Supporting Information.

New Method for the Synthesis of Isoxazoline-*N*-oxides.

Roman A. Kunetsky, Alexander D. Dilman,* Sema L. Ioffe,* Marina. I. Struchkova, Yury A. Strelenko, Vladimir A. Tartakovsky.

N. D. Zelinsky Institute of Organic Chemistry, 119991 Moscow, Leninsky prosp. 47, Russian Federation.

dilman@ioc.ac.ru, iof@ioc.ac.ru

General.

NMR spectra were recorded in $CDCl_3$ on Bruker AM-300, WC-250, AC-200 instruments. Chemical shifts were measured relative to solvent residual peak.[1] Coupling constants are given in Hz. IR spectra were recorded on a Bruker VECTOR22 in KBr tablets.

Starting α-bromonitrocompounds **4a-d**[2a] and **4e**[2b] were prepared by literature procedures.

The cycloaddition reactions of alkenes **5** with α-bromonitrocompounds **4** were performed under dry argon using CH_2Cl_2 freshly distilled from CaH_2.

Phenylbromonitromethane 4c from phenylnitromethane[3a]**:**

The crude product was distilled in vacuum: bp 86-89°C (0.5 Torr). Lit. [4] bp 75°C (0.4 Torr).

Yield 71%.

^{1}H (δ): 6.92 (s, 1H, $CHNO_2$), 7.37-7.59 (m, 3H, CH_{Ph}), 7.60-7.80 (m, 2H, CH_{Ph});

^{13}C (δ): 80.4 (CNO_2), 128.2, 129.2 (o,m-CH_{Ph}), 131.4 (p-CH_{Ph}), 132.9 (i-C_{Ph}).

1-Bromo-2-phenylnitroethane 4d from 2-phenylnitroethane[3b]**:**

The crude product was distilled in vacuum: bp 80-84°C (0.5 Torr). Lit. [5] 106-107 (1 Torr).

Yield 83%.

^{1}H (δ): 3.52 (dd, ^2J = 14.7, ^3J = 5.9, 1H, CH_AH$_B$), 3.77 (dd, ^2J = 14.7, ^3J = 8.1, 1H, CH$_A$$H_B$), 6.07 (dd, ^3J = 8.1, ^3J = 5.9, 1H, $CHNO_2$), 7.17-7.28 (m, 2H, CH_{Ph}), 7.30-7.43 (m, 3H, CH_{Ph});

^{13}C (δ): 43.5 (CH_2), 79.2 ($CHNO_2$), 128.3 (p-CH_{Ph}), 129.1, 129.2 (o,m-CH_{Ph}), 133.3 (i-C_{Ph}).

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(3) Preparation of starting nitro compounds: (a) Kornblum, N.; Larson, H. O.; Blackwood, R. K.; Mooberry, D. D.; Oliveto, E. P.; Graham, G. E. *J. Am. Chem. Soc*. **1956**, *78*, 1497. (b) Bhattacharjya, A.; Mukhopadhyay, R.; Pakrashi, S. C. *Synthesis* **1985**, 886.

(4) Trippett, S.; Walker, D. M. *J. Chem. Soc*. **1960**, 2976.

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Isoxazoline-*N*-oxides 2a-n from α-bromonitrocompounds 4a-d; General procedure

To a solution of bromonitro compound **4** (1 mmol) and TBSCl (1.1 mmol, 166 mg) in CH_2Cl_2 (3.5 ml) at 0°C was added Et_3N (1.1 mmol, 0.15 ml). The silylation was allowed to proceed at room temperature for the time T_1 (see Table S1). Alkene **5** was added and the reaction mixture was kept for time T_2 (see Table S1), quenched by Et_3N (1.5 mmol, 0.21 ml), MeOH (1.5 mmol, 0.06 ml) and stirred for additional 5 min. The most part of the solvent was evaporated in vacuum and the residue was subjected to column chromatography (silica gel, petroleum ether / EtOAc).

5-Phenylisoxazoline-*N*-oxide 2o.

The reaction was run according to general procedure described above. For the work up, instead of quenching the reaction mixture with Et_3N/MeOH, after time T_2 (3 d) the solvent was evaporated in vacuum and the residue was subjected to column chromatography (silica gel, petroleum ether / EtOAc).

Table S1. Reaction time.

Entry	**4**	**5**	**2**	Time	
				T_1	T_2
1	**4a**	**5a**	**2a**	1 d	3 d
2	**4a**	**5b**	**2b**	1 d	7 d
3	**4a**	**5c**	**2c**	1 d	3 d
4	**4a**	**5d**	**2d**	2 h	7 d
5	**4a**	**5e**	**2e**	2 h	3 d
6	**4a**	**5f**	**2f**	2 h	14 d
7	**4b**	**5b**	**2g**	1 d	7 d
8	**4b**	**5d**	**2h**	2 h	7 d
9	**4b**	**5e**	**2i**	2 h	3 d
10	**4c**	**5a**	**2j**	1 d	30 d
11	**4d**	**5b**	**2k**	1 d	7 d
12	**4d**	**5e**	**2l**	2 h	3 d
13	**4d**	**5g**	**2m**	2 h	30 d
14	**4d**	**5h**	**2n**	2 h	14 d
15	**4e**	**5d**	**2o**	5 min	3 d
16	**4a**	**5a**	**8a**	1 d	4 d

The dipolarophiles bearing electron-withdrawing group (i.e. methyl acrylate **5a**, methyl metacrylate **5b**, methyl vinyl ketone **5c**) may undergo Michael reaction with nitro compound. To avoid this problem it is better to allow the silylation of bromonitro compound **4** to go to completion before the addition of such kind of alkenes. This explains the necessity for the stepwise addition of the reagents.

3-Methyl-5-methoxycarbonylisoxazoline-*N*-oxide (2a):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→EtOAc; R_f (Petroleum ether / EtOAc, 1:3) 0.26; oil.

^{1}H (δ): 1.96 (s, 3H, Me), 3.23 (ddq, ^2J = 17.7, ^3J = 5.1, ^4J = 1.5, 1H, CH_AH$_B$), 3.44 (ddq, ^2J = 17.7, ^3J = 10.3, ^4J = 2.2, 1H, CH$_A$$H_B$), 3.79 (s, 3H, MeO), 4.97 (dd, ^3J = 5.1, ^3J = 10.3, 1H, CH);

^{13}C (δ): 11.3 (Me), 37.3 (CH_2), 52.7 (MeO), 70.1 (CH), 110.1 (CN), 169.6 (*C*OOMe);

IR(cm^{-1}): 1657 (C=N), 1745 (C=O).

The neat product decomposes at room temperature within few hours; it can be stored for a long time at –30 ˚C.

3,5-Dimethyl-5-methoxycarbonylisoxazoline-*N*-oxide (2b):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→EtOAc; R_f (Petroleum ether / EtOAc, 1:3) 0.26; mp 33-34^{o}C (Et_2O).

^{1}H (δ): 1.59 (s, 3H, Me), 1.90 (s, 3H, MeCN), 2.95 (d, ^2J = 17.7, 1H, CH_AH$_B$), 3.41 (d, ^2J = 17.7, 1H, CH$_A$$H_B$), 3.73 (s, 3H, MeO);

^{13}C (δ): 11.7 (*Me*CN), 23.3 (Me), 43.8 (CH_2), 53.2 (MeO), 78.3 (C), 112.2 (CN), 171.8 (*C*OOMe);

IR (cm^{-1}): 1657 (C=N), 1743 (C=O).

Anal. Calcd for $C_7H_{11}NO_4$: C, 48.55; H, 6.40; N, 8.09; Found: C, 48.27; H, 6.64; N, 8.12.

5-Acetyl-3-methylisoxazoline-*N*-oxide (2c): The yield of 77 % was determined by ^{1}H NMR spectroscopy with *trans*-stilbene as standard. This product decomposes upon isolation affording after chromatography purified material in only 11 % yield.

Eluent for column chromatography: Petroleum ether / EtOAc (1:3)→EtOAc; R_f (Petroleum ether / EtOAc, 1:3) 0.18; mp 42-45^{o}C (Et_2O).

^{1}H (δ): 1.97 (s, 3H, Me), 2.35 (s, 3H, MeCO), 3.22-3.34 (m, 2H, CH_2), 4.78 (dd, ^3J = 9.8, ^3J = 5.9, 1H, CH);

^{13}C (δ): 11.7 (Me), 26.5 (*Me*CO), 36.3 (CH_2), 76.3 (CH), 111.3 (CN), 206.3 (CO);

IR (cm^{-1}): 1654 (C=N), 1725 (C=O).

The neat product decomposes at room temperature within few hours; it can be stored for a long time at –30 ˚C.

3-Methyl-5-phenylisoxazoline-*N*-oxide (2d):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→EtOAc; R_f (Petroleum ether / EtOAc, 1:3) 0.29; mp 77-78^{o}C (Et_2O).

^{1}H (δ): 2.02 (s, 3H, Me), 3.08 (ddq, ^2J =16.9, ^3J = 7.4, ^4J = 1.5, 1H, CH_AH$_B$), 3.48 (ddq, ^2J = 16.9, ^3J = 9.6, ^4J = 2.2, 1H, CH$_A$$H_B$), 5.58 (dd, ^3J = 9.6, ^3J = 7.4, 1H, CH), 7.28-7.49 (m, 5H, Ph);

^{13}C (δ): 11.8 (Me), 42.2 (CH_2), 75.4 (CH), 112.1 (CN), 125.6, 128.9 (o,m-CH_{Ph}), 128.6 (p-CH_{Ph}), 139.0 (i-C_{Ph});

IR (cm^{-1}): 1641 (C=N).

Anal. Calcd for $C_{10}H_{11}NO_2$: C, 67.78; H, 6.26; N, 7.90; Found: C, 67.80; H, 6.45; N, 8.05.

3-Methyl-5-(2-pyridyl)isoxazoline-*N*-oxide (2e):

The crude product contains *ca*. 10 % of bicyclic adduct which has the retention time similar to that of the desired product. Because of this, the later fractions can be slightly contaminated.

Eluent for column chromatography: EtOAc→MeOH / EtOAc (1:2); R_f (MeOH / EtOAc, 1:4) 0.35; oil.

^{1}H (δ): 1.90 (s, 3H, Me), 3.31 (ddq, ^2J = 17.3, ^3J = 5.6, ^4J = 1.7, 1H, CH_AH$_B$), 3.50 (ddq, ^2J = 17.3, ^3J = 9.7, ^4J = 1.7, 1H, CH$_A$$H_B$), 5.53 (dd, ^3J = 9.7, ^3J = 5.6, 1H, CH), 7.10-7.24 (m, 1H, 5-CH_{Py}), 7.48 (d, ^3J = 7.6, 1H, 3-CH_{Py}), 7.65 (td, ^3J = 7.6, ^4J = 1.7, 1H, 4-CH_{Py}), 8.49 (d, ^3J = 4.9, 1H, 6-CH_{Py});

^{13}C (δ): 11.6 (Me), 39.8 (CH_2), 74.6 (CH), 111.9 (CN), 120.3, 123.2 (3,5-CH_{Py}),136.9, 149.4 (4,6-CH_{Py}), 158.0 (2-C_{Py});

IR (cm^{-1}): 1651 (C=N).

Anal. Calcd for $C_9H_{10}N_2O_2$: C, 60.66; H, 5.66; N, 15.72; Found: C, 60.40; H, 5.52; N, 15.83.

3-Methyl-5-ethoxyisoxazoline-*N*-oxide (2f):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→Petroleum ether / EtOAc (1:3); R_f (Petroleum ether / EtOAc, 1:3) 0.29; oil.

^{1}H (δ): 1.14 (t, ^3J = 7.2, 3H, CH_2*Me*), 1.92 (s, 3H, Me), 2.75 (d, ^2J = 18.4, 1H, CH_AH$_B$), 3.28 (dd, ^2J = 18.4, ^3J = 6.6, 1H, CH$_A$$H_B$), 3.49 (qvint, ^3J = 7.2, 1H, CH_AH$_B$Me), 3.82 (qvint, ^3J = 7.2, 1H, CH$_A$$H_B$Me), 5.36 (d, ^3J = 6.6, 1H, CH);

^{13}C (δ): 11.4 (Me), 14.8 (*Me*CH_2), 41.0 (CH_2), 64.1 (*C*H_2Me), 96.3 (CH), 111.4 (CN);

IR (cm^{-1}): 1651 (C=N).

Anal. Calcd for $C_6H_{11}NO_3$: C, 49.65; H, 7.64; N, 9.65; Found: C, 49.51; H, 7.76; N, 9.68.

3-Ethyl-5-methyl-5-methoxycarbonylisoxazoline-*N*-oxide (2g):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→Petroleum ether / EtOAc (1:3);

R_f (Petroleum ether / EtOAc, 1:3) 0.44; oil.

^{1}H (δ): 1.05 (t, ^3J = 7.7, 3H, CH_2*Me*), 1.60 (s, 3H, MeC), 2.33 (q, ^3J = 7.7, 2H, C*H*$_2$Me), 2.95 (d, ^2J = 17.6, 1H, C*H*$_A$H$_B$), 3.42 (d, ^2J = 17.6, 1H, CH$_A$*H*$_B$), 3.74 (s, 3H, MeO);

^{13}C (δ): 9.5 (CH_2*Me*), 19.4 (*C*H_2Me), 23.1 (Me), 42.0 (CH_2), 53.0 (MeO), 76.7 (C), 116.4 (CN), 171.6 (*C*OOMe);

IR (cm^{-1}): 1650 (C=N), 1743 (C=O).

Anal. Calcd for $C_8H_{13}NO_4$: C, 51.33; H, 7.00; N, 7.48; Found: C, 51.20; H, 7.20; N, 7.27.

3-Ethyl-5-phenylisoxazoline-*N*-oxide (2h):

Eluent for column chromatography: Petroleum ether / EtOAc (2:1)→Petroleum ether / EtOAc (1:3);

R_f (Petroleum ether / EtOAc, 1:1) 0.26; mp 52-54°C (Et_2O).

^{1}H (δ): 1.12 (t, ^3J = 7.6, 3H, Me), 2.44 (q, ^3J = 7.6, 2H, C*H*$_2$Me), 3.04 (ddt, ^2J = 17.0, ^3J = 7.6, ^4J = 1.6, 1H, C*H*$_A$H$_B$), 3.48 (ddt, ^2J = 17.0, ^3J = 9.7, ^4J = 1.4, 1H, CH$_A$*H*$_B$), 5.56 (dd, ^3J = 9.7, ^3J = 7.6, 1H, CH), 7.26-7.46 (m, 5H, Ph);

^{13}C (δ): 9.8 (Me), 19.6 (*C*H_2Me), 40.5 (CH_2), 75.5 (CH), 116.6 (CN), 125.6, 128.9 (o,m-CH_{Ph}), 128.7 (p-CH_{Ph}), 139.2 (i-C_{Ph});

IR (cm^{-1}): 1639 (C=N).

Anal. Calcd for $C_{11}H_{13}NO_2$: C, 69.09; H, 6.85; N, 7.32; Found: C, 69.16; H, 7.01; N, 7.42.

3-Ethyl-5-(2-pyridyl)isoxazoline-*N*-oxide (2i):

Eluent for column chromatography: Petroleum ether / EtOAc (1:2)→EtOAc; R_f (EtOAc) 0.21; oil.

^{1}H (δ): 1.03 (t, ^3J = 7.4, 3H, CH_2*Me*), 2.25-2.46 (m, 2H, C*H*$_2$Me), 3.33 (ddt, ^2J = 17.7, ^3J = 5.9, ^4J = 1.5, 1H, C*H*$_A$H$_B$), 3.51 (ddt, ^2J = 17.7, ^3J = 9.6, ^4J = 1.5, 1H, CH$_A$*H*$_B$), 5.56 (dd, ^3J = 9.6, ^3J = 5.9, 1H, CH), 7.19 (dd, ^3J = 7.4, ^3J = 5.2, 1H, 5-CH_{Py}), 7.52 (d, ^3J = 7.4, 1H, 3-CH_{Py}), 7.67 (td, ^3J = 7.4, ^4J = 1.5, 1H, 4-CH_{Py}), 8.51 (d, ^3J = 5.2, 1H, 6-CH_{Py});

^{13}C (δ): 9.6 (CH_2*Me*), 19.6 (*C*H_2Me), 38.3 (CH_2), 75.0 (CH), 116.3 (CN), 120.3, 123.3 (3,5-CH_{Py}), 137.0, 149.5 (4,6-CH_{Py}), 158.2 (2-C_{Py});

IR (cm^{-1}): 1645 (C=N).

Anal. Calcd for $C_{10}H_{12}N_2O_2$: C, 62.16; H, 6.78; N, 14.50; Found: C, 62.20; H, 6.45; N, 14.74.

3-Phenyl-5-methoxycarbonylisoxazoline-*N*-oxide (2j):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→Petroleum ether / EtOAc (1:3); R_f (Petroleum ether / EtOAc, 1:3) 0.56; mp 123-125°C (Et_2O).

^{1}H (δ): 3.71 (dd, ^2J = 16.6, ^3J = 5.1, 1H, CH_AH$_B$), 3.88 (dd, ^2J = 16.6, ^3J = 10.3, 1H, CH$_A$$H_B$), 3.83 (s, 3H, MeO), 5.13 (dd, ^3J = 10.3, ^3J = 5.1, 1H, CH), 7.33-7.53 (m, 3H, o,p-CH$_{Ph}$), 7.79-7.97 (m, 2H, m-CH$_{Ph}$);

^{13}C (δ): 35.8 (CH_2), 53.1 (MeO), 70.5 (CH), 111.9 (CN), 126.0 (i-C$_{Ph}$), 126.1, 128.8 (o,m-CH$_{Ph}$), 129.8 (p-CH$_{Ph}$), 169.8 (*C*OOMe);

IR (cm^{-1}): 1624 (C=N), 1742 (C=O).

Anal. Calcd for $C_{11}H_{11}NO_4$: C, 59.73; H, 5.01; N, 6.33; Found: C, 59.65; H, 5.01; N, 6.27.

3-Benzyl-5-methyl-5-methoxycarbonylisoxazoline-*N*-oxide (2k):

Eluent for column chromatography: Petroleum ether / EtOAc (2:1)→Petroleum ether / EtOAc (1:3); R_f (Petroleum ether / EtOAc, 1:1) 0.26; mp 67-68°C (Et_2O).

^{1}H (δ): 1.62 (s, 3H, Me), 2.87 (dt, ^2J = 17.4, ^4J = 1.7, 1H, CH_AH$_B$), 3.34 (dt, ^2J = 17.4, ^4J = 1.6, 1H, CH$_A$$H_B$), 3.69 (s, 2H, C$H_2$Ph), 3.77 (s, 3H, MeO), 7.16-7.40 (m, 5H, CH$_{Ph}$);

^{13}C (δ): 23.1 (Me), 32.5 (*C*H$_2$Ph), 42.4 (CH_2), 53.1 (MeO), 76.5 (C), 114.4 (CN), 127.5 (p-CH$_{Ph}$), 128.7, 129.0 (o,m-CH$_{Ph}$), 134.8 (i-C$_{Ph}$), 171.6 (*C*OOMe);

IR (cm^{-1}): 1646 (C=N), 1733 (C=O).

Anal. Calcd for $C_{13}H_{15}NO_4$: C, 62.64; H, 6.07; N, 5.62; Found: C, 62.66; H, 6.14; N, 5.39.

3-Benzyl-5-(2-pyridyl)isoxazoline-*N*-oxide (2l):

Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→Petroleum ether / EtOAc (1:3); R_f (Petroleum ether / EtOAc, 1:3) 0.30; mp 51-52°C (Et_2O).

^{1}H (δ): 3.26 (dd, ^2J = 17.3, ^3J = 5.9, 1H, CH_AH$_B$), 3.44 (dd, ^2J = 17.3, ^3J = 9.7, 1H, CH$_A$$H_B$), 3.63 (d, ^2J = 23.2, 1H, CH_AH$_B$Ph), 3.75 (d, ^2J = 23.2, 1H, CH$_A$$H_B$Ph), 5.58 (dd, ^3J = 9.7, ^3J = 5.9, 1H, CH), 7.08-7.7.42 (m, 6H, CH$_{Ph}$ and 5-CH$_{Py}$), 7.55 (d, ^3J = 7.6, 1H, 3-CH$_{Py}$), 7.71 (td, ^3J = 7.6, ^4J = 1.2, 1H, 4-CH$_{Py}$), 8.52 (d, ^3J = 4.5, 1H, 6-CH$_{Py}$);

^{13}C (δ): 32.5 (*C*H$_2$Ph), 38.5 (CH_2), 75.2 (CH), 114.5 (CN), 120.3, 123.3 (5,3-CH$_{Py}$), 127.3 (p-CH$_{Ph}$), 128.7, 128.9 (o,m-CH$_{Ph}$), 134.8 (i-C$_{Ph}$), 137.0, 149.6 (4,6-CH$_{Py}$), 158.0 (2-CH$_{Py}$);

IR (cm^{-1}): 1638 (C=N).

Anal. Calcd for $C_{15}H_{14}N_2O_2$: C, 70.85; H, 5.55; N, 11.02; Found: C, 70.92; H, 5.60; N, 10.93.

3-Benzyl-5-pentylisoxazoline-*N*-oxide (2m):

Eluent for column chromatography: Petroleum ether / EtOAc (3:1)→Petroleum ether / EtOAc (1:1); R_f (Petroleum ether / EtOAc, 3:1) 0.19; oil.

^{1}H (δ): 0.84 (t, ^3J = 6.6, 3H, Me), 1.12-1.83 (m, 8H, CH_2 groups of pentyl), 2.57 (dd, ^2J = 17.0, ^3J = 8.0, 1H, CH_AH$_B$), 2.97 (dd, ^2J = 17.0, ^3J = 9.0, 1H, CH$_A$$H_B$), 3.66 (s, 2H, C$H_2$Ph), 4.42-4.61 (m, 1H, CH), 7.12-7.36 (m, 5H, CH_{Ph});

^{13}C (δ): 13.7 (Me), 22.3, 24.3, 31.2, 32.4, 34.7, 37.7 (all CH_2), 74.9 (CH), 115.4 (CN), 127.1 (p-CH_{Ph}), 128.6, 128.8 (o,m-CH_{Ph}), 135.1 (i-C_{Ph});

IR(cm^{-1}): 1640 (C=N).

Anal. Calcd for $C_{15}H_{21}NO_2$: C, 72.84; H, 8.56; N, 5.66; Found: C, 72.87; H, 8.52; N, 5.79.

3-Benzyl-5-triethylsilylisoxazoline-*N*-oxide (2m):

Eluent for column chromatography: Petroleum ether / EtOAc (10:1)→Petroleum ether / EtOAc (5:1); R_f (Petroleum ether / EtOAc, 5:1) 0.19; oil.

^{1}H (δ): 0.57-0.73 (m, 6H, 3×MeCH_2), 0.91-1.04 (m, 9H, 3×*Me*CH_2), 2.76-3.10 (m, 2H, CH_2), 3.64 (d, ^2J = 15.6, 1H, CH_AH$_B$Ph), 3.80 (d, ^2J = 15.6, 1H, CH$_A$$H_B$Ph), 4.20 (dd, ^3J = 14.0, ^3J = 9.9, 1H, CH), 7.18-7.42 (m, 5H, CH_{Ph});

^{13}C (δ): 1.5 (CH_2Si), 7.2 (*Me*CH_2), 32.6 (*C*H_2Ph), 35.9 (CH_2), 67.5 (CH), 115.0 (CN), 127.2 (p-CH_{Ph}), 128.7, 128.9 (o,m-CH_{Ph}), 135.5 (i-C_{Ph});

IR(cm^{-1}): 1637 (C=N).

Anal. Calcd for $C_{16}H_{25}NO_2Si$: C, 65.93; H, 8.65; N, 4.81; Found: C, 65.78; H, 8.70; N, 4.79.

5-Phenylisoxazoline-*N*-oxide (2n):

Eluent for column chromatography: Petroleum ether / EtOAc (3:1)→Petroleum ether / EtOAc (1:1); R_f (Petroleum ether / EtOAc, 1:1) 0.24.

^{1}H (δ): 3.05 (ddd, ^2J = 17.7, ^3J = 7.9, ^3J = 2.6, 1H, CH_AH$_B$), 3.46 (ddd, ^2J = 17.7, ^3J = 9.8, ^3J = 2.6, 1H, CH$_A$$H_B$), 5.69 (dd, ^3J = 9.8, ^3J = 7.9, 1H, CHO), 6.10 (t, ^2J = 2.6, 1H, CHN), 7.17-7.54 (m, 5H, CH_{Ph});

^{13}C (δ): 38.0 (CH_2), 78.3 (CHO), 103.8 (CN), 128.8 (p-CH_{Ph}), 125.7, 128.9 (o,m-CH_{Ph}), 138.6 (i-C_{Ph});

The product is fairly prone to decomposition at room temperature. Purified material can be stored at –78 °C.

3,7-Dicarbmethoxy-5-methyl-2,8-dioxa-1-azabicyclo[3,3,0]octane (8a): This product was obtained according to general procedure using 50 eq. of methyl acrylate. Mixture of diastereoisomers 1 : 1. Eluent for column chromatography: Petroleum ether / EtOAc (1:1)→Petroleum ether / EtOAc (1:3); R_f (Petroleum ether / EtOAc, 1:3) 0.40 (for two isomers); mp 68-69^{o}C (Et_2O).

^{1}H (δ): 1.27 (s, Me) and 1.31 (s, Me)(3H), 2.16-2.34 and 2.46-2.69 (m, 2×CH_2)(4H), 3.62-3.70 (m, 6H, 2×MeO), 4.67-4.87 (m, 2H, 2×CH);

^{13}C (δ): 23.2, 23.3 (Me), 39.6, 40.0, 41.2, 41.8 (2×CH_2), 52.2, 52.3, 52.4 (2×CH), 74.9, 77.2, 78.7 (2×MeO), 170.3, 170.9 (2×*C*OOMe);

Anal. Calcd for $C_{10}H_{15}NO_6$: C, 48.98; H, 6.17; N, 5.71; Found: C, 48.64; H, 6.06; N, 5.69.

Silyl nitronate 6a from 1-bromonitroethane:

To a solution of 1-bromonitroethane (5 mmol, 770 mg) and TBSCl (5.5 mmol, 166 mg) in CH_2Cl_2 (17.5 ml) at 0^{o}C was added Et_3N (5.5 mmol, 0.77 ml). After 24 h at room temperature the reaction mixture was evaporated and petroleum ether (30 ml) was added. The mixture was stirred for 10 min, Et_3NHCl was filtered off under argon atmosphere and solvent was evaporated in vacuum. The residue was distilled in a Kugelrohr apparatus: 60-80 ^{o}C (0.5 Torr). Yield 88%.

^{1}H (δ): 0.34 (s, 6H, Me_2Si), 0.96 (s, 9H, Me_3C), 2.50 (s, 3H, Me);

^{13}C (δ): -4.2 (Me_2Si), 17.9 (Me_3*C*), 23.3 (CBr), 25.9 (*Me*$_3$C), 104.6 (CN);

^{29}Si (δ): 30.50.

Anal. Calcd for $C_8H_{18}BrNO_2Si$: C, 35.82; H, 6.76; N, 5.22; Found: C, 35.92; H, 6.99; N, 5.47.