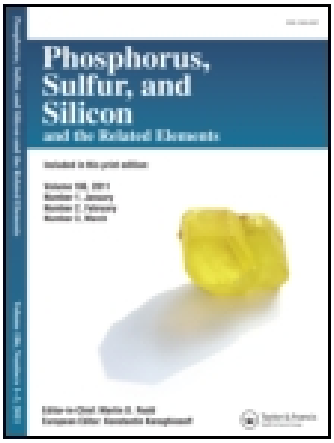

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
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Amidoalkylation of Phosphorus Trichloride with Acetamide and Functionalized Cyclic Ketones-Evidence of Dominating Role of Side-Reactions

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Accepted author version posted online: 14 May 2014.Published online: 04 Aug 2014.

To cite this article: Zofia A. Dziuganowska, Iga Andrasiak, Katarzyna Ślepokura & Paweł Kafarski (2014) Amidoalkylation of Phosphorus Trichloride with Acetamide and Functionalized Cyclic Ketones-Evidence of Dominating Role of Side-Reactions, Phosphorus, Sulfur, and Silicon and the Related Elements, 189:7-8, 1068-1075, DOI: 10.1080/10426507.2014.905573

To link to this article: http://dx.doi.org/10.1080/10426507.2014.905573

Phosphorus, Sulfur, and Silicon, 189:1068–1075, 2014

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426507.2014.905573

AMIDOALKYLATION OF PHOSPHORUS TRICHLORIDE WITH ACETAMIDE AND FUNCTIONALIZED CYCLIC KETONES-EVIDENCE OF DOMINATING ROLE OF SIDE-REACTIONS

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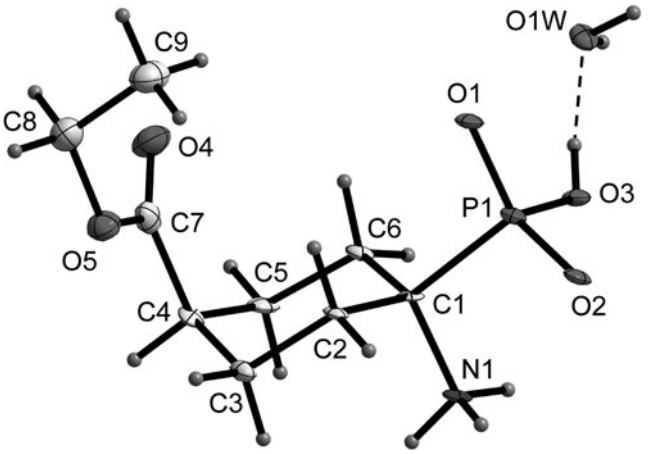
GRAPHICAL ABSTRACT



Abstract *The course of the reaction between phosphorus trichloride, acetamide and ethyl oxoalkanecarboxylates was studied in terms of the production of side-products. When ethyl 2-oxocycloalkane- and 4-oxocyclohexanecarboxylates were used as substrates, not only mixtures of the expected products—aminophosphonocycloalkanecarboxylic acids and their ethyl esters but also the formation of three side-products, namely 1-aminocycloalkanephosphonic acids (products of decarboxylation of the latter ones) and 1-aminoethane-1,1-diphosphonic and 1-hydroxyethane-1,1diphosphonic acids (products of the reaction of*

Received 30 January 2014; accepted 18 February 2014.

This work is dedicated to Prof. Louis D. Quin on the occasion of his 86th birthday.

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substrate and solvent with phosphonylating agent) were observed. Ethyl 2-oxocyclooctanecarboxylate afforded the expected 2-amino-2-pohosphonocyclooctanecarboxylic acid in minute amounts. For comparison reactions of 1-butyloxocarbonyloxopiperidines afforded the desired aminopiperidinephosphonic acids in good yields and without formation of side-products.

Keywords Phosphonic analogues aspartic and glutamic acids; aminophosphonates; Oleksyszyn-Soroka reaction; side products

INTRODUCTION

Aminoalkanephosphonic acids are broadly defined as analogues of amino acids, which carboxylic group is replaced by phosphonic acid or related moiety. Today they attract considerable interest because of their diverse and useful biological activities[1–3] and consequently, a wide variety of synthetic methods for their preparations have been elaborated.[1] Three-component reaction of amidoalkylation of trivalent phosphorus compounds is perhaps one of the simplest and thus the most commonly used for the preparation of structurally diverse aminophosphonates. Final procedure of amidoalkylation successfully introduced by Oleksyszyn[4–6] and further modified by Soroka[7,8] has been found as a method of choice. The mechanism of this useful reaction is still no fully understood. Previously, we have shown that the reaction between phosphorus trichloride, acetamide, and cyclic oxocarboxylates, upon application of procedure introduced by Soroka[7] yielded side products which derive from the reaction of acetic acid (solvent) and acetamide (substrate) with phosphonylating agent.[8]

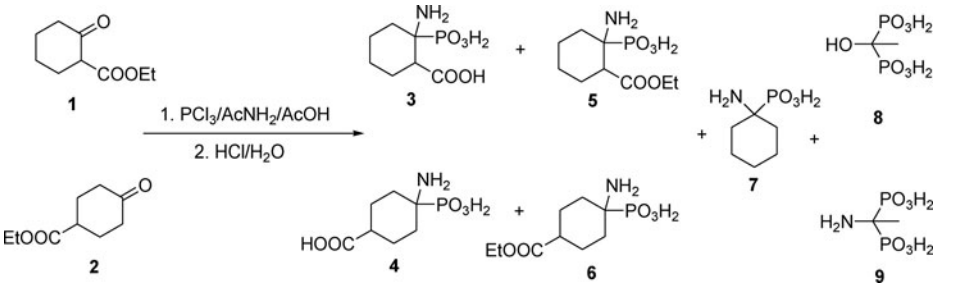
RESULTS AND DISCUSSION

Reexamination of the reaction between ethyl 2-oxocyclohexanecarboxylate (compound **1**) and 4-oxocyclohexanecarboxylate (compound **2**) with acetamide and phosphorus trichloride, followed by acid hydrolysis, surprisingly gave quite different results than those described earlier.[8] Small change in procedure of recovery of crude product enabled better determination of the composition of these products. Thus, the examination of ^{31}P NMR spectra of crude reaction mixtures clearly showed the presence of four products (Scheme 1): desired aminophosphonocycloalkanecarboxylic acids (compounds **3** and **4**), their C-ethyl esters (compounds **5** and **6**), 1-aminocyclohexanephosphonic acid formed by decarboxylation of compounds **3** and **4** (compound **7**), and a product of reaction of phosphorus trichloride with acetic acid, namely 1-hydroxy-1,1-diphosphonic acid (compound **8**). The product of reaction of phosphonylating agent with acetamide—1-aminoethane-1,1-diphosphonate (compound **9**)-crystallized together with ammonium chloride. The presence of these compounds was also confirmed by mass spectrometry (see Experimental).

Fractional crystallization enabled us to obtain pure compounds 3, 4 (84:16 ratio of isomers), 5 (82:18 ratio of isomers), 6 (pure *cis*-isomer), and 9. Structure of compound of *cis* configuration was determined by X-ray crystallography (Figure 1).

As seen from Figure 1, this compound exists as zwitterion and crystallizes with one molecule of water. The ratio of *cis*:*trans* isomers in the crude product was determined as 64:36 basing on ^{1}H NMR studies.

Reaction of ethyl 2-oxocyclooctanecarboxylate (compound **10**, Scheme 2) provided 50% of crude product composed of compound **9** (8% of yield), hydroxyl



Scheme 1

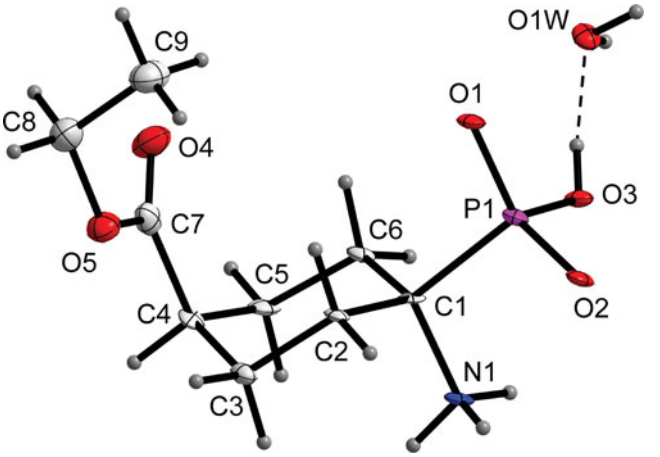
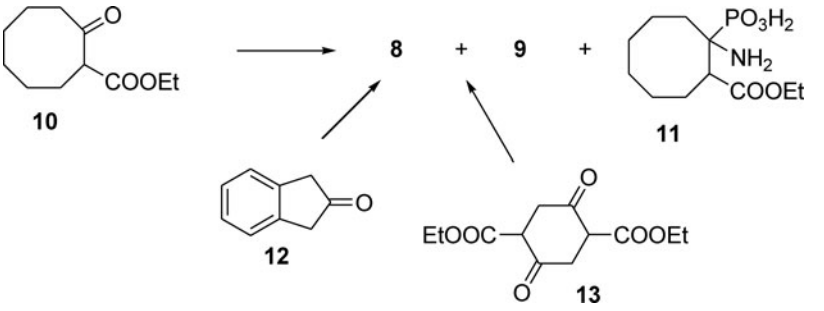


Figure 1 Asymmetric unit of **6·H$_2$O** crystal, showing the atom-numbering scheme and the symmetry-independent O—H···O hydrogen bond (thin dashed line; see Table 1.). Displacement ellipsoids are drawn at the 50% probability level.

derivative **8** (42% of yield) and minute amounts (0.2% of yield) of ethyl 2-amino-2-phosphonocyclooctanecarboxylate (compound **11**).

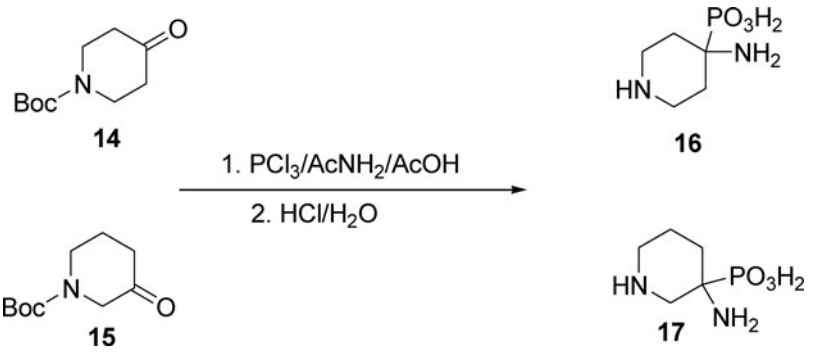


Scheme 2

Reactions of 2-indanone (compound **12**) and diethyl 2,5-dioxo-1,4-dicarboxylate (compound **13**) did not provide desired aminophosphonates (Scheme 2). In the case of

compound **12** hydroxyphosphonate **8** was formed in 100% yield, whereas in the case of compound **13** the mixture of **8** and **9** in a ratio of 15:85 was obtained in 89% of total yield.

For comparison, the same procedure had been applied using 1-butyloxocarbonyl-4-oxopiperidine (compound **14**) and 1-butyloxocarbonyl-3-oxopiperidine (compound **15**) as substrates. In this case, the expected products (compounds **16** and **17**) have been obtained (Scheme 3) with good yields and we had not observed formation of side-products **8** and **9**.



Scheme 3

One of these compounds (**16**) crystallized as hydrochloride and its structure was additionally supported by crystallography (Figure 2). Also in this case the compound crystallizes as a monohydrate.

EXPERIMENTAL

All solvents and reagents were purchased from commercial suppliers (Aldrich, Sigma, Merck, POCh), were of analytical grade and were used without further purification. Unless otherwise specified solvents were removed with a rotary evaporator. Infrared spectra were measured on a 1600 FT-IR Perkin-Elmer spectrometer. NMR experiments were performed

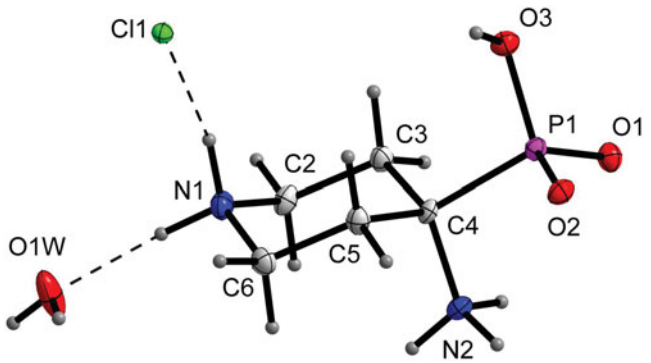


Figure 2 Asymmetric unit of **[16$^+$Cl$^-$]·H$_2$O** crystal, showing the atom-numbering scheme and the symmetry-independent N-H···O and N-H···Cl hydrogen bonds (thin dashed lines; see Table 1). Displacement ellipsoids are drawn at the 50% probability level.

Table 1 Hydrogen-bond geometry for **6·H$_2$O** and **[16$^+$Cl$^-$]·H$_2$O** (Å, °)

D-H···*A*	*D*-H	H···*A*	*D*···*A*	*D*-H···*A*
6·H$_2$O				
O3-H3···O1*W*	0.82(4)	1.69(4)	2.510(3)	174(4)
N1-H3*NA*···O1^{i}	0.92(3)	1.93(3)	2.806(4)	160(3)
N1-H2*NB*···O2^{ii}	1.01(3)	1.75(4)	2.729(4)	165(3)
N1-H1*NC*···O3^{iii}	0.93(3)	1.97(3)	2.864(4)	161(3)
C3-H3*A*···O1*W*^{i}	0.99	2.60	3.191(4)	118
O1*W*-H1*W*···O1^{iv}	0.84	1.90	2.731(3)	172
O1*W*-H2*W*···O2^{v}	0.84	1.88	2.707(3)	169
[16$^+$Cl$^-$]·H$_2$O				
O3-H3···O2^{vi}	0.92(7)	1.64(7)	2.549(3)	170(6)
N1-H1*NA*···Cl1	0.99	2.18	3.107(3)	156
N1-H1*NB*···O1*W*	0.99	1.95	2.787(4)	140
N2-H2*NA*···O1^{vii}	0.98(5)	1.79(5)	2.736(4)	161(4)
N2-H2*NB*···O2^{viii}	0.97(4)	1.80(4)	2.742(4)	162(4)
N2-H2*NC*···Cl1^{ix}	0.93(6)	2.23(6)	3.153(3)	170(5)
C2-H2*B*···Cl1^{ix}	0.99	2.83	3.718(4)	150
C3-H3*A*···O3^{x}	0.99	2.55	3.332(4)	136
C6-H6*A*···Cl1^{ix}	0.99	2.84	3.731(4)	150
C6-H6*B*···Cl1^{xi}	0.99	2.84	3.646(4)	139
O1*W*-H1*W*···Cl1^{xi}	0.84	2.43	3.260(3)	170
O1*W*-H2*W*···O1^{xii}	0.84	2.11	2.912(4)	159

Symmetry codes: (i) $x, -y+1/2, z+1/2$; (ii) $-x+1, y-1/2, -z+1/2$; (iii) $-x+1, y+1/2, -z+1/2$; (iv) $x, y-1, z$; (v) $-x+1, -y, -z$; (vi) $-x+1, -y+1, -z+1$; (vii) $-x+1, y-1/2, -z+3/2$; (viii) $-x+1, y+1/2, -z+3/2$; (ix) $x, -y+3/2, z+1/2$; (x) $-x+1, -y+2, -z+1$; (xi) $-x+2, -y+1, -z+1$; (xii) $x+1, y, z$.

on a Bruker DRX AVANCE^{TM} 300 MHz and Bruker AVANCE^{TM} 600 MHz spectrometers. Measurements were made in $CDCl_3$ (99.5 at. % D) or D_2O (99.8 at. % D) solutions at temperature 300 K, all solvents were supplied by Dr Glaser AG (Basel, Switzerland). Chemical shifts are reported in parts per million relative to TMS or 85% H_3PO_4 used as external standards, and coupling constants are reported in Hertz. Melting points were determined on an Electrothermal 9200 apparatus and are reported uncorrected. Electrospray mass spectra were recorded at Chemistry Department of University of Wroclaw using Finnigan Mat TSQ 700 Electrospray mass spectrometer.

Crystal Structure Determination

The crystallographic measurements were performed at 80(2) or 100(2) K on a κ-geometry Xcalibur *R* (ω scan) or Xcalibur *PX* (ω scan) automated four-circle diffractometer with graphite-monochromatized MoKα or CuKα radiation (Table 2). Data were corrected for Lorentz and polarization effects. Data collection, cell refinement, and data reduction and analyses were carried out with CrysAlis CCD and CrysAlis RED, respectively.[9] Empirical (multi-scan) absorption correction was applied to the data with the use of CrysAlis RED. The structures were solved by direct methods using the SHELXS-97 program[10] and refined on F^2 by a full-matrix least-squares technique using SHELXL-2013[10] with anisotropic thermal parameters for non-H atoms. All H atoms in **6·H$_2$O** and **[16$^+$Cl$^-$]·H$_2$O** were found in difference Fourier maps, and in the final refinement cycles, the C-bonded H atoms and those form NH_2 group in **16** were repositioned in their calculated positions and refined using a riding model, with C—H = 0.98–1.00 Å, N—H = 0.99 Å, and with

Table 2 Crystallographic data for **6·H$_2$O** and **[16$^+$Cl$^-$]·H$_2$O**

	6·H$_2$O	**[16$^+$Cl$^-$]·H$_2$O**
Empirical formula	$C_9H_{20}NO_6P$	$C_5H_{16}ClN_2O_4P$
Formula weight (g mol^{-1})	269.23	234.62
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$ (No. 14)	$P2_1/c$ (No. 14)
a (Å)	14.777(4)	10.734(3)
b (Å)	6.292(2)	6.653(2)
c (Å)	13.113(4)	14.154(4)
β (°)	94.75(3)	90.39(3)
V (Å^3)	1215.0(6)	1010.8(5)
Z	4	4
T (K)	80(2)	100(2)
D_{calcd} (g cm^{-3})	1.472	1.542
μ (mm^{-1})	0.24	4.80
Crystal size (mm)	0.22 × 0.04 × 0.01	0.42 × 0.22 × 0.10
Radiation type, λ (Å)	MoKα, 0.71073	CuKα, 1.5418
θ range (°)	2.8–28.5	4.1–78.0
Index ranges	$-10 \le h \le 19$	$-13 \le h \le 12$
	$-8 \le k \le 4$	$-7 \le k \le 8$
	$-16 \le l \le 17$	$-17 \le l \le 16$
T_{min}/T_{max}	0.918/1.000	0.327/1.000
Reflections collected	5602	4387
Independent reflections	2925	2083
Observed reflections [$I > 2\sigma(I)$]	1779	2006
*R*int	0.071	0.053
Data/restraints/parameters	2925/2/173	2083/2/137
*R*1 [$F_o^2 > 2\sigma(F_o^2)$]	0.070 [a)]	0.066 [a)]
*wR*2 (all data)	0.117 [a)]	0.184 [a)]
GOF = S	1.00	1.12
$\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	0.43/–0.52	0.64/−0.67

[a)] $R1 = \sum ||F_o| - |F_c|| / \sum |F_o|$; $wR2 = \sqrt{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]}$. Detailed values of the weighting scheme (w) in each system are given in the crystallographic information files (CIF) provided as Supporting Information.

$U_{iso}(H) = 1.2U_{eq}(C)$ for CH, CH$_2$, NH$_2$, or $1.5U_{eq}(C)$ for CH$_3$. H atoms from NH$_3$ group in **6** were refined freely. Water H atoms in **6·H$_2$O** and **[16$^+$Cl$^-$]·H$_2$O** were refined isotropically with the O—H distances restrained to 0.840(2) Å, and then were constrained to ride on their parent atoms (AFIX 3 instruction in SHELXL-2013). The structure plots were prepared with DIAMOND.[11]

CCDC 983797 and 983798 for **6·H$_2$O** and **[16$^+$Cl$^-$]·H$_2$O**, respectively contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, B2 1EZ, UK; fax +44 1223 336033; or deposit@ccdc.cam.ac.uk).

1. Amidoalkylation-General Procedure

Reaction was carried out by a small modification of the procedure described by Soroka.[12] Thus, acetamide (0.2 mole) was dissolved in acetic acid (40 mL) and cooled in

ice-bath. Then, acetyl chloride (0.1 mole) was added with cooling and the formation of crystalline by-product was observed. After 15 min ketone was added and the mixture kept for 30 min in ice-bath and then left for a day at room temperature. Then, the mixture was cooled once more in ice-bath and phosphorus trichloride (0.1 mole) was added. The resulting mixture was kept in the bath for 30 min, then allowed to warm to room temperature and finally heated for 1 h at 70–75°C. Evaporation of volatile components of the reaction mixture resulted in oily product, which was refluxed for 8 h in concentrated hydrochloric acid (100 mL). Then, acid was evaporated in vacuo and the resulting product dissolved in ethanol (50 mL), left for completeness of precipitation of ammonium chloride accompanying by 1-aminoethane-1,1-dipohosphonate ammonium salt, which was filtered off and ethanol was evaporated under reduced pressure. The obtained oily residue was dissolved in ethanol (50 mL) and aminophosphonate precipitated by addition of one droplet of pyridine. Then, ^{31}P NMR spectra of crude products were recorded and the fractional crystallization of the resulting mixture was carried out. Yields of single products one were estimated on the basis of ^{31}P NMR and total weight of crude product.

1.1. Reaction of ethyl 2-oxocyclohexanecarboxylate as Substrate. Yields of single products in crude product were: 16% of compound 5 ($\delta = 13.43$ ppm), 32% of compound 3 ($\delta = 15.29$ ppm); 4% of compound 8 ($\delta = 19.01$ ppm) and 3% of compound 7 ($\delta = 22.24$ ppm). Fractional crystallization afforded:

1-aminocyclohexanephosphonic acid (7) and 2-amino-2-phosphonocyclohexane carboxylic acid (3) of physicochemical data identical as reported in the literature.[4,8] HRMS for compound 3 (H_2O, TOF MS ESI$^+$): found 224.0688 [MH$^+$], $C_7H_{15}NO_5P$ requires 224.1723.

Ethyl 2-amino-2-phosphonocyclohexanecarboxylate (5): yield 30%; mp. 224°C with decomposition; ^{31}P NMR (D_2O): $\delta = 15.29$ and 15.23 ppm (82:18 molar ratio); ^{1}H NMR (D_2O): $\delta = 1.23$ (t, $J = 7.1$ Hz, $POCH_3$, minor isomer), 1.32 (t, $J = 7.1$ Hz, $POCH_3$, major isomer), 1.40–1.58 (m, 4H, CH_2), 1.60–1.83 (m, 4H, CH_2), 1.80–1.95 (m, 2H, CH_2), 2.62 (m, $J = 7.0$ Hz, 1H, CH), 2.96 (m, OCH_2, major), 3.58 (q, $J = 7.1$ Hz, OCH_2, minor) ppm; HRMS (H_2O, TOF MS ESI$^+$): found 252.1122 [MH$^+$], $C_9H_{19}NO_5P$ requires 252.2256.

1.2. Reaction of ethyl 4-oxocyclohexanecarboxylate as Substrate. Yields of single products in crude solid were: 24% of compound 6 ($\delta = 13.43$ ppm), 18% of compound 4 ($\delta = 15.29$ and 15.30 ppm, (84:16 molar ratio)); 8% of compound 8 ($\delta = 19.01$ ppm) and 10% of compound 7 ($\delta = 22.24$ ppm). Fractional crystallization afforded 1-aminocyclohexanephosphonic acid (7) and 4-amino-4-phosphonocyclohexanenecarboxylic acid (4), which physicochemical data are in accordance with the literature.[4,8] HRMS for compound 4 (H_2O, TOF MS ESI$^+$): found 224.0743 [MH$^+$], $C_7H_{15}NO_5P$ requires 224.1723, (TOF MS ESI$^-$) found 222.0535 [M$^-$], $C_7H_{13}NO_5P$ requires 222.1564.

Ethyl *cis*-4-amino-4-phosphonocyclohexanenecarboxylate (6): mp. 225°C with decomposition ^{31}P NMR (D_2O): $\delta = 16.56$ ppm; ^{1}H NMR (D_2O): $\delta = 1.13$ and 1.14 (t, $J = 7.1$ Hz, 3H, OCH_2CH_3), 1.65–1.77 (m, 2H, CH_2), 1.91–2.01(m, 4H, CH_2), 2.20-2.29 (m, 2H each, CH_2), 2.53–2.60 (m, 1H, CH), 3.60 and 3.61 (q, J= 7.1 Hz, 2H, OCH_2) ppm. HRMS (H_2O, TOF MS ESI$^+$): found 252.1010 [MH$^+$], $C_9H_{19}NO_5P$ requires 252.2256, (TOF MS ESI$^-$) found 250.0842 [M$^-$], $C_9H_{17}NO_5P$ requires 250.2098.

1.3. Reaction of ethyl 4-oxocyclooctanecarboxylate as Substrate. Yields of single products in crude solid were: 0.2% of compound 11 (mixture of two isomers), 8% of compound 9 ($\delta = 12.89$ ppm); 42% of compound 8 ($\delta = 19.33$ ppm).

Ethyl 2-amino-2-phosphonocyclooctanecarboxylate (11): mp. 227°C with decomposition; ^{31}P NMR (D_2O): $\delta = 24.72$ and 24.97 (83:17 molar ratio); ^{1}H NMR (D_2O): $\delta = 1.04$

(t, $J = 7.1$ Hz, 3H, $POCH_3$), 1.18–1.38 (m, 2H, CH_2), 1.54–1.75 (m, 8H, CH_2), 2.16–2.20 (m, 2H, CH_2), 2.50–2.54 (m, 1H, CH), 3.50 (q, $J = 7.1$ Hz, OCH_2, major), 3.505 (q, $J = 7.1$ Hz, OCH_2, minor).

1.4. 4-Aminopiperidine-4-phosphonic acid (16). 89% of yield; mp. 200°C with decomposition; ^{31}P NMR (D_2O): $\delta = 13.25$ ppm; ^1H NMR (D_2O): $\delta = 1.90–2.01$ (m, 2H, CH_2), 2.27–2.30 (m, 2H, CH_2), 3.19–3.24 (m, 2H, CH_2), 3.40–3.46 (m, 2H, CH_2); ^{13}C NMR (D_2O): $\delta = 27.08$, 39.64, 39.70, 51.15 (d, $J = 141.1$ Hz, CP). HRMS (H_2O, TOF MS ESI$^+$): found 181.0738 [MH^+], $C_5H_{14}N_2O_3P$ requires 181.1507.

1.5. 3-Aminopiperidine-3-phosphonic acid (17). 100% of yield; mp. 250°C with decomposition; ^{31}P NMR (D_2O): $\delta = 8.80$ and 8.00 ppm (35:65); ^1H NMR (D_2O): $\delta = 1.52–2.19$ (m, 4H, CH_2), 2.85-3.14 (m, 1.35 H, CH_2), 3.30–3.49 (m, 2H, CH_2), 3.79–3.86 (m, 0.65H, CH_2); ^{13}C NMR (D_2O): $\delta = 16.57$, 20.16, 24.54 (d, $J = 6.8$ Hz, major), 24.88 (d, $J = 9.1$ Hz, minor), 43.75 (d, $J = 4.5$ Hz, major), 44.54 (d, $J = 6.0$ Hz, minor), 45.52 (d, $J = 3.8$ Hz, major), 46.64 (d, $J = 3.0$ Hz, minor), 54.36 (d, $J = 129.0$ Hz, CP, major), 53.92 (d, $J = 129.8$ Hz, CP, minor). HRMS (H_2O, TOF MS ESI$^+$): found 181.0742 [MH^+], $C_5H_{14}N_2O_3P$ requires 181.1507.

FUNDING

This work was supported by Polish Ministry of Science and Higher Education.

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