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Dedicated to the Full Member of the Russian Academy of Sciences V.A.Tartakovsky on occasion of his 75th birthday

Syntheses Based on α-Azidooximes: I. Reduction of α-Azidooximes

A. Yu. Sukhorukov, A. N. Semakin, A. V. Lesiv, Yu. A. Khomutova, and S. L. Ioffe

Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, Moscow, 119991 Russia
e-mail: iof@ioc.ac.ru

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Abstract—Convenient procedures were developed for selective and exhaustive reduction of α-azido-oximes that were easily prepared from aliphatic nitro compounds. Nitroalkanes were shown to be convenient precursors of β-functionalized amines (1,2-diamines, iminophosphonates, β azidohydroxyl-amines, α-aminooximes).

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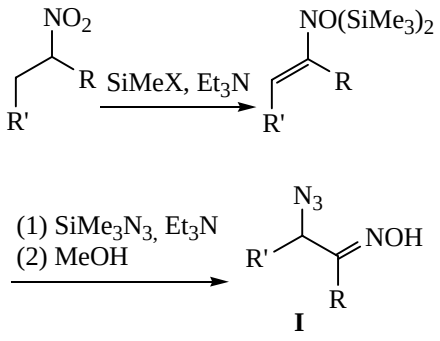
The silylation of aliphatic nitro compounds significantly extends the opportunities of their application to organic synthesis [1]. In particular, this strategy provides a possibility to regard nitroalkanes as fairly convenient precursors of various α-functionalized oxime. Among several procedures developed for preparation of these products the synthesis of poorly known α-azido-oximes **I** is distinguished by efficiency, universality, and simplicity [2] (Scheme 1). In this event α-azido-oximes **I** become promising reagents for preparation of versatile polyfunctional compounds.

We report here on various aspects of selective and exhaustive reduction of oximes **I**. The reduction of the oximimino fragment was attempted using versatile reducers and approaches, like the hydrogenation on Raney nickel (Ni/Ra) [3] or on palladium on carbon [4], lithium aluminum hydride [5], sodium in alcohol [6], titanium(III) chloride [7], and many other. Interestingly, the same reducers can be applied to the reduction of azido group (see, e.g., [8–12]). Analysis of data from [3–12] shows that oximes apparently easier react with hydride reducers whereas in contrast azides are better reduced by the catalytic hydrogenation. In general it may be concluded that the exhaustive reduction of azido-oximes **I** should not be a great problem but the selective reduc-tion of azido or oximimino fragments is quite non-trivial.

Scheme 1.



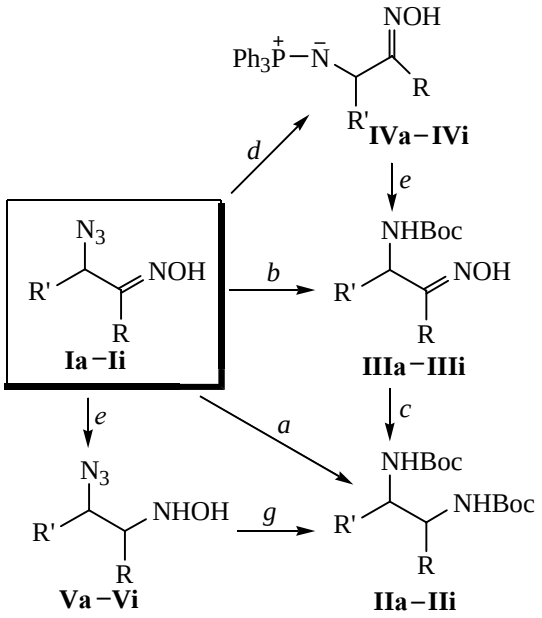
X = Cl, Br, OTf.

Some examples of exhaustive and selective reduction of α-azidooximes and their derivatives were described in [13, 14] but there was not shown a general pattern of this considerably complex process.

Using a fairly representative set of azidooximes **Ia–Ii** that we had previously prepared from nitroalkanes [2] we performed a systematic investigation of their reduction (see the table and Scheme 2).

The complete reduction of oximes **Ia–Ii** → **IIa–IIi** is advantageously performed by the catalytic hydrogenation on Ni/Ra at elevated pressure and high temperature in the presence of di-*tert*-butyl dicarbonate (Boc_2O) necessary for protection of the arising amino groups (Scheme 2, *a*). Special experiments carried out with azidooxime **Ic** showed that the use of Pd/C instead of

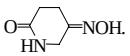
Scheme 2.



R = R' = H (**a**); R = H, R' = Me (**b**); R' = H, R = Me, (**c**), Ph (**d**), Bn (**e**), COOEt (**f**), R = COOMe, R' = Me (**g**); R = H, R' = CH_2OH (**h**), R = H, R' = $(CH_2)_2COOMe$ (**i**). (*a*) H_2, Ni/Ra, 70 at H_2, 80°C, Boc_2O; (*b*) H_2, Ni/Ra, 1 at H_2, 20°C, Boc_2O; (*c*) H_2, Ni/Ra, 50 at H_2, 70°C, Boc_2O; (*d*) PPh_3, Et_2O, 20°C; (*e*) THF, H_2O, then Boc_2O; (*f*) $NaBH_3CN$/AcOH (or HCl); (*g*) H_2, Ni/Ra, 10 at H_2, 20°C, Boc_2O.

Yields of products of azidooximes **Ia–Ii** transformation

Azidooxime	Yield, %			
	II (*a*)	**III** (*e*)	**IV** (*d*)	**V** (*f*)[a]
Ia	58	57	87	–[b] (40)
Ib	50	64	72	30 (56)
Ic	46	90	71	65 (58)
Id	75	65	92	58 (79)
Ie	65	60	45	85 (65)
If	42	–	–[a]	–[d] (–[d])
Ig	85[e]	–[c]	68[e]	21 (–[d])
Ih	65	97	88	–[b] (–[b])
Ii	61	59[g]	79[d]	48 (38)

[a] The yield at the use of HCl instead of AcOH is given in parentheses.
[b] Unidentified products.
[c] Only products of tarring.
[d] No reaction occurred.
[e] Diastereomers mixture, 1.2 : 1.
[f] Unstable ylides.
[g] Compound **IIIi** has a structure  .

Ni/Ra resulted in formation of complex reaction mixtures. The lack of amino groups protection led to a sharp decrease in the yield of target diamines **II** apparently due to their decomposition on the catalyst. The reaction performed under milder conditions resulted in incomplete hydrogenation of initial oximes **I**.

Evidently first the azido group is reduced as has been specially demonstrated by an example of azidooxime **Ic**: Under milder conditions (Scheme 2, *b*) the prevailing reaction product is the corresponding aminooxime **IIIc**.

The catalytic hydrogenation seems more preferable than the application of chemical reducers (e.g., $LiAlH_4$) for the latter are capable of reducing also functional groups (for instance, ester group) present in the initial substrates.

The hydrogenation of substarate **Ig** shows that this reaction is characterized by low diastereoselectivity.

In order to perform the selective reduction of azido group in azidooximes **I** we used the ability of the group to form ylides with triphenylphosphine (**I** → **IV**, Scheme 2, *d*). A mild hydrolysis of iminophosphonates **IV** in most cases led to the formation of the aminooximes **III** (Scheme 2, *e*). The reduction of alkylazides in this manner was known [13, 16]. As a rule this reaction sequence was performed without isolation of the intermediate iminophosphonates. However the isolation of ylides **IV** increased the yield and simplified the purification of target aminooximes **III**.*

The sequence **I** → **IV** → **III** is of a general character and makes it possible to obtain quite different aminooximes **III** in moderate yields. Only azidooximes **If** and **Ig** containing an ester group in the α-position with respect to oximino fragment form an exception.

Basically, aminoximes **III** can be used for catalytic hydrogenation providing diamines **II** (Scheme 2, *c*) but the one-stage hydrogenation of azidooximes **I** (procedure *a*) appears more preferable.

We already mentioned that hydride reducers reduced the azido group slower than the C=N bond of the oximino fragment. Therefore the use of weaker hydride reducer and application of acid catalysis may result in a selective reduction of the oximino fragment in azidooximes **I**. Actually, with $NaBH_3CN$ a selective reduction of the C=N bond became possible for the most of tested azidooximes

* Iminophosphonates can be used in the synthesis of Schiff's bases [17], pyrroles [18], and other compounds.

I (see the table, Scheme 2, *f*).* Acetic and hydrochloric acids are employed as activating acids since they transform the oximino group into a more active iminium cation **A** (Scheme 3).

As a result we obtained and characterized previously unknown β-azidosubstituted hydroxylamines **V**. A strong electronegative substituent, for instance, R = CO_2Alk (**If** and **Ig**) in the α-position to the oximino fragment decreased the nucleophilicity of the nitrogen in the oximino moiety thus hampering the formation of the corresponding activated cations **A**. Therefore no good results were obtained in reaction of $NaBH_3CN$ with azidooximes **If** and **Ig**.

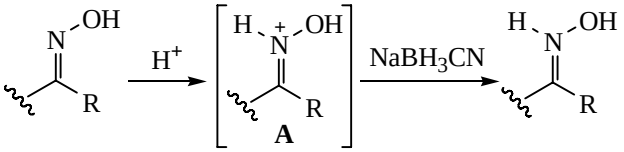
Both acetic and hydrochloric acids can provoke side processes. The former can acylate the arising hydroxylamines to hydroxamic acids that are involved into further reduction (see, e.g., [20]), the latter causes a hydrolysis of the oximino fragment. Therefore the yields of the target hydroxylamines **V** were not necessarily high and depended on the structure of azidooximes **I** subjected to reduction.

We demonstrated by an example of hydroxylamine **Vc** that azidohydroxylamines can be hydrogenated to yield the corresponding Boc-protected diamines **II** (Scheme 2, *g*). Although the conditions of the process are milder than those of the direct exhaustive hydrogenation of azidooximes **I**, the one-stage reaction **I** → **II** is still as a rule preferable.

The composition and structure of compounds **II–IV** were confirmed by elemental analysis and NMR spectroscopy. The configuration of substituted oximes **III** and **IV** was derived from the data of ^1H and ^{13}C NMR spectra applying the rules described before (see, e.g., [21]). Azidohydroxylamines **V** could be isolated by column chromatography without impurities (according to NMR data). However we failed to obtain plausible results of elemental analysis due to the instability of these compounds. The structure of azidohydroxylamines was proved by ^1H and ^{13}C NMR spectra and also by their conversion into substituted diamines **II** (by an example of compound **Vc**). The presence of the azido and hydroximino groups was additionally confirmed by IR spectra. Thus we demonstrated that azidooximes prepared in two stages from nitroalkanes were precursors of β-functionalized amines (1,2-diamines, oximinophosphonates, β-azidohydroxylamines, α-aminooximes) and hydroxylamines.

* $NaBH_3CN$ was successfully used for selective reduction of the C=N bond [19].

Scheme 3.



EXPERIMENTAL

NMR spectra were registered on a spectrometer Bruker AM-300. Chemical shifts (^1H and ^{13}C) are reported in the δ scale and are measured from the solvent signals used as internal reference [22]. IR spectra were recorded on a spectrophotometer Bruker Vector-22. Elemental analyses were carried out in the microanalysis laboratory of the Institute of Organic Chemistry and in the analytical center of the Moscow Chemical Lycee. Melting points were measured on a Koeffler heating block and were reported without correction. TLC was performed on plates purchased from Merck (silica gel with QF-254 indicator). Spots were visualized under UV irradiation and/or using ninhydrine solution in ethanol. The preparative liquid chromatography was done on columns packed with silica gel Merck Kieselgel 60A 230-400 mesh.

Initial α-azidooximes **I** were prepared by published procedures [2]. The catalytic hydrogenation was carried out in a steel pressure reactor (Pike instrument) equipped with a magnetic stirrer.

Hydrogenation of α-azidooximes I into diamines II. *a*. Raney nickel (about 0.05 g in methanol) was added to a solution of an appropriate α-azidooxime **I** (1 mmol) and 0.55 g (2.5 mmol) of Boc_2O in 2.5 ml of methanol. The mixture was hydrogenated at 80°C and 70 at of H_2 for 3 h, then the catalyst was filtered off and the solvent was distilled off in a vacuum. The reaction product was subjected to column chromatograpy on silica gel (eluent hexane–AcOEt, 10:1 → 3:1, for **IIh** 5:1 → 1:1).

***tert*-Butyl N-2-[(*tert*-butoxycarbonyl)amino]-ethylcarbamate (IIa).** mp 120–125°C, R_f 0.54 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.45 s (18H, H_3C), 3.23 m (4H, H_2C), 4.39 br.s (2H, HN). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.3 (CH_3), 41.1 (CH_2), 79.4 ($\underline{C}CH_3$), 156.2 (C=O). All spectral data are consistent with published results [23].

***tert*-Butyl N-2-[(*tert*-butoxycarbonyl)amino]-1-methylethylcarbamate (IIb).** mp 112–115°C (110°C [15]), R_f 0.57 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.13 d (3H, $\underline{H_3}\underline{C}CH$, *J* 6.6 Hz),

1.45 s (18H, H_3C), 3.11 d.d (1H, H_2C, *J* 7.4, 14.0 Hz), 3.19 d.d (1H, H_2C, *J* 5.5, 14.0 Hz), 3.72 m (1H, HC, *J* 6.6 Hz), 4.35 and 4.58 br.s (2H, HN). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 18.5 ($\underline{H_3C}CH$), 28.3 (CH_3), 46.4 (H_2C), 47.4 (HC), 79.3 ($\underline{C}CH_3$), 155.7 and 156.3 (C=O). All spectral data are consitent with published results [15] .

***tert*-Butyl *N*-2-[(*tert*-butoxycarbonyl)amino]-1-phenylethylcarbamate (IId).** mp 132–134°C, R_f 0.70 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.41 s, 1.45 s (18H, H_3C), 3.42 m (2H, H_2C), 4.71 m (1H, HC), 4.84 br.s, 5.43 br.s (2H, HN), 7.22–7.41 m (5H, H_{arom}). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.2 (CH_3), 45.7 (H_2C), 55.9 (HC), 79.4 and 79.6 ($\underline{C}CH_3$), 126.3, 127.5 and 128.6 (C_{arom}), 140.1 (C^i), 155.7 and 156.7 (C=O). Found, %: C 64.24; H 8.59; N 8.10. $C_{18}H_{28}N_2O_4$. Calculated, %: C 64.24; H 8.39; N 8.33.

***tert*-Butyl *N*-1-benzyl-2-[(*tert*-butoxycarbonyl)-amino]ethylcarbamate (IIe)** mp 125–127°C, R_f 0.66 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.42 s and 1.45 s (18H, H_3C), 2.74 d.d (1H, H_2C, *J* 7.3, 14.0 Hz), 2.86 d.d (1H, H_2C, *J* 6.6, 14.0 Hz), 3.20 m (2H, H_2CN), 3.88 m (1H, HC), 4.73 br.s, 5.30 br.s (2H, HN), 7.15–7.37 m (5H, H_{arom}). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.2 (H_3C), 39.0 (H_2C), 44.0 (H_2CN), 52.6 (HC), 79.4 and 79.5 ($\underline{C}CH_3$), 126.3, 128.3 and 129.1 (C_{arom}), 137.4 (Ci), 156.0 and 156.7 (C=O). Found, %: C 65.41; H 8.99; N 7.68. $C_{19}H_{30}N_2O_4$. Calculated, %: C 65.12; H 8.63; N 7.68.

Ethyl 2,3-bis[(*tert*-butoxycarbonyl)amino]-propionate (IIf). mp 113–114°C, R_f 0.62 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 t (3H, $\underline{H_3C}CH_2$, *J* 7.4 Hz), 1.45 s, 1.46 s (18H, H_3C), 3.51 d.d (2H, H_2C, *J* 5.2, 5.9 Hz), 4.22 q (2H, H_2CO, *J* 7.4 Hz), 4.32 d.t (1H, HC, *J* 5.9, 7.4 Hz), 4.78 br.s, 5.31 br.s (2H, HN). ^{13}C NMR spectrum ($CDCl_3$), d, ppm: 14.0 (H_3C), 28.3 ($\underline{H_3C}C$), 42.7 (H_2C), 54.5 (HC), 61.6 (H_2CO), 79.7 and 80.1 ($\underline{C}CH_3$), 155.3 and 155.9 (NC=O), 170.6 (C=O). Found, %: C 53.98; H 8.18; N 8.20. $C_{15}H_{28}N_2O_6$. Calculated, %: C 54.20; H 8.49; N 8.43.

Methyl 2,3-bis[(*tert*-butoxycarbonyl)amino]-butanoate (IIg). Oily substance, R_f 0.62 (hexane–ethyl acetate, 1:1). Diastereomers mixture 1.2:1. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.11 d, 1.18 d (3H, $\underline{H_3C}CH$, *J* 6.6 Hz), 1.42 s, 1.45 s (18H, H_3C), 3.74 s (3H, H_3CO), 4.12 m (1H, $\underline{HC}CH_3$), 4.25 d.d 4.43 d.d (1H, HC, *J* 4.2, 8.3 and *J* 3.8, 8.3 Hz), 4.65 br.s, 4.85 br.s, 5.29 br.s (2H, HN). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 16.5 and 18.1 ($\underline{H_3C}CH$), 28.1 (H_3C), 48.3 and 48.5 ($\underline{HC}CH_3$), 52.0 and 52.1 (H_3CO), 57.6 and 58.0 (HC), 79.4, 79.5, 79.9 and 80.0 ($\underline{C}CH_3$), 154.9, 155.0 and 155.4 (NC=O), 171.0 and 171.1 (C=O). Found, %: C 54.27; H 9.01; N 8.18. $C_{15}H_{28}N_2O_6$. Calculated, %: C 54.20; H 8.49; N 8.43.

***tert*-Butyl *N*-2-[(*tert*-butoxycarbonyl)amino]-1-(hydroxymethyl)ethyl]carbamate (IIh).** mp 94–99°C, R_f 0.29 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.46 s (18H, H_3C), 3.16 br.s (1H, HO), 3.24 d.d (1H, H_2CN, *J* 6.6, 14.7 Hz), 3.34 d.d (1H, H_2CN, *J* 7.4, 14.7 Hz), 3.50–3.77 m (3H, HC and H_2C), 4.89 br.s, 5.02 br.s (2H, HN). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.3 (H_3C), 40.9 (H_2CN), 52.8 (HC), 62.2 (H_2C), 79.6 and 80.1 ($\underline{C}CH_3$), 155.8 and 157.3 (C=O). Found, %: C 53.55; H 9.34; N 9.34. $C_{13}H_{26}N_2O_5$. Calculated, %: C 53.78; H 9.03; N 9.65.

Methyl 4,5-bis[(*tert*-butoxycarbonyl)amino]-pentanoate (IIi). mp 70–73°C {for (*S*)-enantiomer 113–115°C [24]}, R_f 0.52 (hexane–ethyl acetate, 1:1). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.44 s and 1.45 s (18H, H_3C), 1.67 m (1H, H_2CCH_2), 1.84 m (1H, H_2CCH_2), 2.41 d.d (2H, H_2CCH_2, *J* 7.4, 7.6 Hz), 3.16 d.d (1H, H_2C, *J* 6.6, 14.0 Hz), 3.23 d.d (1H, H_2C, *J* 5.2, 14.0 Hz), 3.63 m (1H, HC), 3.68 C (3H, H_3CO), 4.41 br.s, 4.65 br.s (2H, HN). ^{13}C ($CDCl_3$), d, ppm: 27.9 and 30.6 (H_2CCH_2) 28.3 (H_3C), 44.9 (H_2C), 51.37 and 51.43 (HC and H_3CO), 79.4 ($\underline{C}CH_3$), 155.9 and 156.3 (NC=O), 173.5 (C=O). All spectral data are consitent with published results [24].

Preparation of phosphinylides (IV). *d*. Triphenylphosphine (0.26 g, 1 mmol) was added to a solution of an appropriate α-azidooxime **I** (1 mmol) in 3.0 ml of anhydrous ethyl ether. The mixture was vigorously stirred for 6 h at room temperature, the separated colorless precipitate was filtered off and dried in a vacuum. Yields are given in the table.

2-[(Triphenylphosphoranylidene)amino]-acetaldoxime (IVa). mp 120–125°C (decomp.). Mixture of isomers *E* and *Z*, 1.4:1. *E*-isomer. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 3.67 d.d (2H, H_2C, *J* 5.8, 19.8 Hz), 7.24 t (1H, HCN, *J* 5.8 Hz), 7.48–7.72 m (15H_{arom}), 10.22 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 44.0 (H_2C), 128.6 d (C^m, *J* 11.6 Hz), 131.5 (C^p), 131.9 d (C^o, *J* 9.1 Hz), 151.8 d (C=N, *J* 17.6 Hz). *Z*-isomer. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 3.85 d.d (2H, H_2C, *J* 3.7, 17.7 Hz), 6.71 t (1H, HCN, *J* 3.7 Hz), 7.48–7.72 m (15H_{arom}), 10.42 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 40.6 (H_2C), 128.6 d (C^m,

J 11.6 Hz), 131.5 (C^p), 131.9 d (HC^o, *J* 9.1 Hz), 155.3 d (C=N, *J* 18.7 Hz). Found, %: C 71.64; H 5.70; N 8.35. $C_{20}H_{19}N_2OP$. Calculated, %: C 71.84; H 5.73; N 8.38.

***E*-2-[(Triphenylphosphoranylidene)amino]-propanaloxime (IVb).** mp 146–154°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.11 d (3H, H_3C, *J* 6.6 Hz), 3.76 d.d.q (1H, HCN, *J* 6.6, 6.6, 20.6 Hz), 7.13 d (1H, HC, *J* 6.6 Hz), 7.47–7.66 m ($15H_{arom}$), 10.0 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 25.1 d (H_3C, *J* 14.0 Hz), 49.2 (HC), 128.4 d (C^m, *J* 11.5 Hz), 131.3 (C^p), 132.0 d (C^o, *J* 8.7 Hz), 155.5 d (C=N, *J* 11.3 Hz). Found, %: C 72.72; H 6.14; N 7.56. $C_{21}H_{21}N_2OP$. Calculated, %: C 72.40; H 6.08; N 8.04.

***E*-Oxime of 2-[(triphenylphosphoranylidene)-amino]acetone (IVc).** mp 139–141°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.76 s (3H, H_3C), 3.60 d (2H, H_2C, *J* 18.3 Hz), 7.50–7.65 m (15H, H_{arom}), 10.0 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 11.7 (H_3C), 48.9 d (H_2C, *J* 3.6 Hz), 128.67 d (C^m, *J* 12.6 Hz), 131.6 d (C^p, *J* 3.6 Hz), 132.1 d (C^o, *J* 9.0 Hz), 157.9 d (C=N, *J* 19.8 Hz). Found, %: C 72.30; H 6.08; N 8.12. $C_{21}H_{21}N_2OP$. Calculated, %: C 72.40; H 6.08; N 8.04.

***E*-Oxime of 2-[(triphenylphosphoranylidene)-amino]acetophenone (IVd).** mp 139–143°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 4.24 d (2H, H_2C, *J* 18.3 Hz), 7.22–7.40 m (5H, H_{arom}), 7.42–7.66 m (15H, HC_{PPh_3}), 10.39 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 49.0 (H_2C), 126.6, 127.9 and 128.7 (C_{arom}), 128.7 d (C^m, *J* 10.8 Hz), 131.6 (C^p), 132.1 d (C^o, *J* 9.0 Hz), 136.6 (C=N), 157.5 d (C=N, *J* 16.2 Hz). Found, %: C 76.18; H 5.88; N 6.64. $C_{26}H_{23}N_2OP$. Calculated, %: C 76.08; H 5.65; N 6.83.

***E*-Oxime of 1-[(triphenylphosphoranylidene)-amino]-3-phenylacetone (IVe).** mp 84–92°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.55 d (2H, H_2CN, *J* 16.0 Hz), 3.77 s (2H, H_2C), 7.05–7.33 m (5H, H_{arom}), 7.50–7.65 m (15H, HC_{PPh_3}), 10.38 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 30.5 (H_2C), 46.8 (H_2CN), 125.7, 128.1 and 128.9 (C_{arom}), 128.7 d (C^m, *J* 12.6 Hz), 131.5 (C^p), 132.1 d (C^o, *J* 9.0 Hz), 137.9 (CCH_2), 159.5 d (C=N, *J* 21.5 Hz). Found, %: C 76.02; H 5.73; N 6.41. $C_{27}H_{25}N_2OP$. Calculated, %: C 76.40; H 5.94; N 6.60.

***Z*-Methyl 2-(hydroximino)-3-[(triphenylphosphoranylidene)amino]butanoate (IVg).** Unstable at room temperature. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.23 t (3H, H_3C, *J* 6.6 Hz), 3.64 s (3H, H_3CO), 4.48 d.q (1H, HC, *J* 6.6, 19.5 Hz), 7.50–7.75 m (15H, H_{arom}), 10.1 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 22.6 d (H_3C, *J* 9.0 Hz), 48.6 (HC), 51.7 (H_3CO), 129.1 d (C^m, *J* 12.6 Hz), 132.2 d (C^o, *J* 9.0 Hz), 132.6 d (C^p, *J* 3.6 Hz), 155.0 d (C=N, *J* 9.0 Hz), 164.2 (C=O).

***E*-Oxime of 1-hydroxy-3-[(triphenylphosphoranylidene)amino]acetone (IVh).** mp 102–109°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.31 br.s (1H, HOC), 3.80 d (2H, H_2CN, *J* 17.0 Hz), 4.44 s (2H, H_2C), 7.50–7.81 m (15H, H_{arom}), 10.3 br.s (1H, HO). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 46.6 (H_2CN), 58.2 (H_2C), 128.6 d (C^m, *J* 12.6 Hz), 131.6 (C^p), 132.1 d (C^o, *J* 9.0 Hz), 159.6 d (C=N, *J* 18.0 Hz). Found, %: C 68.87; H 5.63, N 7.24. $C_{21}H_{21}N_2O_2P$. Calculated, %: C 69.22; H 5.81; N 7.69.

Preparation of α-aminooximes III. *e*. To a dispersion of 1 mmol of ylide **IV** in 5.6 ml of THF was added 0.054 ml (3 mmol) of water, and the mixture was kept at room temperature for 24 h. Then 0.22 g (1 mmol) of Boc_2O was added, and the mixture was kept for another 24 h. The solution was evaporated in a vacuum, and the residue was subjected to chromatography on silica gel (eluent hexane–AcOEt, 10:1 → 3:1). Yields are given in the table.

1,1-Dimethylethyl (2*E*)-2-(hydroximino)ethylcarbamate (IIIa). mp 74–80°C, R_f 0.46 (hexane–ethylacetate, 1:1). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.44 s (9H, H_3C), 3.89 m (2H, H_2C), 5.08 br.s (1H, HN), 7.43 t (1H, HC, *J* 4.4 Hz), 8.74 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.3 (H_3C), 39.7 (H_2C), 80.0 (CCH_3), 147.6 (HC), 156.0 (C=O). All spectral data are consitent with published results [25].

***tert*-Butyl *N*-[2-(hydroximino)-1-methylethyl]-carbamate (IIIb).** mp 65–70°C, R_f 0.54 (hexane–ethyl acetate, 1:1) Mixture of isomers *E* and *Z*, 3.5:1. *E*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.31 d (3H, H_3CCH, *J* 6.6 Hz), 1.46 s (9H, H_3C), 4.41 m (1H, HC), 4.81 br.s (1H, HN), 7.42 d (1H, HCN, *J* 3.7 Hz), 7.78 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 19.2 (H_3CCH), 28.4 (H_3C), 46.0 (HC), 80.0 (CCH_3), 151.6 (C=O and C=N). *Z*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.31 d (3H, H_3CCH, *J* 6.6 Hz), 1.46 C (9H, H_3C), 4.41 m (1H, HC), 5.53 br.s (1H, HN), 6.69 d (1H, HCN, *J* 3.7 Hz), 7.99 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 17.4 (H_3CCH), 28.4 (H_3C), 43.1 (HC), 80.0 (CCH_3), 151.6 (C=O and C=N). Found, %: C 51.41; H 8.90; N 14.41. $C_8H_{16}N_2O_3$. Calculated, %: C 51.05; H 8.57; N 14.88.

2-(Hydroximino)-1,1-dimethylethylpropylcarbamate (IIIc). mp 86–88°C, R_f 0.55 (hexane–ethyl acetate, 1:1) Mixture of isomers *E* and *Z*, 10:1. *E*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.44 s (9H, H_3C), 1.87 s (3H, H_3CCN), 3.84 d (2H, H_2C, *J* 5.9 Hz), 5.20 br.s (1H, HN), 8.99 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 12.2 ($\underline{H_3C}CN$), 28.4 (H_3C), 44.4 (H_2C), 79.9 ($\underline{C}CH_3$), 155.1 and 156.0 (C=O and C=N). *Z*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.44 s (9H, H_3C), 1.87 s (3H, H_3CCN), 4.04 d (1H, H_2C, *J* 5.2 Hz), 5.73 br.s (1H, HN), 9.74 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 18.0 ($\underline{H_3C}CN$), 28.4 (H_3C), 38.3 (H_2C), 79.9 ($\underline{C}CH_3$), 155.1 and 156.0 (C=O and C=N). Found, %: C 51.03; H 8.50; N 14.90. $C_8H_{16}N_2O_3$. Calculated, %: C 51.05; H 8.57; N 14.88.

***tert*-Butyl *N*-[(2*E*)-2-(hydroximino)-2-phenylethyl]carbamate (IIId).** mp 94–97°C, R_f 0.54 (hexane–ethyl acetate, 1:1). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.44 s (9H, H_3C), 4.44 d (2H, H_2C, *J* 5.9 Hz), 5.37 br.s (1H, HN), 7.40 m, 7.73 m (5H, H_{arom}), 9.80 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.4 (H_3C), 36.6 (H_2C), 79.9 ($\underline{C}CH_3$), 126.9, 128.7 and 129.6 (C_{arom}), 134.2 (Ci), 155.9 and 157.3 (C=N and C=O). Found, %: C 62.46; H 7.55; N 11.16. $C_{13}H_{18}N_2O_3$. Calculated, %: C 62.38; H 7.25; N 11.19.

2-(Hydroximino)-1,1-dimethylethyl-3-phenylpropylcarbamate (IIIe). mp 75–80°C, R_f 0.53 (hexane–ethyl acetate, 1:1). Mixture of isomers *E* and *Z*, 3.75:1. *E*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.43 C (9H, H_3C), 3.74 C (2H, H_2C), 3.85 d (2H, H_2CN, *J* 5.2 Hz), 5.00 br.s (1H, HN), 7.18–7.36 m (5H, H_{arom}), 8.00 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.4 (H_3C), 32.6 (H_2C), 43.0 (H_2CN), 79.8 ($\underline{C}CH_3$), 126.8, 128.7 and 129.1 (H_{arom}), 135.9 (Ci), 155.0 and 155.1 (C=N and C=O). *Z*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.45 C (9H, H_3C), 3.58 C (2H, H_2C), 3.99 d (2H, H_2CN, *J* 6.6 Hz), 5.62 br.s (1H, HN), 7.18–7.36 m (5H, H_{arom}), 9.59 br.s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.4 (H_3C), 37.3 (H_2C), 39.0 (H_2CN), 79.8 ($\underline{C}CH_3$), 126.9, 129.0 and 129.1 (C_{arom}), 135.9 (C^i), 156.0 and 156.1 (C=O and C=N). Found, %: C 63.61; H 7.92; N 10.73. $C_{14}H_{20}N_2O_3$. Calculated, %: C 63.62; H 7.63; N 10.60.

***tert*-Butyl *N*-[3-hydroxy-2-(hydroximino)propyl]carbamate (IIIh).** Oily substance, R_f 0.32 (hexane–ethyl acetate, 1:1). Mixture of isomers *E* and *Z*, 1:1. *E*-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.45 s (9H, H_3C), 3.86 br.s (1H, HO), 4.07 d (2H, H_2CN, J 6.6 Hz), 4.22 s (2H, H_2C), 5.26 br.s (1H, HN), 8.62 br.s (1H, HON). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.3 (H_3C), 35.6 (H_2CN), 61.8 (H_2C), 80.6 ($\underline{C}CH_3$), 156.8 and 157.7 (C=O and C=N). Z-isomer. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.45 s (9H, H_3C), 3.86 br.s (1H, HO), 3.98 d (2H, H_2CN, *J* 6.6 Hz), 4.48 s (2H, H_2C), 5.26 br.s (1H, HN), 8.62 br.s (1H, HON). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.3 (H_3C), 40.3 (H_2CN), 56.6 (H_2C), 80.6 ($\underline{C}CH_3$), 156.9 and 159.0 (C=O and C=N). Found, %: C 47.11; H 7.76; N 14.03. $C_8H_{16}N_2O_4$. Calculated, %: C 47.05; H 7.90; N 13.72.

Tetrahydro-2,5-pyridinedione 5-oxime (IIIi). To a solution of 0.19 g (1 mmol) of azidooxime **Ii** in 3 ml of ether was added at stirring 0.26 g (1 mmol) of triphenylphosphine. The reaction mixture was kept at room temperature for 6 h and then it was evaporated in a vacuum.The residue consisted of an unstable ylide **IVi** as a mixture of *E*- and *Z*-isomers in a ratio 1.1:1. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.29–2.80 m (4H, $\underline{H_2C}H_2CCO$ and $H_2C\underline{H_2C}CO$), 3.61 s, 3.63 s (3H, $\underline{H_3C}OCO$), 3.81 d (2H, $\underline{H_2C}CN$, *E*-isomer, *J* 19.0 Hz), 4.00 d (2H, $\underline{H_2C}CN$, *Z*-isomer, *J* 13.8 Hz), 6.37 br.s (1H, HON), 7.40–7.73 m (15H_{arom}).

Ylide **IVi** obtained was dissolved in a mixture of 4.0 ml of THF and 0.043 ml (2.4 mmol) of water, and the solution was kept at room temperature for 24 h. The separated colorless precipitate was filtered off and dried in a vacuum. Yield of compound **IIIi** 0.06 g (47% calculated on initial azidooxime **Ii**). mp 169–172°C (decomp.). Mixture of isomers *E* and *Z*, 1:1. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.31 m and 2.55 m (4H, H_2CCO and $H_2CC{=}N$), 3.76 s (2H, H_2CNH, *E*-isomer), 3.98 s (2H, H_2CNH, *Z*-isomer), 7.51 s and 7.75 s (1H, HN), 10.69 s and 10.74 s (1H, HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 21.2 ($\underline{H_2C}C{=}N$, *E*-isomer), 25.6 ($H_2CC{=}N$, *Z*-isomer), 29.5 ($\underline{H_2C}CO$, *E*-isomer), 30.5 (H_2CCO, *Z*-isomer), 39.8 (H_2CNH, *Z*-isomer), 43.0 (H_2CNH, *E*-isomer), 151.9 and 153.1 (C=N), 171.6 and 172.6 (C=O). Found, %: C 46.82; H 6.31; N 21.62. $C_5H_8N_2O_2$. Calculated, %: C 46.87; H 6.29; N 21.68.

Synthesis of azidohydroxylamines Vb–Ve, Vg, and Vi. *f*. A dispersion of $NaBH_3CN$ (0.19 g, 3 mmol) in acetic acid (2.2 ml) was added at stirring to a solution of azidooxime **I** (1 mmol) in acetic acid (2.2 ml). The reaction mixture was stirred at room temperature for 2 h and then poured into a mixture of ethyl acetate (100 ml) and saturated solution of Na_2CO_3 (100 ml). The water phase was treated with ethyl acetate (2×50 ml), the combined organic solutions were washed with a saturated sodium chloride solution (50 ml) and dried with Na_2SO_4.

The solution was evaporated in a vacuum, the residue was subjected to chromatography on silica gel.

Synthesis of azidohydroxylamines Va–Ve and Vi. To a solution of 1 mmol of azidooxime **I** in 2 ml of dichloromethane was added 0.10 g (1.6 mmol) of $NaBH_3CN$ and then a mixture of 0.2 ml of concn. HCl and 1.2 ml of MeOH. The reaction mixture was stirred at room temperature for 2 h and then poured into a mixture of ethyl acetate (50 ml) and saturated solution of Na_2CO_3 (50 ml). The water phase was treated with ethyl acetate (2×30 ml), the combined organic solutions were washed with a saturated sodium chloride solution (50 ml) and dried with Na_2SO_4. The solution was evaporated in a vacuum, the residue was subjected to chromatography on silica gel.

1-Azido-2-(hydroxylamino)ethane (Va). Oily substance, R_f 0.17 (hexane–ethyl acetate, 1:1). IR spectrum (KBr), ν, cm^{-1}: 3410 (OH, NH), 3262 (OH, NH), 2103 (N_3), 1442, 1253, 1070, 949. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 3.07 t and 3.56 t (4H, $2H_2C$, *J* 5.2 Hz), 6.05 br.s (2H, HN and HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 48.3 and 52.1 ($2CH_2$).

2-Azido-1-(hydroxylamino)propane (Vb). Oily substance, R_f 0.26 (hexane–ethyl acetate, 1:1). IR spectrum (KBr), ν, cm^{-1}: 3400 (OH, NH), 3258 (OH, NH), 2929, 2104 (N_3), 1448, 1377, 1280, 1101, 1014, 963, 814. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.29 d (3H, H_3C, *J* 6.6 Hz), 2.80 d.d (1H, H_2C, *J* 9.2, 13.2 Hz), 2.99 d.d (1H, H_2C, *J* 3.6, 13.2 Hz), 3.91 m (1H, HC), 5.89 br.s (2H, HN and HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 16.9 (H_3C), 54.5 and 58.1 (HC and H_2C).

1-Azido-2-(hydroxylamino)propane (Vc). Oily substance, R_f 0.27 (hexane–ethyl acetate, 1:1). IR spectrum (KBr), ν, cm^{-1}: 3390 (OH, NH), 3251 (OH, NH), 2921, 2105 (N_3), 1439, 1372, 1261, 1170, 1058, 943, 880. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.11 d (3H, H_3C, *J* 6.6 Hz), 3.14 m (1H, HC), 3.40 d.d (1H, H_2C, *J* 6.6, 11.8 Hz), 3.50 d.d (1H, H_2C, *J* 4.6, 11.8 Hz), 6.30 br.s (2H, HN and HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 15.1 (H_3C), 53.2 and 56.2 (HC and H_2C).

1-[2-Azido-1-(hydroxylamino)ethyl]benzene (Vd). Oily substance, R_f 0.50 (hexane– ethyl acetate, 1:1). IR spectrum (KBr), ν, cm^{-1}: 3418 (OH, NH), 3250 (OH, NH), 2927, 2104 (N_3), 1489, 1450, 1218, 1036, 730, 529. ^1H ($CDCl_3$), δ, ppm: 3.69 d (2H, H_2C, *J* 6.6 Hz), 4.13 t (1H, HC, *J* 6.6 Hz), 7.04 br.s (2H, HN and HO), 728–7.42 m ($5H_{arom}$). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 52.9 (H_2C), 65.2 (HC), 127.8, 128.7 and 128.9 (C_{arom}), 137.2 (C^i).

1-[3-Azido-2-(hydroxylamino)propyl]benzene (Ve). Oily substance, R_f 0.48 (hexane–ethyl acetate, 1:1). IR spectrum (KBr), ν, cm^{-1}: 3420 (OH, NH), 3255 (OH, NH), 2925, 2103 (N_3), 1603, 1496, 1454, 1286, 1077, 745, 700, 533. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.73 d.d (1H, H_2C, *J* 7.2, 13.8 Hz), 2.88 d.d (1H, H_2C, *J* 7.2, 13.8 Hz), 3.21 d.d.d.d (1H, HC, *J* 7.2, 7.2, 5.9 and 4.3 Hz), 3.44 d.d (1H, H_2CN, *J* 5.9, 12.5 Hz), 3.55 d.d (1H, H_2CN, *J* 4.3, 12.5 Hz), 6.75 br.s (2H, HN and HO), 7.14–7.40 m ($5H_{arom}$). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 35.4 (H_2C), 51.2 (H_2CN), 62.1 (HC), 126.9, 128.9 and 129.3 (C_{arom}), 137.4 (C^i).

Methyl 3-azido-2-(hydroxylamino)butyrate (Vg). Oily substance, R_f 0.37 (hexane–ethyl acetate, 1:1). Mixture of diastereomers, 1:1.1. IR spectrum (KBr), ν, cm^{-1}: 3450 (OH, NH), 3258 (OH, NH), 2962, 2103 (N_3), 1714 (CO), 1440, 1269, 1212, 1059, 852. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.36 d and 1.37 d (3H, H_3C, *J* 6.6, 7.2 Hz), 3.61 d and 3.69 d (1H, HCNO, *J* 6.6, 5.3 Hz), 3.82 s (3H, H_3CO), 3.88 2 m (1H, HC), 6.02 br.s (2H, HN and HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 16.0 and 16.7 (H_3C), 52.5 and 52.6 (H_3CO), 56.1 and 56.3 (HC), 68.4 and 68.9 (HCNO), 166.2 and 166.4 (C=O).

Methyl 5-azido-4-(hydroxylamino)pentanoate (Vi). Oily substance, R_f 0.34 (hexane–ethyl acetate, 1:1). IR spectrum (KBr), ν, cm^{-1}: 3450 (OH, NH), 3264 (OH, NH), 2953, 2105 (N_3), 1734 (CO), 1439, 1274, 1203, 1175, 1064, 883, 653. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.71 d.t (1H, H_2CCNO, *J* 5.3, 7.2 Hz), 1.83 d.t (1H, H_2CCNO, *J* 7.2, 7.2 Hz), 2.41 t (2H, H_2CCO, *J* 7.2, 7.2 Hz), 2.88 t.t (1H, HC, *J* 7.2, 5.3 Hz), 3.48 d.d (2H, H_2CN, *J* 7.2, 10.5 Hz), 3.66 s (3H, H_3CO), 6.11 br.s (2H, HN and HO). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 24.0 ($\underline{H_2}C$CNO), 30.7 ($\underline{H_2}$CCO), 51.6 and 51.9 (H_2CN and H_3CO), 60.1 (HC), 174.2 (C=O).

Hydrogenation of azidohydroxylamine Vc. *g*. Raney nickel (about 0.05 g in methanol) was added to a solution of 0.10 g (0.86 mmol) of azidohydroxylamine **Vc** and 0.38 g (1.72 mmol) of BoC_2O in 3 ml of MeOH. The mixture was hydrogenated at room temperature and 10 at of H_2 for 2 h, , then the catalyst was filtered off and the solvent was distilled off in a vacuum. The reaction product was subjected to column chromatograpy on silica gel (eluent hexane–AcOEt, 10:1 → 3:1). Yield of compound **IIc** 0.90 g (43%).

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