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Cyclopropanecarbaldehyde with Two Protected Hydroxymethyl Groups

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Abstract—3,5-Dioxabicyclo[5.1.0]octane-8-carbaldehyde was synthesized in three steps, including initial stereoselective *exo*-cyclopropanation of 1,3-dioxacycloheptene-5-ene with ethyl diazoacetate. The subsequent phosphorylation according to Abramov and Kabachnik–Fields gave the corresponding α-hydroxy(acetoxy)- and α-aminocyclopropylmethylphosphonates with protected hydroxymethyl moieties at the three-membered ring.

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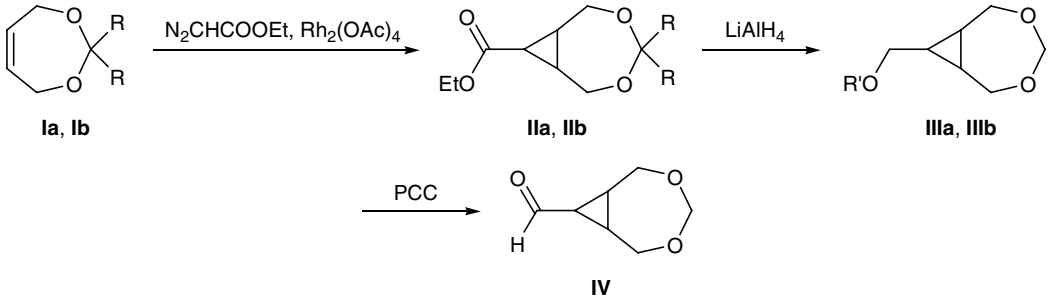
Cyclopropane derivatives are widely used in synthetic practice [1]; in particular, compounds with formyl [2] and hydroxymethyl fragments [3–9], especially those in which both these fragments are present, attract much interest. In the present communication we report on the synthesis of a 2,3-bis(hydroxymethyl)-cyclopropane-1-carbaldehyde derivative in which the hydroxymethyl moieties are protected via cyclic acetalization. The synthesis of 3,5-dioxabicyclo[5.1.0]octane-8-carbaldehyde (**IV**) is illustrated by Scheme 1.

Compounds **II**–**IV** can be regarded as 1,2,3-trisubstituted cyclopropane derivatives; their stereochemical structure is set even at the cyclopropanation stage. Detailed examination of the ^{1}H and ^{13}C NMR spectra of the reaction mixtures containing acetal **IIa** showed high stereoselectivity of the process which leads to the formation of a single diastereoisomer. The configuration of adduct **IIa** was proved by analysis of spin–spin coupling constants for the CH protons in the three-membered ring. The 8-H signal is a distinct triplet with a coupling constant ^3J of 4.9 Hz [10]. Therefore, acetal **IIa** has *exo* configuration, otherwise (*endo* isomer) a ^3J value of larger than 7 Hz should be expected [11]. The use of $Rh_2(OAc)_4$ instead of $CuSO_4$ [12] in the synthesis of acetal **IIa** allowed us to perform the reaction under very mild conditions and improve the yield of the target product to 70%. Acetal **IIa** was subjected to hydrolysis [9] without additional purification. It is known that the *exo*-stereoselectivity in the cyclopropanation of cycloheptene (which is a carbocyclic analog of **IIa**) is much lower (38%) [13]. Almost complete stereoselectivity was also observed in the cyclopropanation of cyclohexanone acetal **Ib** with ethyl diazoacetate: The coupling constant ^3J for 8-H in adduct **IIb** was 4.8 Hz.

In the synthesis of aldehyde **IV** we used only acetal **IIa**. Its reduction with $LiAlH_4$ gave 74% of alcohol **IIIa** whose structure was confirmed by the transforma-

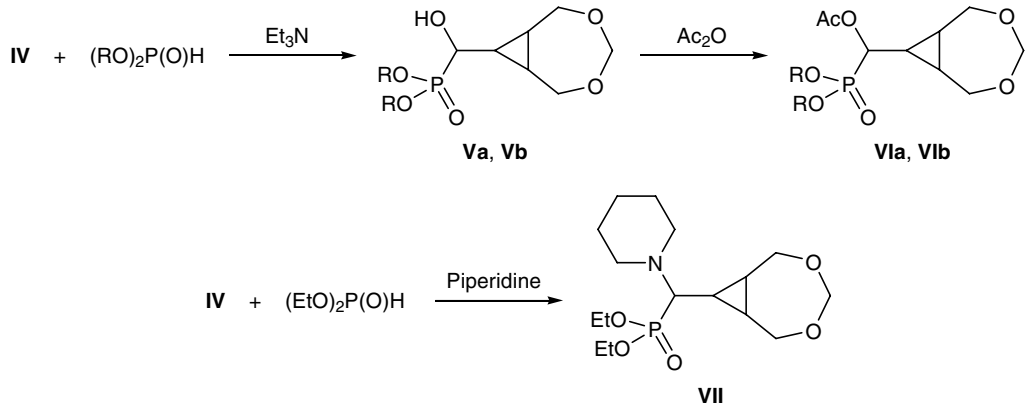
Scheme 1.



Ia, **Ib** **IIa**, **IIb** **IIIa**, **IIIb** **IV**

I, **II**, R = H (**a**); RR = $(CH_2)_5$ (**b**); **III**, R′ = H (**a**), 3,5-$(O_2N)_2C_6H_3CO$ (**b**); PCC is pyridinium chlorochromate.

Scheme 2.



V, **VI**, R = Me (**a**), *i*-Pr (**b**).

tion into ester **IIIb**, as well as by the ^{1}H and ^{13}C NMR spectra. By oxidation of alcohol **III** with pyridinium chlorochromate (PCC) we obtained aldehyde **IV** in 65% yield. The structure of **IV** was proved by chemical transformations: its phosphorylation with dialkyl phosphonates lead to the formation of α-hydroxy(acetoxy)- and α-aminophosphonates **V**–**VII** (Scheme 2).

Functionally substituted phosphonates attract interest from the viewpoint of bioorganic and medicinal chemistry due to broad spectrum of their biological activity [14–17], which gives an impetus to systematic studies in this line.

EXPERIMENTAL

The ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on a Varian Unity-300 spectrometer at 300, 75.43, and 121.42 MHz, respectively. The chemical shifts were measured relative to the solvent signals ($CHCl_3$, δ 7.27 ppm; $CDCl_3$, δ_C 77.7 ppm) or 85% H_3PO_4 (^{31}P, external reference). Thin-layer chromatography was performed on Silufol UV-254 plates, and silica gel L, 60–120 μm, was used for column chromatography. The mass spectra (electron impact, 70 eV, and chemical ionization, reactant gas pentane) were obtained on a Finnigan MAT-212 mass spectrometer with direct sample admission into the ion source.

Ethyl 3,5-dioxabicyclo[5.1.0]octane-*exo*-8-carboxylate (IIa). A solution of 0.88 g (7.85 mmol) of ethyl diazoacetate in 10 ml of methylene chloride was added dropwise over a period of 4.5 h under stirring to a mixture of 1.95 g (19.4 mmol) of 1,3-dioxacyclohept-5-ene [18] and a catalytic amount (0.03 g) of dirhodium tetraacetate in 10 ml of methylene chloride, maintaining the temperature at 45°C (bath). The mixture was then heated for 0.5 h more, cooled, and passed through a column charged with anhydrous magnesium sulfate. Fractional distillation gave 1.06 g (73%) of compound **IIa** as a colorless liquid with bp 83°C (0.2 mm), n_D^{24} = 1.4590; the product crystallized on storage in a refrigerator, mp 27–28°C; published data [12]: bp 87–115°C (0.1 mm). ^{1}H NMR spectrum, δ, ppm: 1.25 t (3H, CH_3, ^3J = 7.2 Hz), 1.76 m (2H, 1-H, 7-H), 2.1 t (1H, 8-H, ^3J = 4.9 Hz), 3.92–4.25 m (5H, 2-H, 4-H, 6-H), 4.11 q (2H, $CH_3C\mathbf{H}_2$, ^3J = 7.2 Hz), 4.89 (1H, 4-H, a part of *AB* quartet, ^2J = –7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 15.14 (CH_3), 19.76 (C^8), 28.14 (C^1, C^7), 60.89 ($\mathbf{C}H_2CH_3$), 69.08 (C^2, C^6), 100.35 (C^4), 173.59 (CO).

Ethyl 3,5-dioxabicyclo[5.1.0]octane-4-spiro-1′-cyclohexane-*exo*-8-carboxylate (IIb) was synthesized in a similar way from 3.68 g (19.8 mmol) of 7,12-dioxaspiro[5.6]dodec-9-ene [18] and 1 g (8.9 mmol) of ethyl diazoacetate. The product was recrystallized from ethanol–hexane (1:3). Yield 3.08 g (55%), mp 42–43°C. ^{1}H NMR spectrum, δ, ppm: 1.19 t (3H, CH_3, ^3J = 7.2 Hz), 1.28–163 m (10H, CH_2), 1.68 m (2H, 1-H, 7-H), 1.92 t (1H, 8-H, ^3J = 4.8 Hz), 3.78–4.2 m (4H, 2-H, 6-H), 4.10 q (2H, $C\mathbf{H}_2CH_3$, ^3J = 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 15.34 (CH_3); 23.98 (C^8); 28.21 (C^1, C^7); 23.72, 26.78, 33.28, 34.71 (CH_2); 59.92 ($\mathbf{C}H_2CH_3$); 60.64 (C^2, C^6); 102.94 (C^4), 173.26 (CO). Found, %: C 66.12; H 8.75. $C_{14}H_{22}O_4$. Calculated, %: C 66.14; H 8.66.

3,5-Dioxabicyclo[5.1.0]oct-*exo*-8-ylmethanol (IIIa). A solution of 4 g (21.5 mmol) of compound **IIa** in 40 ml of anhydrous THF was added dropwise over a period of 1.5 h under vigorous stirring to a mixture of 0.6 g (15.8 mmol) of $LiAlH_4$ and 50 ml of anhydrous THF. The mixture was heated for 8 h at 70°C, treated

with water, filtered, and extracted with diethyl ether. The extract was dried over Na_2SO_4 and subjected to fractional distillation under reduced pressure to isolate 2.3 g (74.2%) of acetal **IIIa** with bp 109°C (0.15 mm). ^{1}H NMR spectrum, δ, ppm: 1.14 m (2H, 1-H, 7-H), 1.43 m (1H, 8-H), 3.20 br.s (1H, HO), 3.43 d (2H, HOC**H**$_2$, ^3J = 6.4 Hz), 3.79 m (2H, 2-H), 4.08 m (2H, 6-H), 4.43 and 4.69 (2H, 4-H, *AB* quartet, $^2J_{AB}$ = –7.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 23.49 (C^1, C^7), 24.39 (C^8), 65.13 (C^2, C^6), 71.78 ($HOCH_2$), 100.98 (C^4).

3,5-Dioxabicyclo[5.1.0]oct-*exo*-8-ylmethyl 3,5-dinitrobenzoate (IIIb). A solution of 0.16 g (0.69 mmol) of 3,5-dinitrobenzoyl chloride in 5 ml of methylene chloride was added dropwise under stirring to a mixture of 0.1 g (0.69 mmol) of alcohol **IIIa** and 1 ml of pyridine in 20 ml of methylene chloride. After 2 h, the mixture was washed with water, the organic phase was separated and dried over Na_2SO_4, the solvent was removed, and the residue was recrystallized from ethanol. Yield 0.21 g (89%), mp 102–103°C. ^{1}H NMR spectrum, δ, ppm: 1.37 m (2H, 1-H, 7-H), 1.85 m (1H, 8-H), 4.10 m (4H, 2-H, 6-H), 4.37 and 4.92 (2H, 4-H, *AB* quartet, $^2J_{AB}$ = –7.2 Hz), 4.43 d (2H, $COOCH_2$, ^3J = 7.2 Hz), 9.20 s (2H, H_{arom}), 9.27 s (1H, H_{arom}). Found, %: C 50.03; H 4.51; N 8.36. $C_{14}H_{14}N_2O_8$. Calculated, %: C 49.71; H 4.17; N 8.28.

3,5-Dioxabicyclo[5.1.0]octane-*exo*-8-carbaldehyde (IV). A solution of 2 g (14 mmol) of hydroxy acetal **IIIa** in 20 ml of methylene chloride was added under stirring to a solution of 4.5 g (21 mmol) of pyridinium chlorochromate in 15 ml of methylene chloride. The mixture was stirred for 1.5 h at room temperature (it turned dark) and extracted with diethyl ether (3×30 ml). The extract was passed through a thin layer of silica gel and evaporated under reduced pressure. Yield 1.29 g (65.5%), light yellow oily substance. ^{1}H NMR spectrum, δ, ppm: 1.71 m (2H, 1-H, 7-H), 2.02 m (1H, 8-H), 3.84 m (4H, 2-H, 6-H), 3.99 and 4.65 (2H, 4-H, *AB* quartet, $^2J_{AB}$ = –7.1 Hz), 8.95 d (1H, CHO, ^3J = 5.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 27.92 (C^1, C^7), 29.60 (C^8), 69.31 (C^2, C^6), 100.66 (C^4), 172.27 (CHO).

Dimethyl 3,5-dioxabicyclo[5.1.0]oct-*exo*-8-yl(hydroxy)methylphosphonate (Va). A solution of 0.44 g (4 mmol) of dimethyl phosphonate in 10 ml of diethyl ether was added dropwise to a solution of 0.5 g (3.5 mmol) of compound **IV** and 0.3 ml of triethylamine in 30 ml of diethyl ether. After 12 h, the precipitate was filtered off and recrystallized from diethyl ether–methylene chloride (9:1). Yield 0.69 g (78%), mp 101°C. ^{1}H NMR spectrum, δ, ppm: 1.36 m (2H, 1-H, 7-H), 1.58 m (1H, 8-H), 3.05 br.s (1H, OH), 3.58 d.d (1H, PCH, $^2J_{PH}$ = 7.2, ^3J = 7.2 Hz), 3.78 d (3H, CH_3, $^3J_{PH}$ = 10.5 Hz), 3.80 d (3H, CH_3, $^3J_{PH}$ = 10.5 Hz), 4.02 m (4H, 2-H, 6-H), 4.32 and 4.79 (2H, 4-H, *AB* quartet, ^2J = –7.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 20.70 br.s (C^8), 22.87 d (C^1 or C^7, $^3J_{PC}$ = 4.5 Hz), 23.20 d (C^7 or C^1, $^3J_{PC}$ = 10.5 Hz), 53.79 and 54.12 (CH_3), 69.26 d (PC, $^1J_{PC}$ = 162.4 Hz), 70.80 and 71.09 (C^2, C^6), 100.83 (C^4). ^{31}P NMR spectrum: δ_P 26.83 ppm. Found, %: C 43.03; H 7.09; P 12.06. $C_9H_{17}O_6P$. Calculated, %: C 42.86; H 6.80; P 12.28.

Diisopropyl 3,5-dioxabicyclo[5.1.0]oct-*exo*-8-yl-(hydroxy)methylphosphonate (Vb) was synthesized in a similar way from 0.5 g (3.5 mmol) of compound **IV** and 0.66 g (4 mmol) of diisopropyl phosphonate. The product was isolated by chromatography using toluene–diethyl ether (9:1) as eluent. Yield 0.91 g (84%), R_f 0.23 (ethyl acetate), mp 68°C. ^{1}H NMR spectrum, δ, ppm: 1.27 d (6H, CH_3, $^4J_{PH}$ = 6.1 Hz), 1.30 d (6H, CH_3, $^4J_{PH}$ = 7.4 Hz), 1.31 m (2H, 1-H, 7-H), 1.54 m (1H, 8-H), 2.04 br.s (1H, OH), 3.41 d.d (1H, PCH, $^2J_{PH}$ = 6.2, ^3J = 7.2 Hz), 3.86 m and 4.05 m (2H each, 2-H, 6-H), 4.38 and 4.72 (2H, 4-H, *AB* quartet, ^2J = –6.9 Hz), 4.64–4.75 m (2H, POCH). ^{13}C NMR spectrum, δ_C, ppm: 21.90 br.s (C^8), 23.10 d (C^1 or C^7, $^3J_{PC}$ = 3.9 Hz), 23.43 d (C^7 or C^1, $^3J_{PC}$ = 10.4 Hz), 23.57 d (CH_3, $^3J_{PC}$ = 4.5 Hz), 24.72 d (CH_3, $^3J_{PC}$ = 3.4 Hz), 24.76 d (CH_3, $^3J_{PC}$ = 3.4 Hz), 69.78 d (PC, $^1J_{PC}$ = 164.4 Hz), 71.17 and 71.49 (C^2, C^6), 71.66 d and 71.88 d (COP, $^2J_{PC}$ = 7.4 Hz), 100.93 (C^4). ^{31}P NMR spectrum: δ_P 22.89 ppm. Found, %: C 51.03; H 8.39; P 10.26. $C_{13}H_{25}O_6P$. Calculated, %: C 50.64; H 8.17; P 10.05.

Dimethoxyphosphoryl(3,5-dioxabicyclo[5.1.0]-oct-*exo*-8-yl)methyl acetate (VIa). A mixture of 0.2 g (0.79 mmol) of phosphonate **Va** and 0.085 g (0.83 mmol) of acetic anhydride in 1.2 ml of anhydrous pyridine was stirred for 18 h at room temperature. Fractional distillation under reduced pressure gave 0.2 g (86%) of compound **VIa** as a yellowish oily substance, bp 127°C (0.02 mm). ^{1}H NMR spectrum, δ, ppm: 1.33 m (2H, 1-H, 7-H), 1.68 m (1H, 8-H), 2.11 s (3H, CH_3CO), 3.78 d and 3.82 d (3H each, CH_3OP, $^3J_{PH}$ 10.5 Hz), 3.95–4.98 m (4H, 2-H, 6-H), 4.22 and 4.86 (2H, 4-H, *AB* quartet, ^2J = –7.4 Hz), 4.81 d.d (1H, PCH, $^2J_{PH}$ = 9.5, ^3J 9.7 Hz). ^{13}C NMR spectrum, δ_C, ppm: 17.68 br.s (C^8), 21.33 (**C**H_3CO), 23.88 d (C^1 or C^7, $^3J_{PC}$ = 2.1 Hz), 24.77 d (C^7 or C^1, $^3J_{PC}$ = 12.3 Hz), 54.05 d and 54.14 d (COP, $^2J_{PC}$ = 10.6 Hz), 69.94 and 70.16 (C^2, C^6), 70.61 d (PC, $^1J_{PC}$ = 171.0 Hz), 100.62 (C^4), 170.39 d (C=O, $^3J_{PC}$ = 6.7 Hz). ^{31}P NMR spec-

trum: δ_P 26.40 ppm. Found, %: C 44.43; H 6.40. $C_{11}H_{19}O_7P$. Calculated, %: C 44.90; H 6.51.

Diisopropoxyphosphoryl(3,5-dioxabicyclo[5.1.0]-oct-*exo*-8-yl)methyl acetate (VIb) was synthesized in a similar way from 0.2 g (0.65 mmol) of phosphonate **Vb** and 0.07 g (0.69 mmol) of acetic anhydride. The product was isolated by column chromatography on silica gel using methylene chloride–petroleum ether (8:2) as eluent. Yield 0.21 g (93%), yellow oily substance, R_f 0.22 (ethyl acetate). ^{1}H NMR spectrum, δ, ppm: 1.27 d and 1.29 d [12H, (C**H**$_3$)$_2$CH, $^4J_{PH}$ = 10.5 Hz], 1.28 m (2H, 1-H, 7-H), 1.62 m (1H, 8-H), 2.06 s (3H, CH_3CO), 3.93–3.97 m (4H, 2-H, 6-H), 4.24 and 4.79 (2H, 4-H, *AB* quartet, ^2J = –7.2 Hz), 4.68 d.d (1H, PCH, $^2J_{PH}$ = 9.8, ^3J = 9.9 Hz), 4.65–4.75 m (2H, CHO). ^{13}C NMR spectrum, δ_C, ppm: 18.76 br.s (C^8); 21.45 (**C**H$_3$CO); 24.07 d (C^1 or C^7, $^3J_{PC}$ = 4.3 Hz); 24.44 d (C^7 or C^1, $^3J_{PC}$ = 10.5 Hz); 24.50 d, 24.71 d, 24.74 d, and 24.87 d [(**C**H$_3$)$_2$CH, $^3J_{PC}$ = 7.5 Hz); 70.21 and 70.50 (C^2, C^6); 71.39 d (PC, $^1J_{PC}$ = 164.4 Hz); 72.02 d and 72.08 d (CHOP, $^2J_{PC}$ = 7.4 Hz); 100.70 (C^4); 170.46 d (C=O, $^3J_{PC}$ = 7.2 Hz). ^{31}P NMR spectrum: δ_P 17.95 ppm. Mass spectrum, *m*/*z* (I_{rel}, %): 350 (6) $[M]^+$, 349.140 (21) $[M - H]^+$, 307 (7), 279 (7), 265 (23), 249 (7), 223 (7), 207 (14), 177 (11), 176 (8), 166 (15), 154 (6), 149 (8), 148 (7), 138 (8), 135 (9), 125 (10), 124 (43), 123 (6), 113 (7), 109 (13). $C_{15}H_{27}PO_7$. Calculated: $[M - H]^+$ 349.142.

Diethyl 3,5-dioxabicyclo[5.1.0]oct-*exo*-8-yl-(piperidino)methylphosphonate (VII). A mixture of 0.2 g (1.4 mmol) of compound **IV**, 0.23 g (1.7 mmol) of diethyl phosphonate, and 0.15 g (1.7 mmol) of piperidine was heated for 20 min at 85°C. The mixture was cooled, 0.1 g of anhydrous Na_2SO_4 was added, and the mixture was evacuated and subjected to column chromatography using toluene–ethyl acetate (8:2) as eluent. Yield 0.4 g (82%), light brown oily substance, R_f 0.17 (ethyl acetate). ^{1}H NMR spectrum, δ, ppm: 1.15 m (2H, 1-H, 7-H); 1.22 t and 1.24 t (3H each, C**H**$_3$CH$_2$, ^3J = 7.1 Hz); 1.30–1.36 m, 1.42–1.48 m, 2.50–2.61 m, and 2.79–2.86 m (10H, CH_2); 1.58 m (1H, 8-H); 2.22 d.d (1H, PCH, $^2J_{PH}$ = 18.3, ^3J = 10.7 Hz); 3.89–4.01 m (4H, 2-H, 6-H); 4.01–4.13 m (4H, CH_2OP); 4.30 and 4.70 (2H, 4-H, *AB* quartet, ^2J = –7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 15.99 br.s (C^8); 16.92 d and 16.98 d (**C**H$_3$CH$_2$, $^3J_{PC}$ = 4.7 Hz); 24.07 d (C^1 or C^7, $^3J_{PC}$ = 8.6 Hz); 24.62 d (C^7 or C^1, $^3J_{PC}$ = 15.9 Hz); 27.00, 52.82, and 52.92 (CH_2); 61.64 d and 63.09 d (POCH$_2$, $^2J_{PC}$ = 7.2 Hz); 67.42 d (PS, $^1J_{PC}$ = 156.0 Hz); 70.61 and 70.95 (C^2, C^6); 100.68 (C^4). ^{31}P NMR spectrum: δ_P 25.04 ppm. Mass spectrum (chemical ionization, 200 eV): *m*/*z* 348 (I_{rel} 100%) $[M - H]^+$. $C_{16}H_{30}O_5P$. Calculated: *M* 347.396.

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