH, refluxing), the reaction was complete within 2 h but was accompanied by the formation of side products: the content of iminophosphonates **9a** and **9b** in the reaction mixture was of 30 and 70%, respectively. The interaction of fluorobenzoylphosphonates **8a** and **8b** with 2-trifluoromethylaniline **6a** was more selective. The reaction was complete within 48 h under the same conditions, and the reaction

Scheme 2.



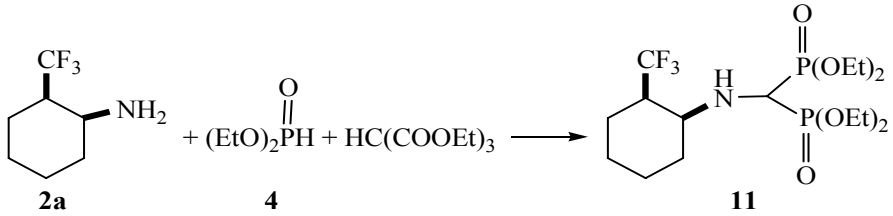
R = 2-CF_3 (**a**), 3-CF_3 (**b**).

Scheme 3.



R = 3-F (**a**), 4-F (**b**).

Scheme 4.

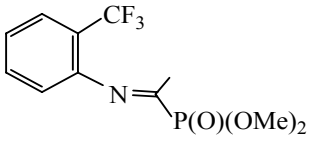
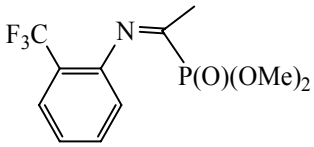
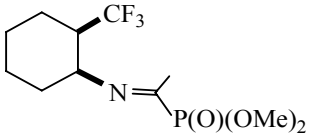


mixture contained >90% of iminophosphonates **10a** and **10b**, the content of dimethyl phosphite not exceeding 5–7% (^{31}P and ^{19}F NMR data) (Scheme 3).

However, pure iminophosphonates **10a** and **10b** could not be isolated (even by means of chromatography), for in the course of workup they are subject to ambiguous transformations resulting in the formation of complex mixtures of products. We believe that the low stability of compounds **10** was not due to the presence of the *o*-CF_3 group, but rather owing to the capability of the P–C=N fragment to the cleavage of the P–C bond, especially in the presence of basic or acidic admixtures [4, 8, 16, 17]. For example, the aminobisphosphonate **11** prepared via Scheme 4 was sufficiently stable under ordinary conditions. Noteworthily, the *N*-(cycloalkyl)aminobisphosphonic acids have been recognized as efficient phosphatase inhibitors and are therefore regarded as promising candidates for development of drugs for malaria and dysentery treatment [18].

The spectral data confirmed the suggested structures of iminophosphonates **3**, **7**, **9**, and **10**. The most indicative feature was the position of phosphorus nuclei signal (3.2–8.8 ppm), typical of alkyl- [19, 20] and benzimidoylphosphonates [16, 20] and allowing for easy identification of those compounds, including the reaction mixtures case. ^{13}C NMR spectrum of compound **3a** contained a downfield doublet signal of the imine carbon atom (165 ppm) exhibiting the strong spin-spin coupling ($^1J_{CP}$ = 218 Hz) typical of the phosphorus atom adjacent to the carbon one; that unambiguously confirmed the presence of the P–C=N fragment. Positions of the phosphorus nucleus signals in the acetylimidoylphosphonates **3** and **7** are in accordance with the *E* configuration of the C=N bond in those compounds [14, 19, 20]. At the same time, benzimidoylphosphonates **9** and **10** (similarly to other representatives of that class) existed as mixtures of *E* and *Z* isomers, the *E* form being predominant [*E* : *Z* ≈ (3–5) : 1]. Signals of phosphorus atoms of the *E* isomers of compounds **9** and **10** (7.8–8.8 ppm) were

Chemical shifts δ_F (ppm) of the fluorosubstituted benzenes XC_6H_4F and σ constants of *N*-substituted imidoylphos-phonate groups

Run no.	X	$\delta^F(XC_6H_4F)$		σ_I	σ_R	σ_p
		3-F	4-F			
1		2.15	4.55	0.39	0.08	0.47
2		1.10	4.82	0.24	0.13	0.37
3		0.76	1.6	0.19	0.03	0.22

detected down field from those of the *Z* isomers (3.2–5.5 ppm), in accordance with the literature data [16, 17, 20].

Electronic nature of *N*-[(trifluoromethyl)cyclo-hexyl]- and *N*-[(trifluoromethyl)phenyl]-substituted imidoylphosphoryl groups. We took advantage of sensitivity of fluorine nucleus to the electronic effects of the substituents to quantify the effects of *N*-[(tri-fluoromethyl)cyclohexyl]- and *N*-[(trifluoromethyl)-phenyl]-substituted imidoylphosphoryl groups. In particular, we measured chemical shifts of fluorine atom of the fluorophenyl group in imidoylphosphonates **9** and **10** (in $CDCl_3$, relative to fluorobenzene as internal reference) and applied the Taft equations (1) and (2) [21, 22] to elucidate the inductive and resonance σ constants of imidoylphosphoryl groups containing aryl and cycloalkyl substituents at the nitrogen atom.

$$\sigma_I = (\delta_m^F + 0.6)/7.1, \quad (1)$$

$$\sigma_R = (\delta_p^F - \delta_m^F)/29.5. \quad (2)$$

Analysis of the results (see table) showed that the imidoylphosphonate groups containing trifluoromethyl-phenyl substituent at the nitrogen atom (nos. 1 and 2) were strong electron acceptors (σ_p 0.37–0.47). The substitution of phenyl fragment with cyclohexyl one significantly deteriorated the electron-acceptor properties (σ_p = 0.22). Noteworthily, the σ constants were affected by the molecule geometry: The electron-acceptor ability of the *E*-imidoylphosphonate group (the major form of the imidoylphosphonates **10a** and **10b**) was significantly stronger than that of the corresponding *Z* form. The similar effect of the C=N bond geometry has been earlier revealed for the imidoylphosphonates containing substituted cyclo-propyl substituents at the nitrogen atom [10]. In general, the electron-acceptor properties of the imino-phosphonates followed the following series of the substituents at the nitrogen atom: $ArSO_2$, $(RO)_2P(O)$ [23, 24] > Ar > $PhCH_2$ [25] > cyclohexyl > cyclo-propyl [10].

In summary, we showed that preparative application of the reaction between α-ketophos-phonates and amines to synthesize *N*-aryl- and *N*-cyclohexyl-substituted iminophosphonates was sig-nificantly limited. The outcome was affected by the amine nature: The lower amine nucleophilicity favored the formation of the iminophosphonates, whereas the higher nucleophilicity led to the cleavage of the C–P bond. The introduction of 3- and 4-fluorobenzoyl-phosphonates in the reaction gave the first-time reported estimation of electronic effects of the iminophosphonate groups containing cyclohexyl or aryl substituents at the nitrogen atom.

EXPERIMENTAL

^1H, ^{19}F, ^{31}P, and ^{13}C NMR spectra were recorded using a Varian VXR–300 spectrometer at 299.95, 282.20, 121.42, and 75.429 MHz, respectively. The

chemical shifts were reported relative to the internal TMS (^{1}H and ^{13}C), $CFCl_3$ (in Experimental) or PhF (in table) for ^{19}F or external 85% H_3PO_4 (^{31}P) references. All reactions were performed in anhydrous conditions under argon atmosphere.

***O*,*O*-Diethyl-*N*-[(*cis*-2-trifluoromethyl)cyclohexyl]-acetimidoylphosphonate (3a).** A drop of glacial acetic acid was added to a solution of *O*,*O*-diethyl-acetylphosphonate **1** (1 mmol) and 2-(trifluoromethyl)-cyclohexylamine **2a** (1 mmol) in 5 mL of anhydrous diethyl ether. The mixture was stirred at room temperature under argon atmosphere during 24 h; the reaction course was monitored by means of ^{31}P and ^{19}F NMR spectroscopy. The solvent was evaporated; the residue was washed with a small amount of petroleum ether and kept in a high vacuum to give the iminophosphonate **3a** containing >90% of the main component (yield 95%, NMR data). The crude product was distilled in a vacuum to afford the pure product. mp 101–102°C (0.12 mmHg). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.34 t (6H, $OCH_2C\underline{H}_3$, $^3J_{HH}$ 6.0), 1.39–2.05 m (8H, CH_2, cyclohexyl), 2.09 d (3H, CH_3C=N, $^3J_{HP}$ 10.6), 2.33 m (1H, $CHCF_3$), 4.16 m (4H, OCH_2). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz): 15.79 d ($\underline{C}H_3C$=N, $^2J_{CP}$ 43.0), 16.22 d ($\underline{C}H_3CH_2$, $^3J_{CP}$ 6.0), 16.26 d ($\underline{C}H_3CH_2$, $^3J_{CP}$ 7.0); 20.00, 20.15, 25.26, 32.54 (C^4, C^5, C^3, C^6, cyclohexyl), 46.38 q ($\underline{C}HCF_3$, $^2J_{CF}$ 24.0), 54.02 d (CHN, $^3J_{CP}$ 32.0), 62.76 d (OCH_2, $^2J_{CP}$ 7.0), 63.28 d (OCH_2, $^2J_{CP}$ 6.0), 126.94 q (CF_3, $^1J_{CF}$ 280.0), 165.29 q (C=N, $^1J_{CP}$ 218.0). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –70.52 d ($^3J_{CF}$ 8.0 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 7.64 ppm. Calculated, %: N 4.25; P 9.41. $C_{13}H_{23}F_3NO_3P$. Found, %: N 4.08; P 9.63.

The reaction of *cis*-3-trifluoromethylcyclohexyl-amine **2a** with compound **1** under the same conditions yielded a mixture of iminophosphonate **3b** (δ_P = 8.15 ppm), diethyl phosphite **4** (δ_P = 7.2 ppm, $^1J_{PH}$ = 695 Hz), and *O*,*O*-diethyl-1-hydroxyethylidenebis-phosphonate **5** (δ_P = 21.7 ppm; δ_P = 21.02 ppm [26]) in the **3b** : **4** : **5** ≈ 1 : 1.5 : 3 ratio.

Interaction of fluorobenzoylphosphonates 8a and 8b with trifluoromethylcyclohexylamines 2 or trifluoromethylanilines 6. 1 mmol of the correspond-ing amine and 15 mg of TsOH were added to a solution of 1 mmol of the compound **8** in 15 mL of anhydrous benzene. The mixture was refluxed during 48 h in an apparatus equipped with a Dean–Stark trap. The reaction course was monitored by means of ^{31}P and ^{19}F spectroscopy. Iminophosphonates **7**, **9**, and **10** were not isolated and were identified by the spectral methods.

***O*,*O*-Diethyl-*N*-[(2-trifluoromethyl)phenyl]acet-imidoylphosphonate (7a).** ^{31}P NMR spectrum (Et_2O): δ_P 6.83 ppm.

***O*,*O*-Diethyl-*N*-[(3-trifluoromethyl)phenyl]acet-imidoylphosphonate (7b).** ^{31}P NMR spectrum (Et_2O): δ_P 7.54 ppm.

***O*,*O*-Dimethyl-*N*-[(2-trifluoromethyl)cyclohexyl]-3-fluorobenzimidoylphosphonate (9a).** ^{19}F NMR spectrum (C_6H_6), δ_F, ppm: –69.50 d (CF_3, $^3J_{FH}$ 8.0 Hz, *E* isomer), –69.38 (CF_3, $^3J_{FH}$ 9.0 Hz, *Z* isomer), –112.23 (ArF, *E* isomer), –111.34 (ArF, *Z* isomer). ^{31}P NMR spectrum (C_6H_6), δ_P, ppm: 4.59 (*Z*), 8.07 (*E*), *E* : *Z* ≈ 5 : 1.

***O*,*O*-Dimethyl-*N*-[(2-trifluoromethyl)cyclohexyl]-4-fluorobenzimidoylphosphonate (9b).** ^{19}F NMR spectrum (C_6H_6), δ_F, ppm: –69.50 d (CF_3, $^3J_{FH}$ 10.0 Hz, *E* isomer), –69.37 (CF_3, $^3J_{FH}$ 8.0 Hz, *Z* isomer), –111.40 (ArF, *E* isomer), –109.35 (ArF, *Z* isomer). ^{31}P NMR spectrum (C_6H_6), δ_P, ppm: 3.19 (*Z*), 8.75 (*E*), *E* : *Z* ≈ 5 : 1.

***O*,*O*-Dimethyl-*N*-[(2-trifluoromethyl)phenyl]-3-fluorobenzimidoylphosphonate (10a).** ^{19}F NMR spectrum (C_6H_6), δ_F, ppm: –61.09 (CF_3, *E* isomer), –60.89 (CF_3, *Z* isomer), –111.04 (ArF, *E* isomer), –111.95 (ArF, *Z* isomer). ^{31}P NMR spectrum (C_6H_6), δ_P, ppm: 5.13 (*Z*), 7.81 (*E*), *E* : *Z* ≈ 3 : 1.

***O*,*O*-Dimethyl-*N*-[(2-trifluoromethyl)phenyl]-4-fluorobenzimidoylphosphonate (10b).** ^{19}F NMR spectrum (C_6H_6), δ_F, ppm: –61.07 (CF_3, *E* isomer), –60.89 (CF_3, *Z* isomer), –108.80 (ArF, *E* isomer), –108.43 (ArF, *Z* isomer). ^{31}P NMR spectrum (C_6H_6), δ_P, ppm: 5.5 (*Z*), 8.1 (*E*), *E* : *Z* ≈ 3 : 1.

***O*,*O*,*O*,*O*-Tetraethyl-({[2-(trifluoromethyl)cyclo-hexyl]amino}methylene)bisphosphonate (11).** A mixture of compound **2a** (0.334 g, 2 mmol), triethyl orthoformate (0.556 g, 8 mmol), and diethyl phosphite (1.104 g, 8 mmol) was heated at 150°C during 15 h. The volatile products were evaporated in a high vacuum, and the residue was purified by preparative TLC (CH_3OH : $CHCl_3$ = 1 : 49, R_f 0.1). Yield 28%. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.22 m (12H, CH_3), 1.35–1.95 m? (8H, CH_2, cyclohexyl), 2.08 m (1H, $CHCF_3$), 3.25 t (CHP, $^2J_{HP}$ 21.7 Hz), 3.51 br.s (1H, NH), 4.10 m (8H, OCH_2). ^{13}C NMR spectrum

($CDCl_3$), δ_C, ppm (J, Hz): 16.18 (CH_3), 16.23 (CH_3), 16.30 (CH_3), 18.81 (CH_2^4), 19.53 (CH_2^5), 24.81 (CH_2^3), 28.16 (CH_2^6), 45.68 q ($\underline{C}HCF_3$, $^2J_{CF}$ 25.0), 49.72 d ($CH_2\underline{C}HN$, $^3J_{CP}$ 11.0), 50.67 d. d (CHP, $^1J_{CP}$A 138.0, $^1J_{CP}$B 152.0), 62.46 d (OCH_2, $^3J_{CP}$ 7.0), 62.84 (OCH_2, $^3J_{CP}$ 6.0), 63.00 (OCH_2, $^3J_{CP}$ 7.0), 127.34 q (CF_3, $^1J_{CF}$ 280.0). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –68.71 d ($^3J_{FH}$ 10.0 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 20.06 ppm. Calculated, %: N 3.09; P 13.66. $C_{16}H_{32}F_3NO_6P_2$. Found, %: N 3.21; P 13.37.

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