Synthesis of functionalized furan-containing phosphonous and phosphinic acids

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A convenient synthesis of functionalized furan-containing phosphonous and phosphinic acids has been developed. The radical addition of bis(trimethylsiloxy)phosphine to 2-vinylfuran and trimethylsilyl 3-(2-furyl)acrylate proceeds regioselectively to afford new 2-furyl-substituted alkylphosphonites, the subsequent aminomethylation and carboxyethylation of which give various target phosphinic acids.

Key words: bis(trimethylsiloxy)phosphine, 2-vinylfuran, trimethylsilyl 3-(2-furyl)-acrylate, radical addition, azobis(isobutyronitrile), aminomethylation, carboxyethylation, functionalized phosphonous and phosphinic acids, biologically active substances, polydentate ligands.

Recently, functionalized furan derivatives such as furfural, 5-hydroxymethylfurfural, and 2,5-furandicarboxylic acid, derived from virtually inexhaustible plant biomass, have attracted attention as promising compounds with high synthetic potential that can serve as a basis for the transition to renewable chemical production of organic materials and fuels in the future.[1—7] Of particular interest among furan derivatives are substances that exhibit various biological activities, such as furacilin and furazolidone, as well as plant growth regulators containing furan fragments.[8] It was noted that promising organosilicon furan derivatives show antiviral, fungicidal and antitumour activities.[9] In this regard, it seems relevant to develop methods for the synthesis of furans containing fragments of organophosphorus analogs of amino acids such as functionalized derivatives of phosphonic and phosphinic acids, which are well-known biologically active substances[10,11] and are widely used as effective polydentate ligands.[12]

Previously,[13,14] we have presented preliminary data on the synthesis of some functionalized phosphinates using trimethylsilyl esters of phosphinic acid[15—17] and phosphorous acid.[18—23] The successful use of organosilicon methodology to create P—C bonds allowed us to prepare a number of promising 2-phosphorus-substituted tetraorganosilanes,[15,16] phosphorus-substituted amines, azaheterocycles, amino acids and peptides containing methylenediphosphonic acid fragments.[17—23] In continuation of these studies, the present work provides an access to functionalized furan-containing phosphonous and phosphinic acids using highly reactive bis(trimethylsiloxy)phosphine[24,25] and also readily available 2-vinylfuran and trimethylsilyl 3-(2-furyl)acrylate.

Radical addition of P—H phosphines to unsaturated compounds is an effective method for creating P—C bonds.[15,16,26,27] We found that the radical addition of bis(trimethylsiloxy)phosphine to 2-vinylfuran gave phosphonites **1** and **2** in 64 and 25% yields, respectively. The reaction proceeds only in the presence of the reaction initiator, azobis(isobutyronitrile) (AIBN), under its thermolysis conditions (100—120 °C) (Scheme 1).

Thus, the formation of phosphonite **1** follows the classical path of chain reaction involving phosphinyl radical **A**, through an intermediate radical **B** with a radical center on the carbon atom (Scheme 2). The formation of phosphonite **2** is associated with the chain transfer from intermediate radical **B** to the second molecule of 2-vinylfuran followed by generation of radical **C**. These results fits well with the

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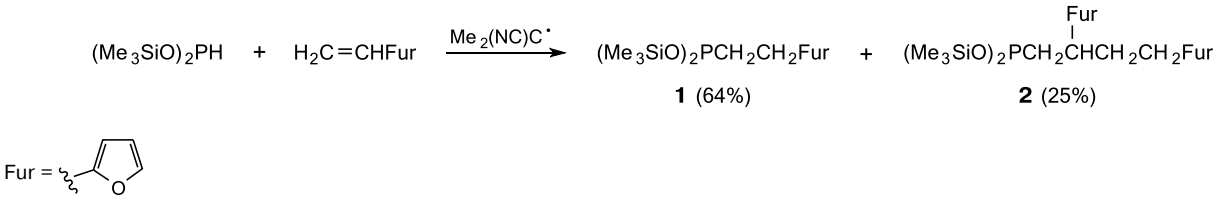
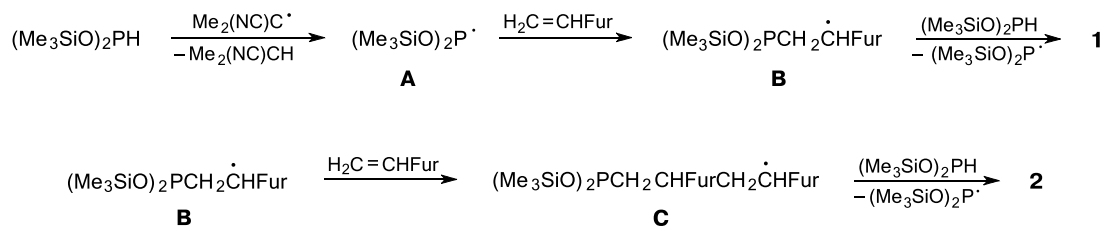
Scheme 1



Reagents and conditions: AIBN, 100—120 °C.

Scheme 2



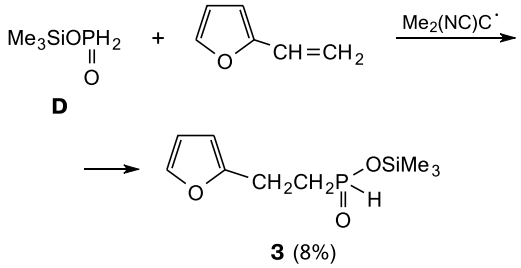
known data on similar radical addition reactions,[28,29] which has also been recently confirmed by us.[15,16]

Along with phosphonites **1** and **2**, redistillation of the high-boiling fraction gave hydrophosphoryl-containing phosphonite **3** in a low yield (8%) (Scheme 3). Such a result may be due to the presence in the reaction mixture of trimethylsilyl hypophosphite **D**, which is usually formed in the preparation of the starting bis(trimethylsiloxy)phosphine.[24,25] It is also capable of radical addition to the C=C bond, as mentioned earlier.[15,16,29,30]

Under similar conditions, bis(trimethylsiloxy)-phosphine adds to trimethylsilyl 3-(2-furyl)acrylate regioselectively to afford exclusively phosphonite **4** in 89% yield, *i.e.*, the directing effect of the furan moiety is decisive during the process and completely coincides with the effect of the triorganosilyl group in the synthesis of 2-phosphorus-substituted tetraorganosilanes[15,16] (Scheme 4).

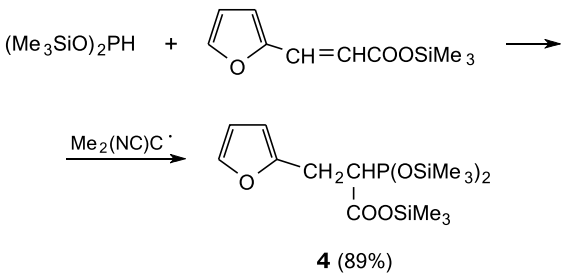
Phosphonites **1** and **4** contain highly reactive P—O—Si units and, therefore, can serve as starting materials for the preparation of various functionalized furan-containing phosphinates **5—7**. Thus, phosphonites **1** and **4** are readily aminomethylated, and the conditions of the processes are determined by the reactivity of aminomethylating reagents.[15,16,31] Weakly electrophilic bis(dialkylamino)methanes react with phosphonites **1** and **4** only when heated to 130 °C in the presence of zinc chloride catalyst to

Scheme 3



Reagents and conditions: AIBN, 100—120 °C.

Scheme 4

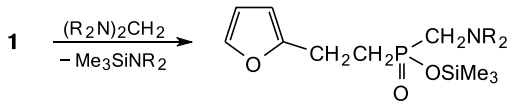


Reagents and conditions: AIBN, 100—130 °C.

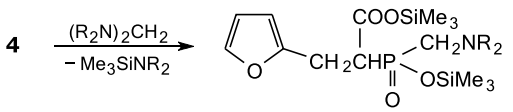
form phosphinates **5a—c** and **6a—d** in high yields of 72—86% (Scheme 5).

Zinc chloride as the catalyst activates the starting bis(dialkylamino)methanes to generate electrophilic

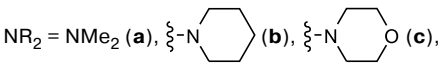
Scheme 5



5a—c (78—86%)



6a—d (72—78%)

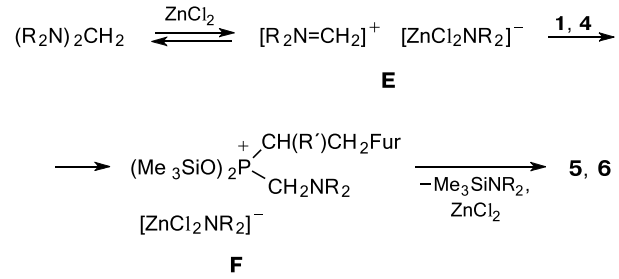


N(Me)CH_2COOEt (**d**)

Reagents and conditions: $ZnCl_2$, 110—130 °C.

iminium salts **E** on heating. These salts would react with phosphonites **1** and **4** along the classical Arbuzov reaction pathway[31] *via* intermediate adducts **F** to give the target phosphinates **5a—c** and **6a—d** (Scheme 6).

Scheme 6



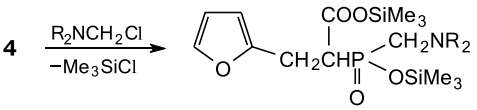
R′ = H, $COOSiMe_3$

The use of electrophilic *N*-chloromethylamides in aminomethylation of phosphonite **4** provides target phosphinates **6e,f** already at 10 °C in dichloromethane in 80—82% yields (Scheme 7).

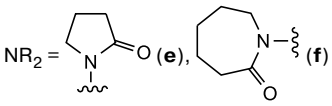
Under similar conditions, the reaction of phosphonite **4** with trimethylsilyl acrylate proceeds as 1,4-coupling of the P—O—Si fragment to form first an adduct, thermally unstable phosphorus-containing ketene acetal **G** (Scheme 8). Its selective desilylation using diethyl phosphite affords phosphinate **7** comprising two different carboxy-containing moieties in 76% yield.

The treatment of phosphonites **1—4** with a dilute solution of sodium methoxide in methanol gave

Scheme 7

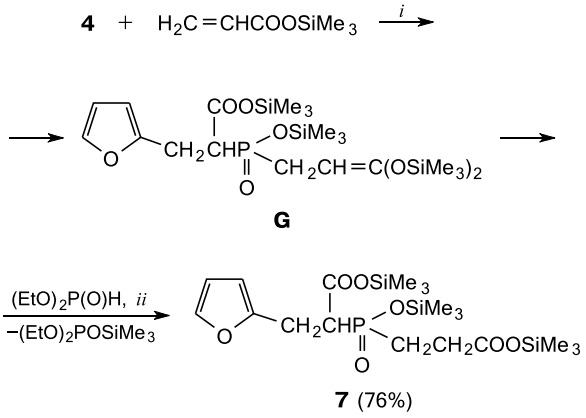


6e (80%)
6f (82%)



Reagents and conditions: CH_2Cl_2, 10—20 °C.

Scheme 8



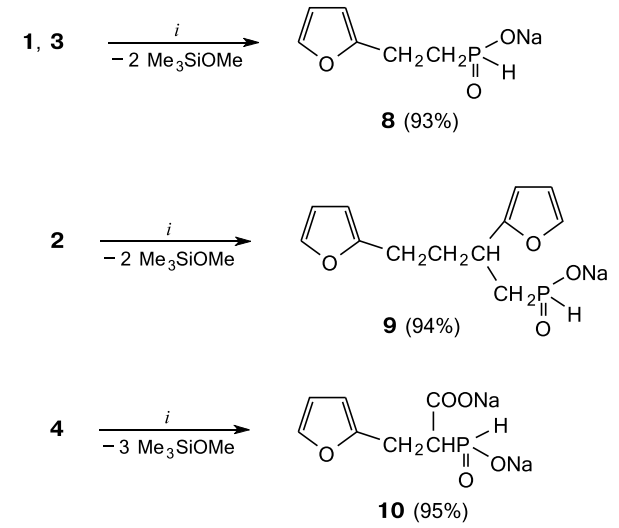
Reagents and conditions: *i*. CH_2Cl_2, 10—20 °C; *ii*. 40—120 °C.

water-soluble sodium salts of phosphonic acids **8—10**, which contain furan fragments and are white crystals (Scheme 9). The yields of these salts are 93—95%.

A similar reaction of phosphinates **5—7** under mild conditions provides high yields of sodium phosphinates **11—13** as white crystals (Scheme 10). These compounds include furyl, aminomethyl and carboxyl groups in the hydrolysis-resistant C—P—C unit.

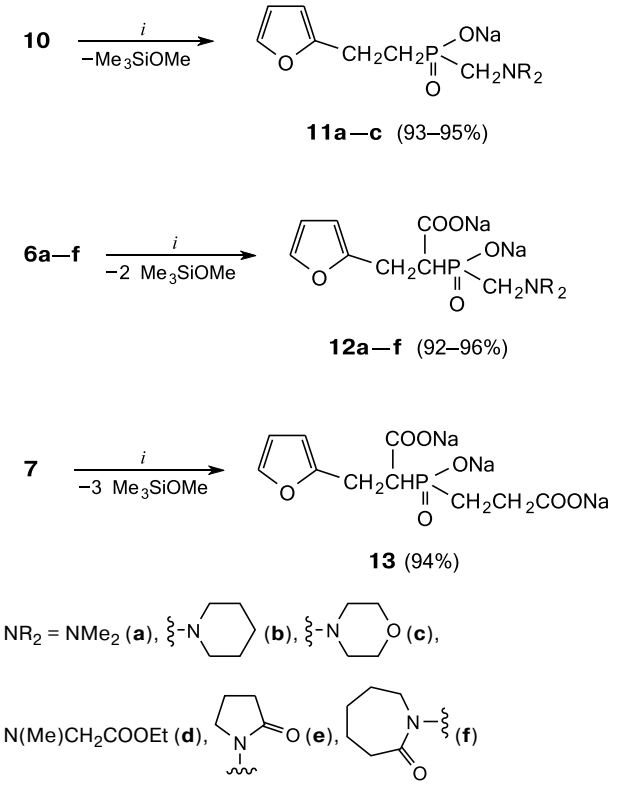
The NMR spectra of compounds **1—13** show characteristic signals for $PC(1)H_2C(2)H_2C_{Fur}(3)$, $PC(1)H_2C(2)H[C_{Fur}(3)]C(4)H_2C(5)H_2C_{Fur}(6)$, $PC(1)H[C(4)=O]C(2)H_2C_{Fur}(3)$, $PC(5)H_2NC(6)H_2$, and $PC(7)H_2C(8)H_2C(9)=O$ moieties, the parameters of which are given in Experimental. Compounds **6** and **7** contain asymmetric phosphorus and carbon atoms and are mixtures of two stereoisomers differing in the ^{31}P NMR spectra. The contents of diastereomers were determined from the ^{31}P NMR spectra.

Scheme 9



Reagents and conditions: MeONa, MeOH, 10—40 °C.

Scheme 10



Reagents and conditions: MeONa, MeOH, 10—40 °C.

To summarize, a variety of phosphonous and phosphinic acids **1—13** comprising furan fragments were obtained by creating phosphorus—carbon bonds based on readily available compounds such as bis(trimethylsiloxy)phosphine, 2-vinylfuran, and trimethylsilyl 3-(2-furyl)acrylate. These compounds containing bioactive aminomethyl, carboxyl and heterocyclic moieties along with fragments of organophosphorus analogues of amino acids, are promising biologically active compounds, and can also find application as effective polydentate ligands.

Experimental

^{1}H, $^{13}C\{^{1}H\}$, and $^{31}P\{^{1}H\}$ NMR spectra were recorded on a Bruker Avance 400 spectrometer at 400, 100, and 162 Hz operating frequencies, respectively, against Me_4Si as an internal standard (^{1}H, $^{13}C\{^{1}H\}$) and 85% H_3PO_4 as an external standard in D_2O ($^{31}P\{^{1}H\}$). Melting points were measured in open cappillaries and were uncorrected. All reactions were carried out in a dry argon atmosphere in anhydrous solvents. Elemental analysis data were obtained on a Perkin Elmer Series II CHNS/O 2400 Analyser. Elemental analysis of compounds **1—4**, which are readily oxidized and hydrolyzed, was carried out for their stable derivatives, sodium salts **8—10**; structures of phosphonites **1—4** were additionally confirmed by NMR.

The starting bis(trimethylsiloxy)phosphine was prepared according to the known procedure.[24,25] Commercially available 2-vinylfuran, trimethylsilyl 3-(2-furyl)acrylate, and azobis(isobutyronitrile) (Acros Organics and Alfa Aesar) were used as purchased.

Synthesis of 2-furyl-substituted phosphonites 1—4 (general procedure). A mixture of bis(trimethylsiloxy)phosphine (70 mmol), 2-vinylfuran or trimethylsilyl 3-(2-furyl)acrylate (25 mmol), and azobis(isobutyronitrile) (5 mmol) was heated to 100 °C, the temperature was raised to 130 °C for 1 h, then the mixture was distilled *in vacuo* to give phosphonites **1** and **4**. Redistillation of high-boiling fractions gave also the corresponding phosphonites **2** and **3**.

Bis(trimethylsilyl) [2-(2-furyl)ethyl]phosphonite (1). Yield 64%, b.p. 92 °C (1 Torr). ^{1}H NMR ($CDCl_3$), δ: 0.26 (s, 18 H, 2 Me_3Si); 1.71—1.77 (m, 2 H, C(1)H_2); 2.76—2.82 (m, 2 H, C(2)H_2); 6.02—6.04 (m, 1 H, $C_{Fur}H$); 6.28—6.30 (m, 1 H, $C_{Fur}H$); 7.31—7.33 (m, 1 H, $C_{Fur}H$). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$), δ: 1.14 (s, 2 Me_3Si); 19.64 (d, C(2), $^{2}J_{PC}$ = 15.1 Hz); 38.97 (d, C(1), $^{1}J_{PC}$ = 25.1 Hz); 104.28 (s, C_{Fur}); 109.86 (s, C_{Fur}); 140.50 (s, C_{Fur}); 156.19 (d, C(3), $^{3}J_{PC}$ = 10.6 Hz). $^{31}P\{^{1}H\}$ NMR ($CDCl_3$), δ: 157.22 (s).

Bis(trimethylsilyl) [2,4-di(2-furyl)butyl]phosphonite (2). Yield 25%, b.p. 135 °C (1 Torr). ^{1}H NMR ($CDCl_3$), δ: 0.22 (s, 9 H, Me_3Si); 0.26 (s, 9 H, Me_3Si); 1.69—1.78 (m, 2 H, C(1)H_2); 2.01—2.14 (m, 2 H, C(4)H_2); 2.48—2.66 (m, 2 H, C(5)H_2); 3.02—3.11 (m, 1 H, C(2)H_2); 5.80—6.08 (m, 2 H, 2 $C_{Fur}H$); 6.26—6.34 (m, 2 H, 2 $C_{Fur}H$); 7.26—7.38 (m, 2 H, 2 $C_{Fur}H$). $^{13}C\{^{1}H\}$ NMR

($CDCl_3$), δ: 1.13 (s, 2 Me_3Si); 25.35 (s, C(5)); 32.43 (d, C(4), $^3J_{PC}$ = 12.6 Hz); 33.76 (d, C(2), $^2J_{PC}$ = 6.9 Hz); 46.09 (d, C(1), $^1J_{PC}$ = 28.1 Hz); 104.55 (s, C_{Fur}); 104.99 (s, C_{Fur}); 109.64 (s, C_{Fur}); 109.85 (s, C_{Fur}); 140.44 (s, C_{Fur}); 140.62 (s, C_{Fur}); 155.39 (s, C(6)); 157.81 (d, C(3), $^3J_{PC}$ = 4.6 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 158.33 (s).

Trimethylsilyl [2-(2-furyl)ethyl]phosphonite (3). Yield 8%, b.p. 115 °C (1 Torr). ^1H NMR ($CDCl_3$), δ: 0.37 (s, 9 H, Me_3Si); 1.72—1.76 (m, 2 H, C(1)H_2); 2.77—2.81 (m, 2 H, C(2)H_2); 6.03—6.05 (m, 1 H, $C_{Fur}H$); 6.30—6.32 (m, 1 H, $C_{Fur}H$); 6.94 (d, 1 H, PH, $^1J_{PH}$ = 543.2 Hz); 7.33—7.35 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: 1.44 (s, Me_3Si); 29.21 (d, C(1), $^1J_{PC}$ = 94.0 Hz); 38.70 (s, C(2)); 105.45 (s, C_{Fur}); 110.06 (s, C_{Fur}); 141.20 (s, C_{Fur}); 156.89 (d, C(3), $^3J_{PC}$ = 14.8 Hz). ^{31}P NMR ($CDCl_3$), δ: 22.42 (dt, $^1J_{PH}$ = 543.2 Hz, $^2J_{PH}$ = 15.1 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 22.42 (s).

Bis(trimethylsilyl) [1-trimethylsiloxycarbonyl-2-(2-furyl)ethyl]phosphonite (4). Yield 89%, b.p. 126 °C (1 Torr). ^1H NMR ($CDCl_3$), δ: 0.04 (s, 9 H, Me_3Si); 0.06 (s, 9 H, Me_3Si); 0.11 (s, 9 H, Me_3Si); 2.46—2.72 (m, 2 H, C(2)H_2); 3.00—3.06 (m, 1 H, C(1)H); 5.95—5.98 (m, 1 H, $C_{Fur}H$); 6.16—6.24 (m, 1 H, $C_{Fur}H$); 7.18—7.22 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –0.67 (s, Me_3Si); 0.85 (s, Me_3Si); 0.93 (s, Me_3Si); 32.27 (d, C(2), $^2J_{PC}$ = = 15.0 Hz); 46.24 (d, C(1), $^1J_{PC}$ = 28.9 Hz); 105.90 (d, C_{Fur}, $^4J_{PC}$ = 5.9 Hz); 110.96 (s, C_{Fur}); 140.96 (s, C_{Fur}); 152.44 (d, C(3), $^3J_{PC}$ = 8.9 Hz); 172.62 (d, C(4), $^2J_{PC}$ = 9.1 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 144.55 (s).

Synthesis of 2-furyl-substituted phosphinates 5a—c, 6a—d (general procedure). A mixture of phosphonite **1** or phosphonite **4** (20 mmol), bis(dialkylamino)methane (25 mmol), and zinc chloride (1.5 mmol) was heated at 110—130 °C for 1.5 h, then distilled *in vacuo* to give phosphinates **5a—c** or **6a—d,** respectively.

Trimethylsilyl [(dimethylaminomethyl)[2-(2-furyl)-ethyl]phosphinate (5a). Yield 83%, b.p. 118 °C (1 Torr). Found (%): C, 49.52; H, 8.20. $C_{12}H_{24}NO_3PSi$. Calculated (%): C, 49.81; H, 8.36. ^1H NMR ($CDCl_3$), δ: 0.20 (s, 9 H, Me_3Si); 1.55—1.68 (m, 2 H, C(1)H_2); 1.84 (s, 6 H, 2 C(6)H_3); 2.01 (1 H, C(5)H_B) and 2.08 (1 H, C(5)H_A) (m, ABX-system, $^2J_{HAHB}$ = 14.8 Hz, $^2J_{PHA}$ = 9.6 Hz, $^2J_{PHB}$ = 10.4 Hz); 2.36—2.49 (m, 2 H, C(2)H_2); 5.52—5.54 (m, 1 H, $C_{Fur}H$); 5.75—5.77 (m, 1 H, $C_{Fur}H$); 6.80—6.82 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: 0.22 (s, Me_3Si); 19.83 (s, C(2)); 26.05 (d, C(1), $^1J_{PC}$ = 93.1 Hz); 46.60 (d, C(6), $^3J_{PC}$ = 10.0 Hz); 57.67 (d, C(5), $^1J_{PC}$ = = 113.7 Hz); 104.12 (s, C_{Fur}); 109.14 (s, C_{Fur}); 140.04 (s, C_{Fur}); 153.39 (d, C(3), $^3J_{PC}$ = 16.4 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 39.32 (s).

Trimethylsilyl (piperidin-1-ylmethyl)[2-(2-furyl)ethyl]-phosphinate (5b). Yield 78%, b.p. 158 °C (1 Torr), m.p. 42 °C. Found (%): C, 52.72; H, 8.78. $C_{14}H_{28}NO_3PSi$. Calculated (%): C, 52.97; H, 8.89. ^1H NMR ($CDCl_3$), δ: 0.03 (s, 9 H, Me_3Si); 1.10—1.16 (m, 2 H, CH_2); 1.20—1.35 (m, 4 H, 2 CH_2); 1.75—1.90 (m, 2 H, C(1)H_2); 2.10—2.35 (m, 6 H, C(2)H_2, 2 C(6)H_2); 2.55—2.80 (m, 2 H, C(5)H_2); 5.75—5.77 (m, 1 H, $C_{Fur}H$); 5.87—6.10 (m, 1 H, $C_{Fur}H$); 7.01—7.05 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: 0.70 (s, Me_3Si); 20.37 (s, C(2)); 23.21 (s, CH_2); 25.48 (s, 2 CH_2); 26.77 (d, C(1), $^1J_{PC}$ = 93.1 Hz); 56.03 (d, C(6), $^3J_{PC}$ = 9.2 Hz); 57.79 (d, C(5), $^1J_{PC}$ = 113.8 Hz); 104.47 (s, C_{Fur}); 109.56 (s, C_{Fur}); 140.46 (s, C_{Fur}); 154.04 (d, C(3), $^3J_{PC}$ = 16.4 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 39.81 (s).

Trimethylsilyl (morpholin-4-ylmethyl)[2-(2-furyl)ethyl]-phosphinate (5c). Yield 86%, b.p. 152 °C (1 Torr). Found (%): C, 48.59; H, 8.07. $C_{13}H_{26}NO_4PSi$. Calculated (%): C, 48.88; H, 8.21. ^1H NMR ($CDCl_3$), δ: 1.11 (s, 9 H, Me_3Si); 1.62—1.78 (m, 2 H, C(1)H_2); 2.05—2.80 (m, 8 H, C(2)H_2, C(5)H_2, 2 C(6)H_2); 3.18—3.28 (m, 4 H, CH_2O); 5.62—5.68 (m, 1 H, $C_{Fur}H$); 5.84—5.88 (m, 1 H, $C_{Fur}H$); 6.86—6.97 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: 0.38 (s, Me_3Si), 19.99 (s, C(2)); 26.33 (d, C(1), $^1J_{PC}$ = 93.7 Hz); 54.60 (d, C(6), $^3J_{PC}$ = 9.1 Hz); 56.85 (d, C(5), $^1J_{PC}$ = 112.7 Hz); 65.84 (s, 2 CH_2O); 104.32 (s, C_{Fur}); 109.31 (s, C_{Fur}); 140.23 (s, C_{Fur}); 153.45 (d, C(3), $^3J_{PC}$ = 16.0 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 38.59 (s).

Trimethylsilyl (dimethylaminomethyl)[1-trimethylsil-oxycarbonyl-2-(2-furyl)ethyl]phosphinate (6a). Yield 74%, b.p. 143 °C (1.5 Torr). Found (%): C, 47.05; H, 7.81. $C_{16}H_{32}NO_5PSi_2$. Calculated (%): C, 47.38; H, 7.95. First isomer (60%). ^1H NMR ($CDCl_3$), δ: –0.28 (s, 9 H, Me_3Si); –0.19 (s, 9 H, Me_3Si); 1.88 (s, 6 H, 2 C(6)H_3); 2.39—2.74 (m, 2 H, C(2)H_2); 3.22—3.33 (m, 1 H, C(1)H); 3.45—3.55 (m, 2 H, C(5)H); 5.68—5.78 (m, 1 H, $C_{Fur}H$); 5.81—5.88 (m, 1 H, $C_{Fur}H$); 6.82—6.89 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: 0.13 (s, Me_3Si); 1.40 (s, Me_3Si); 32.22 (s, C(2)); 34.88 (d, C(1), $^1J_{PC}$ = 92.0 Hz); 46.53 (d, C(6), $^3J_{PC}$ = 9.9 Hz); 56.09 (d, C(5), $^1J_{PC}$ = = 117.2 Hz); 106.50 (d, C_{Fur}, $^4J_{PC}$ = 6.1 Hz); 109.69 (s, C_{Fur}); 140.69 (s, C_{Fur}); 149.49 (d, C(3), $^3J_{PC}$ = 4.8 Hz); 170.18 (d, C(4), $^2J_{PC}$ = 16.5 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.16 (s). Second isomer (40%). ^1H NMR ($CDCl_3$), δ: –0.26 (s, 9 H, Me_3Si); –0.19 (s, 9 H, Me_3Si); 1.84 (s, 6 H, 2 C(6)H_3); 2.39—2.74 (m, 2 H, C(2)H_2); 3.22—3.33 (m, 1 H, C(1)H); 3.45—3.55 (m, 2 H, C(5)H); 5.68—5.78 (m, 1 H, $C_{Fur}H$); 5.81—5.88 (m, 1 H, $C_{Fur}H$); 6.82—6.89 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: 0.22 (s, Me_3Si); 1.40 (s, Me_3Si); 33.13 (s, C(2)); 37.34 (d, C(1), $^1J_{PC}$ = 91.2 Hz); 46.45 (d, C(6), $^3J_{PC}$ = 7.6 Hz); 55.80 (d, C(5), $^1J_{PC}$ = 116.5 Hz); 106.50 (d, C_{Fur}, $^4J_{PC}$ = 6.1 Hz); 109.69 (s, C_{Fur}); 140.69 (s, C_{Fur}); 149.27 (d, C(3), $^3J_{PC}$ = 6.7 Hz); 170.27 (d, C(4), $^2J_{PC}$ = 16.9 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.49 (s).

Trimethylsilyl (piperidin-1-ylmethyl)[1-trimethylsiloxy-carbonyl-2-(2-furyl)ethyl]phosphinate (6b). Yield 72%, b.p. 175 °C (1 Torr). Found (%): C, 51.03; H, 8.06. $C_{19}H_{36}NO_5PSi_2$. Calculated (%): C, 51.21; H, 8.14. First

isomer (65%). ^{1}H NMR ($CDCl_3$), δ: –0.11 (s, 9 H, Me_3Si); –0.02 (s, 9 H, Me_3Si); 1.05—1.16 (m, 2 H, CH_2); 1.18—1.32 (m, 4 H, 2 CH_2); 2.12—2.38 (m, 7 H, C(1)H, $C(2)H_2$, 2 $C(6)H_2$); 2.69—2.95 (m, 2 H, $C(5)H_2$); 5.84—5.90 (m, 1 H, $C_{Fur}H$); 6.00—6.05 (m, 1 H, $C_{Fur}H$); 7.02—7.04 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –1.10 (s, Me_3Si); 0.57 (s, Me_3Si); 23.20 (s, CH_2); 25.29 (s, 2 CH_2); 32.64 (s, C(2)); 35.34 (d, C(1), $^1J_{PC}$ = 91.8 Hz); 55.81 (d, C(6), $^3J_{PC}$ = 9.3 Hz); 56.07 (d, C(5), $^1J_{PC}$ = 116.8 Hz); 106.81 (d, C_{Fur}, $^4J_{PC}$ = 6.4 Hz); 109.96 (s, C_{Fur}); 140.05 (s, C_{Fur}); 149.85 (d, C(3), $^3J_{PC}$ = 8.0 Hz); 170.68 (d, C(4), $^2J_{PC}$ = 16.5 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.66 (s). Second isomer (35%). ^{1}H NMR ($CDCl_3$), δ: –0.09 (s, 9 H, Me_3Si); –0.02 (s, 9 H, Me_3Si); 1.05—1.16 (m, 2 H, CH_2); 1.18—1.32 (m, 4 H, 2 CH_2); 2.12—2.38 (m, 7 H, C(1)H, $C(2)H_2$, 2 $C(6)H_2$); 2.69—2.95 (m, 2 H, $C(5)H_2$); 5.84—5.90 (m, 1 H, $C_{Fur}H$); 6.00—6.05 (m, 1 H, $C_{Fur}H$); 7.02—7.04 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –1.10 (s, Me_3Si); 0.57 (s, Me_3Si); 24.34 (s, CH_2); 25.46 (s, 2 CH_2); 33.70 (s, C(2)); 37.75 (d, C(1), $^1J_{PC}$ = 91.4 Hz); 55.71 (d, C(6), $^3J_{PC}$ = 9.6 Hz); 55.77 (d, C(5), $^1J_{PC}$ = 116.7 Hz); 106.64 (d, C_{Fur}, $^4J_{PC}$ = 6.9 Hz); 110.05 (s, C_{Fur}); 140.05 (s, C_{Fur}); 149.73 (d, C(3), $^3J_{PC}$ = 6.2 Hz); 170.79 (d, C(4), $^2J_{PC}$ = 16.5 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.84 (s).

Trimethylsilyl (morpolin-4-ylmethyl)[1-trimethylsiloxycarbonyl-2-(2-furyl)ethyl]phosphinate (6c). Yield 78%, b.p. 183 °C (1.5 Torr). Found (%): C, 47.98; H, 7.52. $C_{18}H_{34}NO_6PSi_2$. Calculated (%): C, 48.30; H, 7.66. First isomer (60%). ^{1}H NMR ($CDCl_3$), δ: –0.04 (s, 9 H, Me_3Si); 0.05 (s, 9 H, Me_3Si); 2.18—2.44 (m, 7 H, C(1)H, $C(2)H_2$, 2 $C(6)H_2$); 2.47—2.98 (m, 2 H, $C(5)H_2$); 3.38—3.45 (m, 4 H, CH_2O); 5.90—5.97 (m, 1 H, $C_{Fur}H$); 6.04—6.12 (m, 1 H, $C_{Fur}H$); 7.08—7.13 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –0.98 (s, Me_3Si); –0.61 (s, Me_3Si); 32.59 (s, C(2)); 35.36 (d, C(1), $^1J_{PC}$ = 92.7 Hz); 54.81 (d, C(6), $^3J_{PC}$ = 8.8 Hz); 55.97 (d, C(5), $^1J_{PC}$ = 116.1 Hz); 66.34 (s, 2 CH_2O); 106.94 (s, C_{Fur}); 110.15 (s, C_{Fur}); 140.10 (s, C_{Fur}); 149.77 (d, C(3), $^3J_{PC}$ = 7.8 Hz); 170.72 (d, C(4), $^2J_{PC}$ = 16.5 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.00 (s). Second isomer (40%). ^{1}H NMR ($CDCl_3$), δ: –0.02 (s, 9 H, Me_3Si); 0.05 (s, 9 H, Me_3Si); 2.18—2.44 (m, 7 H, C(1)H, $C(2)H_2$, 2 $C(6)H_2$); 2.47—2.98 (m, 2 H, $C(5)H_2$); 3.38—3.45 (m, 4 H, CH_2O); 5.90—5.97 (m, 1 H, $C_{Fur}H$); 6.04—6.12 (m, 1 H, $C_{Fur}H$); 7.08—7.13 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –0.98 (s, Me_3Si); –0.61 (s, Me_3Si); 33.74 (s, C(2)); 37.81 (d, C(1), $^1J_{PC}$ = 92.2 Hz); 54.72 (d, C(6), $^3J_{PC}$ = 8.3 Hz); 55.41 (d, C(5), $^1J_{PC}$ = 111.0 Hz); 66.21 (s, 2 CH_2O); 106.99 (s, C_{Fur}); 110.22 (s, C_{Fur}); 140.10 (s, C_{Fur}); 149.53 (d, C(3), $^3J_{PC}$ = 6.9 Hz); 170.76 (d, C(4), $^2J_{PC}$ = 16.7 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.02 (s).

Trimethylsilyl [1-trimethylsiloxycarbonyl-2-(2-furyl)ethyl](*N*-methylaminomethyl-*N*-ethoxycarbonylmethyl)phosphinate (6d). Yield 78%, b.p. 186 °C (1 Torr). Found (%): C, 47.69; H, 7.53. $C_{19}H_{36}NO_7PSi_2$. Calculated (%): C, 47.78; H, 7.60. First isomer (55%). ^{1}H NMR ($CDCl_3$), δ: –0.12 (s, 9 H, Me_3Si); –0.04 (s, 9 H, Me_3Si); 0.93 (t, 3 H, CH_3, $^2J_{HH}$ = 7.2 Hz); 2.23 (s, 3 H, $C(6)H_3$); 2.45—2.93 (m, 5 H, C(1)H, $C(2)H_2$, $C(5)H_2$); 3.17 (s, 2 H, NCH_2); 3.82 (q, 2 H, CH_2O, $^2J_{HH}$ = 7.2 Hz); 5.86—5.91 (m, 1 H, $C_{Fur}H$); 6.01—6.02 (m, 1 H, $C_{Fur}H$); 7.01—7.05 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –0.97 (s, Me_3Si); 0.56 (s, Me_3Si); 13.71 (s, Me); 32.81 (s, C(2)); 35.20 (d, C(1), $^1J_{PC}$ = 91.1 Hz); 43.34 (d, C(6), $^3J_{PC}$ = 7.5 Hz); 53.35 (d, C(5), $^1J_{PC}$ = 116.0 Hz), 57.82 (d, NCH_2, $^3J_{PC}$ = 8.7 Hz); 59.53 (s, CH_2O); 107.07 (s, C_{Fur}); 110.19 (s, C_{Fur}); 141.21 (s, C_{Fur}); 149.88 (d, C(3), $^3J_{PC}$ = 7.8 Hz); 169.88 (s, C=O); 170.74 (d, C(4), $^2J_{PC}$ = 13.8 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.44 (s). Second isomer (45%). ^{1}H NMR ($CDCl_3$), δ: –0.12 (s, 9 H, Me_3Si); –0.04 (s, 9 H, Me_3Si); 0.93 (t, 3 H, CH_3, $^2J_{HH}$ = 7.2 Hz); 2.18 (s, 3 H, $C(6)H_3$); 2.45—2.93 (m, 5 H, C(1)H, $C(2)H_2$, $C(5)H_2$); 3.16 (s, 2 H, NCH_2); 3.82 (q, 2 H, CH_2O, $^2J_{HH}$ = 7.2 Hz); 5.86—5.91 (m, 1 H, $C_{Fur}H$); 6.01—6.02 (m, 1 H, $C_{Fur}H$); 7.01—7.05 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –0.97 (s, Me_3Si); 0.66 (s, Me_3Si); 33.57 (s, C(2)); 37.61 (d, C(1), $^1J_{PC}$ = 90.9 Hz); 43.27 (d, C(6), $^3J_{PC}$ = 7.5 Hz); 53.09 (d, C(5), $^1J_{PC}$ = 115.5 Hz); 57.91 (d, NCH_2, $^3J_{PC}$ = 8.1 Hz); 59.53 (s, CH_2O); 107.01 (s, C_{Fur}); 110.34 (s, C_{Fur}); 140.18 (s, C_{Fur}); 149.61 (d, C(3), $^3J_{PC}$ = 6.9 Hz); 169.83 (s, C=O); 170.90 (d, C(4), $^2J_{PC}$ = 15.9 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 34.86 (s).

Synthesis of 2-furyl-substituted phosphinates 6e,f (general procedure). To a stirred solution of phosphonite **4** (25 mmol) in dichloromethane (25 mL), cooled to 10 °C, a solution of the appropriate *N*-chloromethylamide (25 mmol) in dichloromethane (25 mL) was added dropwise. The mixture was kept for 0.5 h at 20 °C, then the solvent was distilled off, the residue was distilled *in vacuo* to give phosphinates **6e**,**f**.

Trimethylsilyl (2-oxopyrrolidin-1-ylmethyl)[1-trimethylsiloxycarbonyl-2-(2-furyl)ethyl]phosphinate (6e). Yield 80%, b.p. 188 °C (1.5 Torr). Found (%): C, 48.39; H, 7.18. $C_{18}H_{32}NO_6PSi_2$. Calculated (%): C, 48.52; H, 7.24. First isomer (60%). ^{1}H NMR ($CDCl_3$), δ: –0.26 (s, 9 H, Me_3Si); –0.17 (s, 9 H, Me_3Si); 1.47—1.62 (m, 2 H, CH_2); 1.79—1.92 (m, 2 H, CH_2C=O); 2.34—2.59 (m, 2 H, $C(2)H_2$); 2.88—3.35 (m, 5 H, C(1)H, $C(5)H_2$, $C(6)H_2$); 5.84—5.90 (m, 2 H, 2 $C_{Fur}H$); 6.90—6.93 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: –1.29 (s, Me_3Si); 0.20 (s, Me_3Si); 17.04 (s, CH_2); 29.14 (s, CH_2C=O); 32.46 (s, C(2)); 36.03 (d, C(1), $^1J_{PC}$ = 89.9 Hz); 40.62 (d, C(5), $^1J_{PC}$ = 105.7 Hz); 47.32 (d, C(6), $^3J_{PC}$ = 6.9 Hz); 107.12 (d, C_{Fur}, $^4J_{PC}$ = 6.1 Hz); 109.96 (s, C_{Fur}); 141.17 (s, C_{Fur}); 148.31 (d, C(3), $^3J_{PC}$ = 9.1 Hz); 170.01 (d, NC=O, $^3J_{PC}$ = 15.7 Hz); 173.81 (d, C(4), $^2J_{PC}$ = 3.9 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 31.10 (s). Second isomer (40%). ^{1}H NMR ($CDCl_3$), δ: –0.25 (s, 9 H, Me_3Si); –0.17 (s, 9 H,

Me_3Si); 1.47—1.62 (m, 2 H, CH_2); 1.79—1.92 (m, 2 H, $CH_2C{=}O$); 2.34—2.59 (m, 2 H, $C(2)H_2$); 2.88—3.35 (m, 5 H, C(1)H, $C(5)H_2$, $C(6)H_2$); 5.84—5.90 (m, 2 H, 2 $C_{Fur}H$); 6.90—6.93 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: −1.29 (s, Me_3Si); 0.20 (s, Me_3Si); 16.98 (s, CH_2); 29.17 (s, $CH_2C{=}O$); 32.78 (s, C(2)); 37.13 (d, C(1), $^1J_{PC}$ = 91.0 Hz); 40.18 (d, C(5), $^1J_{PC}$ = 105.7 Hz); 47.32 (d, C(6), $^3J_{PC}$ = 6.9 Hz); 107.22 (d, C_{Fur}, $^4J_{PC}$ = 6.5 Hz); 109.96 (s, C_{Fur}); 141.21 (s, C_{Fur}); 148.17 (d, C(3), $^3J_{PC}$ = 7.1 Hz); 169.85 (d, NC=O, $^3J_{PC}$ = 16.3 Hz); 173.85 (d, C(4), $^2J_{PC}$ = 4.0 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 31.18 (s).

Trimethylsilyl (2-oxoazepan-1-ylmethyl)[1-trimethylsiloxycarbonyl-2-(2-furyl)ethyl]phosphinate (6f). Yield 82%, b.p. 195 °C (2 Torr). Found (%): C, 50.59; H, 7.61. $C_{20}H_{36}NO_6PSi_2$. Calculated (%): C, 50.72; H, 7.66. First isomer (60%). ^1H NMR ($CDCl_3$), δ: −0.15 (s, 9 H, Me_3Si); −0.05 (s, 9 H, Me_3Si); 1.15—1.45 (m, 6 H, 3 CH_2); 2.10—3.71 (m, 9 H, $CH_2C{=}O$, C(1)H, $C(2)H_2$, $C(5)H_2$, $C(6)H_2$); 5.87—6.01 (m, 2 H, 2 $C_{Fur}H$); 6.98—7.02 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: −1.05 (s, Me_3Si); 0.32 (s, Me_3Si); 22.52 (s, CH_2); 27.31 (s, CH_2); 27.38 (s, CH_2); 29.18 (s, $CH_2C{=}O$); 32.95 (s, C(2)); 36.20 (d, C(1), $^1J_{PC}$ = 90.1 Hz); 45.57 (d, C(5), $^1J_{PC}$ = 105.0 Hz); 50.22 (s, C(6)); 107.54 (d, C_{Fur}, $^4J_{PC}$ = 4.6 Hz); 110.18 (s, C_{Fur}); 141.30 (s, C_{Fur}); 148.71 (d, C(3), $^3J_{PC}$ = 9.1 Hz); 170.41 (d, NC=O, $^3J_{PC}$ = 16.9 Hz); 174.98 (s, C(4)). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 32.28 (s). Second isomer (40%). ^1H NMR ($CDCl_3$), δ: −0.16 (s, 9 H, Me_3Si); −0.08 (s, 9 H, Me_3Si); 1.15—1.45 (m, 6 H, 3 CH_2); 2.10—3.71 (m, 9 H, $CH_2C{=}O$, C(1)H, $C(2)H_2$, $C(5)H_2$, $C(6)H_2$); 5.87—6.01 (m, 2 H, 2 $C_{Fur}H$); 6.98—7.02 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: −1.05 (s, Me_3Si); 0.45 (s, Me_3Si); 22.52 (s, CH_2); 27.31 (s, CH_2); 27.38 (s, CH_2); 29.13 (s, $CH_2C{=}O$); 33.97 (s, C(2)); 37.16 (d, C(1), $^1J_{PC}$ = 90.0 Hz); 44.91 (d, C(5), $^1J_{PC}$ = 104.5 Hz); 49.98 (s, C(6)); 107.49 (d, C_{Fur}, $^4J_{PC}$ = 5.9 Hz); 110.15 (s, C_{Fur}); 141.27 (s, C_{Fur}); 148.44 (d, C(3), $^3J_{PC}$ = 7.2 Hz); 170.30 (d, NC=O, $^3J_{PC}$ = 17.9 Hz); 174.43 (s, C(4)). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 32.04 (s).

Trimethylsilyl [2-(trimethylsiloxycarbonyl)ethyl][1-trimethylsiloxycarbonyl-2-(2-furyl)ethyl]phosphinate (7). To a stirred solution of phosphonite **4** (7.3 g, 17 mmol) in dichloromethane (10 mL), a solution of trimethylsilyl acrylate (2.6 g, 18 mmol) in dichloromethane (5 mL) was added. The mixture was refluxed for 0.5 h, then a solution of diethyl phosphite (2.5 g, 18 mmol) in dichloromethane (5 mL) was added upon stirring. The reaction mixture was stirred for 0.5 h, the solvent was distilled off, the residue was heated to 120 °C and then distilled *in vacuo* to give phosphinate **7** (6.5 g, 13 mmol). Yield 76%, b.p. 178 °C (1 Torr). Found (%): C, 46.03; H, 7.49. $C_{19}H_{37}O_7PSi_3$. Calculated (%): C, 46.32; H, 7.57. First isomer (60%). ^1H NMR ($CDCl_3$), δ: −0.15 (s, 9 H, Me_3Si); −0.07 (s, 9 H, Me_3Si); −0.05 (s, 9 H, Me_3Si); 1.45—1.73 (m, 2 H, $C(7)H_2$); 2.05—2.35 (m, 2 H, $C(8)H_2$); 2.40—2.70 (m, 2 H, $C(2)H_2$); 3.28—3.45 (m, 1 H, C(1)H); 5.83—5.90 (m, 1 H, $C_{Fur}H$); 5.96—6.01 (m, 1 H, $C_{Fur}H$); 6.97—7.05 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: −1.05 (s, Me_3Si); −0.91 (s, Me_3Si); 0.52 (s, Me_3Si); 23.27 (d, C(7), $^1J_{PC}$ = 96.4 Hz); 27.50 (s, C(8)); 32.82 (s, C(2)); 37.09 (d, C(1), $^1J_{PC}$ = 93.2 Hz); 107.10 (d, C_{Fur}, $^4J_{PC}$ = 6.7 Hz); 110.21 (s, C_{Fur}); 141.37 (s, C_{Fur}); 149.23 (d, C(3), $^3J_{PC}$ = 7.6 Hz); 170.30 (d, C(4), $^2J_{PC}$ = 16.5 Hz); 171.64 (d, C(9), $^2J_{PC}$ = 17.0 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 38.53 (s). Second isomer (40%). ^1H NMR ($CDCl_3$), δ: −0.11 (s, 9 H, Me_3Si); −0.08 (s, 9 H, Me_3Si); −0.05 (s, 9 H, Me_3Si); 1.45—1.73 (m, 2 H, $C(7)H_2$); 2.05—2.35 (m, 2 H, $C(8)H_2$); 2.40—2.70 (m, 2 H, $C(2)H_2$); 3.28—3.45 (m, 1 H, C(1)H); 5.83—5.90 (m, 1 H, $C_{Fur}H$); 5.96—6.01 (m, 1 H, $C_{Fur}H$); 6.97—7.05 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ: −1.05 (s, Me_3Si); −0.91 (s, Me_3Si); 0.52 (s, Me_3Si); 22.52 (d, C(7), $^3J_{PC}$ = 96.4 Hz); 27.28 (s, C(8)); 33.26 (s, C(2)); 38.07 (d, C(1), $^1J_{PC}$ = 93.1 Hz); 107.31 (d, C_{Fur}, $^4J_{PC}$ = 7.0 Hz); 110.21 (s, C_{Fur}); 141.83 (s, C_{Fur}); 148.92 (d, C(3), $^3J_{PC}$ = 5.4 Hz); 170.22 (d, C(4), $^2J_{PC}$ = 16.7 Hz); 171.63 (d, C(9), $^2J_{PC}$ = 18.3 Hz). $^{31}P\{^1H\}$ NMR ($CDCl_3$), δ: 39.30 (s).

Synthesis of sodium salts of 2-furyl-substituted phosphinic acids 8—10 (general procedure). To a stirred solution of sodium methoxide (20 mmol) in methanol (30 mL), cooled to 10 °C, a solution of phosphonites **1, 3** (20 mmol) or phosphonites **2** and **4** (10 mmol) in diethyl ether (15 mL) was added. The mixture was heated to reflux, the solvent was distilled off, the residue was kept *in vacuo* (1 Torr) for 1 h to give sodium salts **8—10** as white crystals.

Sodium [2-(2-furyl)ethyl]phosphonite (8). Yield 93%, m.p. 152 °C (dec.). Found (%): C, 39.34; H, 4.59. $C_6H_8NaO_3P$. Calculated (%): C, 39.58; H, 4.43. ^1H NMR (D_2O), δ: 1.72—1.83 (m, 2 H, $C(1)H_2$); 2.70—2.85 (m, 2 H, $C(2)H_2$); 6.09—6.14 (m, 1 H, $C_{Fur}H$); 6.32—6.38 (m, 1 H, $C_{Fur}H$); 6.88 (dt, 1 H, PH, $^3J_{HH}$ = 2.0 Hz, $^1J_{PH}$ = 508.0 Hz); 7.35—7.39 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 20.17 (s, C(2)); 29.71 (d, C(1), $^1J_{PC}$ = 89.4 Hz); 105.16 (s, C_{Fur}); 110.56 (s, C_{Fur}); 141.66 (s, C_{Fur}); 155.55 (d, C(3), $^3J_{PC}$ = 15.5 Hz). ^{31}P NMR (D_2O), δ: 26.15 (d, $^1J_{PH}$ = 508.0 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 26.15 (s).

Sodium 2,4-di(2-furyl)butylphosphonite (9). Yield 94%, m.p. 154 °C (dec.). Found (%): C, 51.87; H, 5.19. $C_{12}H_{14}NaO_4P$. Calculated (%): C, 52.18; H, 5.11. ^1H NMR (D_2O), δ: 1.65—1.95 (m, 4 H, $C(1)H_2$, $C(4)H_2$); 2.24—2.45 (m, 1 H, $C(2)H_2$); 3.15—3.55 (m, 2 H, $C(5)H_2$); 5.75—5.86 (m, 2 H, 2 $C_{Fur}H$); 6.01—6.04 (m, 2 H, 2 $C_{Fur}H$); 6.15—6.17 (m, 2 H, 2 $C_{Fur}H$); 6.74 (dt, 1 H, PH, $^3J_{HH}$ = 2.4 Hz, $^1J_{PH}$ = 510.0 Hz). $^{13}C\{^1H\}$ NMR (D_2O), δ: 25.14 (s, C(5)); 33.18 (s, C(2)); 36.33 (d, C(1), $^1J_{PC}$ = 89.5 Hz); 33.74 (d, C(4), $^3J_{PC}$ = 11.3 Hz); 104.96 (s, C_{Fur}); 106.06 (s, C_{Fur}); 110.22 (s, C_{Fur}); 110.28 (s, C_{Fur}); 140.94 (s, C_{Fur}); 141.43 (s, C_{Fur}); 155.58 (s, C(6)); 156.88 (d, C(3), $^3J_{PC}$ = 6.7 Hz). ^{31}P NMR (D_2O),

δ: 23.53 (d, $^1J_{PH}$ = 510.0 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 23.53 (s).

Disodium [2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphonite (10). Yield 95%, m.p. 165 °C (dec.). Found (%): C, 33.68; H, 3.03. $C_7H_7Na_2O_5P$. Calculated (%): C, 33.89; H, 2.85. ^1H NMR (D_2O), δ: 2.52—2.72 (m, 1 H, C(2)H_2); 3.13—3.42 (m, 1 H, C(1)H); 6.16—6.19 (m, 1 H, $C_{Fur}H$); 6.38—6.42 (m, 1 H, $C_{Fur}H$); 6.84 (d, 1 H, PH, $^1J_{PH}$ = 515.6 Hz); 7.40—7.43 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 34.29 (s, C(2)); 40.24 (d, C(1), $^1J_{PC}$ = = 86.4 Hz); 106.59 (d, C_{Fur}, $^4J_{PC}$ = 6.4 Hz); 110.72 (s, C_{Fur}); 142.31 (s, C_{Fur}); 151.48 (d, C(3), $^3J_{PC}$ = 7.3 Hz); 180.04 (d, C(4), $^2J_{PC}$ = 17.5 Hz). ^{31}P NMR (D_2O), δ: 24.89 (d, $^2J_{PH}$ = 515.6 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 24.89 (s).

Synthesis of sodium salts of 2-(2-furyl)-substituted phosphinic acids 11—13 (general procedure). To a stirred solution of sodium methoxide (30 mmol) in methanol (50 mL), cooled to 10 °C, a solution of phosphinates **5** (30 mmol) or phosphinates **6** (15 mmol) or phosphinate **7** (10 mmol) in diethyl ether (15 mL) was added. The mixture was heated to reflux, the solvent was distilled off, the residue was kept *in vacuo* (1 Torr) for 1 h to give sodium salts **11—13** as white crystals.

Sodium (dimethylaminomethyl)[2-(2-furyl)ethyl]phosphinate (11a). Yield 92%, m.p. 148 °C (dec.). Found (%): C, 44.98; H, 6.25. $C_9H_{15}NNaO_3P$. Calculated (%): C, 45.19; H, 6.32. ^1H NMR (D_2O), δ: 1.50—1.64 (m, 2 H, C(1)H_2); 1.79 (s, 6 H, C(6)H_3); 2.05—2.11 (m, 2 H, C(5)H_2); 2.34—2.45 (m, 2 H, C(2)H_2); 5.53—5.56 (m, 1 H, $C_{Fur}H$); 5.73—5.75 (m, 1 H, $C_{Fur}H$); 6.81—6.84 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 19.76 (s, C(2)); 23.08 (d, C(1), $^1J_{PC}$ = 87.3 Hz); 46.68 (d, C(6), $^3J_{PC}$ = 8.2 Hz); 58.14 (d, C(5), $^1J_{PC}$ = 109.8 Hz); 104.98 (s, C_{Fur}); 109.85 (s, C_{Fur}); 140.15 (s, C_{Fur}); 153.94 (d, C(3), $^3J_{PC}$ = 15.2 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 37.78 (s).

Sodium (piperidin-1-ylmethyl)[2-(2-furyl)ethyl]phosphinate (11b). Yield 94%, m.p. 152 °C (dec.). Found (%): C, 49.28; H, 7.04. $C_{11}H_{19}NNaO_3P$. Calculated (%): C, 49.44; H, 7.17. ^1H NMR (D_2O), δ: 1.09—1.13 (m, 2 H, CH_2); 1.21—1.33 (m, 4 H, 2 CH_2); 1.74—1.87 (m, 2 H, C(1)H_2); 2.12—2.30 (m, 6 H, C(2)H_2, 2 C(6)H_2); 2.59—2.74 (m, 2 H, C(5)H_2); 5.72—5.76 (m, 1 H, $C_{Fur}H$); 5.82—6.04 (m, 1 H, $C_{Fur}H$); 7.03—7.08 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 20.28 (s, C(2)); 23.15 (s, CH_2); 25.42 (s, 2 CH_2); 25.92 (d, C(1), $^1J_{PC}$ = 89.9 Hz); 55.88 (d, C(6), $^3J_{PC}$ = 8.1 Hz); 57.05 (d, C(5), $^1J_{PC}$ = 108.1 Hz); 104.32 (s, C_{Fur}); 109.52 (s, C_{Fur}); 140.38 (s, C_{Fur}); 153.09 (d, C(3), $^3J_{PC}$ = 15.2 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 37.64 (s).

Sodium (morpholin-4-ylmethyl)[2-(2-furyl)ethyl]phosphinate (11c). Yield 95%, m.p. 168 °C (dec.) Found (%): C, 44.18; H, 6.23. $C_{10}H_{17}NNaO_4P$. Calculated (%): C, 44.32; H, 6.36. ^1H NMR (D_2O), δ: 1.59—1.77 (m, 2 H, C(1)H_2); 2.01—2.78 (m, 8 H, C(2)H_2, C(5)H_2, 2 C(6)H_2); 3.26 (s, 4 H, CH_2O); 5.58—5.64 (m, 1 H, $C_{Fur}H$); 5.81—5.86 (m, 1 H, $C_{Fur}H$); 6.89—6.95 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 20.02 (s, C(2)); 26.01 (d, C(1), $^1J_{PC}$ = 90.4 Hz); 54.42 (d, C(6), $^3J_{PC}$ = 8.4 Hz); 56.74 (d, C(5), $^1J_{PC}$ = 109.3 Hz); 65.69 (s, 2 CH_2O); 104.08 (s, C_{Fur}); 109.11 (s, C_{Fur}); 140.01 (s, C_{Fur}); 153.18 (d, C(3), $^3J_{PC}$ = 15.1 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 36.48 (s).

Disodium (dimethylaminomethyl)[2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphinate (12a). Yield 94%, m.p. 182 °C (dec.). Found (%): C, 39.23; H, 4.55. $C_{10}H_{14}NNa_2O_5P$. Calculated (%): C, 39.36; H, 4.62. ^1H NMR (D_2O), δ: 2.27 (s, 6 H, 2 C(6)H_3); 2.34—2.51 (m, 2 H, C(5)H_2); 2.59—2.77 (m, 2 H, C(2)H_2); 3.42—3.53 (m, 1 H, C(1)H); 6.15—6.19 (m, 1 H, $C_{Fur}H$); 6.36—6.40 (m, 1 H, $C_{Fur}H$); 7.40—7.43 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 34.96 (s, C(2)); 38.70 (d, C(1), $^1J_{PC}$ = 86.3 Hz); 46.81 (d, C(6), $^3J_{PC}$ = 7.9 Hz); 56.78 (d, C(5), $^1J_{PC}$ = 104.7 Hz); 106.62 (d, C_{Fur}, $^4J_{PC}$ = 6.3 Hz); 110.73 (s, C_{Fur}); 141.77 (s, C_{Fur}); 152.60 (d, C(3), $^3J_{PC}$ = 6.9 Hz); 180.43 (d, C(4), $^2J_{PC}$ = 15.2 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 32.86 (s).

Disodium (piperidin-1-ylmethyl)[2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphinate (12b). Yield 95%, m.p. 189 °C (dec.). Found (%): C, 44.98; H, 5.12. $C_{13}H_{18}NNa_2O_5P$. Calculated (%): C, 45.23; H, 5.25. ^1H NMR (D_2O), δ: 1.05—1.16 (m, 2 H, CH_2); 1.18—1.32 (m, 4 H, 2 CH_2); 2.12—2.38 (m, 7 H, C(1)H, C(2)H_2, 2 C(6)H_2); 2.69—2.95 (m, 2 H, C(5)H_2); 5.84—5.90 (m, 1 H, $C_{Fur}H$); 6.00—6.05 (m, 1 H, $C_{Fur}H$); 7.02—7.04 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 23.11 (s, CH_2); 24.96 (s, 2 CH_2); 35.37 (s, C(2)); 39.77 (d, C(1), $^1J_{PC}$ = 86.0 Hz); 55.76 (d, C(6), $^3J_{PC}$ = 7.4 Hz); 56.59 (d, C(5), $^1J_{PC}$ = 101.1 Hz); 106.68 (d, C_{Fur}, $^4J_{PC}$ = 6.4 Hz); 110.74 (s, C_{Fur}); 141.75 (s, C_{Fur}); 152.70 (d, C(3), $^3J_{PC}$ = 6.1 Hz); 180.51 (d, C(4), $^2J_{PC}$ = = 16.5 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 32.57 (s).

Disodium (morpholin-4-ylmethyl)[2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphinate (12c). Yield 96%, m.p. 194 °C (dec.). Found (%): C, 41.29; H, 4.68. $C_{12}H_{16}NNa_2O_6P$. Calculated (%): C, 41.51; H, 4.65. ^1H NMR (D_2O), δ: 2.40—2.82 (m, 7 H, C(1)H, C(2)H_2, 2 C(6)H_2); 3.43—3.56 (m, 2 H, C(5)H_2); 3.69—3.71 (m, 4 H, 2 CH_2O); 6.15—6.17 (m, 1 H, $C_{Fur}H$); 6.36—6.38 (m, 1 H, $C_{Fur}H$); 7.38—7.41 (m, 1 H, $C_{Fur}H$). $^{13}C\{^1H\}$ NMR (D_2O), δ: 35.26 (s, C(2)); 39.27 (d, C(1), $^1J_{PC}$ = 87.0 Hz); 54.69 (d, C(6), $^3J_{PC}$ = 7.9 Hz); 56.23 (d, C(5), $^1J_{PC}$ = 103.7 Hz); 66.41 (s, 2 CH_2O); 106.69 (d, C_{Fur}, $^4J_{PC}$ = 7.3 Hz); 110.72 (s, C_{Fur}); 141.73 (s, C_{Fur}); 152.62 (d, C(3), $^3J_{PC}$ = 6.9 Hz); 180.47 (d, C(4), $^2J_{PC}$ = 15.8 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 32.50 (s).

Disodium [2-(2-furyl)-1-(oxidocarbonyl)ethyl)]-[*N*-(methylamino)methyl-*N*-(ethoxycarbonylmethyl)]phosphinate (12d). Yield 94%, m.p. 182 °C (dec.). Found (%): C, 41.26; H, 4.72. $C_{13}H_{18}NNa_2O_7P$. Calculated (%): C, 41.39; H, 4.81. ^1H NMR (D_2O), δ: 0.95 (t, 3 H, CH_3, $^2J_{HH}$ = 7.3 Hz); 2.26 (s, 3 H, C(6)H_3); 2.48—2.90 (m, 5 H, C(1)H, C(2)H_2, C(5)H_2); 3.23 (s, 2 H, C(7)H_3); 3.84 (q, 2 H, CH_2O, $^2J_{HH}$ = 7.3 Hz); 5.82—5.93 (m, 1 H, $C_{Fur}H$); 6.09—6.12 (m, 1 H, $C_{Fur}H$); 7.04—7.10 (m, 1 H, $C_{Fur}H$).

$^{13}C\{^1H\}$ NMR (D_2O), δ: 13.59 (s, Me); 31.64 (s, C(2)); 34.91 (d, C(1), $^1J_{PC}$ = 89.6 Hz); 43.28 (d, C(6), $^3J_{PC}$ = 6.6 Hz); 52.98 (d, C(5), $^1J_{PC}$ = 104.8 Hz); 57.23 (d, C(7), $^3J_{PC}$ = 7.8 Hz); 58.98 (s, CH_2O); 106.94 (s, C_{Fur}); 109.95 (s, C_{Fur}); 140.89 (s, C_{Fur}); 151.28 (d, C(3), $^3J_{PC}$ = 6.4 Hz); 170.40 (s, C=O); 180.34 (d, C(4), $^2J_{PC}$ = = 14.2 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 32.34 (s).

Disodium (2-oxopyrrolidin-1-ylmethyl)[2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphinate (12e). Yield 93%, m.p. 178 °C (dec.). Found (%): C, 41.64; H, 4.02. $C_{12}H_{14}NNa_2O_6P$. Calculated (%): C, 41.75; H, 4.09. ^1H NMR (D_2O), δ: 1.42—1.58 (m, 2 H, CH_2); 1.76—1.89 (m, 2 H, CH_2C=O); 2.30—2.52 (m, 2 H, C(2)H_2); 2.80—3.29 (m, 5 H, C(1)H, C(5)H_2, C(6)H_2); 5.78—5.87 (m, 2 H, 2 C_{Fur}H); 6.91—6.94 (m, 1 H, C_{Fur}H). $^{13}C\{^1H\}$ NMR (D_2O), δ: 16.95 (s, CH_2); 28.86 (s, CH_2C=O); 32.04 (s, C(2)); 35.91 (d, C(1), $^1J_{PC}$ = 87.8 Hz); 40.33 (d, C(5), $^1J_{PC}$ = 104.6 Hz); 47.09 (d, C(6), $^3J_{PC}$ = 6.6 Hz); 107.05 (d, C_{Fur}, $^4J_{PC}$ = 5.9 Hz); 109.70 (s, C_{Fur}); 140.98 (s, C_{Fur}); 148.06 (d, C(3), $^3J_{PC}$ = 8.9 Hz); 169.85 (d, NC=O, $^3J_{PC}$ = 15.5 Hz); 176.34 (d, C(4), $^2J_{PC}$ = 4.1 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 30.94 (s).

Disodium (2-oxoazepan-1-ylmethyl)[2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphinate (12f). Yield 94%, m.p. 183 °C (dec.). Found (%): C, 44.92; H, 4.80. $C_{14}H_{18}NNa_2O_6P$. Calculated (%): C, 45.05; H, 4.86. ^1H NMR (D_2O), δ: 1.13—1.40 (m, 6 H, 3 CH_2); 2.12—3.74 (m, 9 H, CH_2C=O, C(1)H, C(2)H_2, C(5)H_2, C(6)H_2); 5.82—5.99 (m, 2 H, 2 C_{Fur}H); 6.93—7.01 (m, 1 H, C_{Fur}H). $^{13}C\{^1H\}$ NMR (D_2O), δ: 22.47 (s, CH_2); 27.11 (s, CH_2); 27.40 (s, CH_2); 29.29 (s, CH_2C=O); 33.04 (s, C(2)); 36.23 (d, C(1), $^1J_{PC}$ = 89.1 Hz); 45.52 (d, C(5), $^1J_{PC}$ = 104.8 Hz); 50.15 (s, C(6)); 107.41 (d, C_{Fur}, $^4J_{PC}$ = 4.2 Hz); 110.02 (s, C_{Fur}); 141.18 (s, C_{Fur}); 148.58 (d, C(3), $^3J_{PC}$ = 8.9 Hz); 170.19 (d, NC=O, $^3J_{PC}$ = 15.2 Hz); 175.44 (s, C(4)). $^{31}P\{^1H\}$ NMR (D_2O), δ: 31.02 (s).

Trisodium 3-oxido-3-oxopropyl(2-carboxyethyl)[2-(2-furyl)-1-(oxidocarbonyl)ethyl]phosphinate (13). Yield 94%, m.p. 195 °C (dec.). Found (%): C, 35.11; H, 2.95. $C_{10}H_{10}Na_3O_7P$. Calculated (%): C, 34.97; H, 3.01. ^1H NMR (D_2O), δ: 1.42—1.70 (m, 2 H, C(7)H_2); 2.06—2.32 (m, 2 H, C(8)H_2); 2.38—2.66 (m, 2 H, C(2)H_2); 3.26—3.42 (m, 1 H, C(1)H); 5.81—5.88 (m, 1 H, C_{Fur}H); 5.93—6.00 (m, 1 H, C_{Fur}H); 6.95—7.01 (m, 1 H, C_{Fur}H). $^{13}C\{^1H\}$ NMR (D_2O), δ: 22.97 (d, C(7), $^1J_{PC}$ = 92.1 Hz); 27.03 (s, C(8)); 32.30 (s, C(2)); 36.73 (d, C(1), $^1J_{PC}$ = = 90.1 Hz); 106.85 (d, C_{Fur}, $^4J_{PC}$ = 5.4 Hz); 110.02 (s, C_{Fur}); 140.15 (s, C_{Fur}); 148.12 (d, C(3), $^3J_{PC}$ = 6.9 Hz); 170.38 (d, C(4), $^2J_{PC}$ = 15.4 Hz); 175.89 (d, C(9), $^2J_{PC}$ = 16.2 Hz). $^{31}P\{^1H\}$ NMR (D_2O), δ: 34.37 (s).

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