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ASYMMETRIC NITROGEN - 70.* GEMINAL SYSTEMS - 44.* TRIALKOXYAMINES(ORTHONITRITES). SYNTHESIS AND PROPERTIES

VLADIMIR F. RUDCHENKO, SERGEI M. IGNATOV, IVAN I. CHERVIN, REMIR G. KOSTYANOVSKY

Institute of Chemical Physics, Academy of Sciences of the U.S.S.R., Kosygin st. 4, B-334 Moscow, U.S.S.R.

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Abstract - The chlorination of acyclic N,N-dialkoxyamines under the action of tert-BuOCl results in the formation of unstable N-chloro-N,N-dialkoxyamines which in situ react with sodium methoxide to give previously unknown trialkoxyamines (orthonitrites). The properties of the N-chloro-N,N-dialkoxy- and trialkoxyamines have been demonstrated to be similar to those of their carbon analogues: dialkoxyalkylchlorides and orthoesters, respectively.

The ortho-forms of carbonyl compounds, acetals[2] and orthoesters,[3] play an important role in organic chemistry. The nitrogen analogues of the acetals - acyclic,[4,5] mono-,[6] di-,[7] and tricyclic[8] N,N-dialkoxy-N-alkylamines, as well as N,N-dialkoxyamides[9,10] - have recently been studied intensively. During the last period, we have synthesized acyclic[11] and cyclic[12] N,N-dialkoxyamines $(RO)_2NH$ which are the orthoesters of nitrosyl hydride HNO.[13]

The ortho-forms of other oxygenous compounds of nitrogen are represented only by inorganic derivatives - extremly unstable salts of a hypothetic tetraoxyhydrazine $(HO)_2NN(OH)_2$[14] and orthonitrates M_3NO_4(M=K, Na).[15] The orthonitrite structure $N(OM)_3$ of the products obtained by fusion of alkali metal oxides with their nitrites suggested by Zintle[16] was disproved.[17]

The present work deals with the synthesis and properties of trialkoxyamines, i.e. the esters of hypothetic orthonitrous acid $N(OH)_3$(for the preliminary communication see ref.18). We were interested in these compounds due to the following considerations. It is known that inversion barrier of the N atom in amines rises by about 8 kcal/mol when an alkoxy substituent being bonded to this atom.[19] In the case of acyclic N,N-dialkoxy-N-tert-alkylamines the barrier is equal to 22 - 25 kcal/mol, which makes it possible to separate them into antipodes with the asymmetric N atom.[5] However, the half-racemization time of optically active N,N-dialkoxyamines is equal only to several hours at 20 °C.[5] The trialkoxyamines were suggested to possess the superstable N atom pyramid in the open chain.

We have previously developed a preparative method for the synthesis of N,N-dialkoxy-N-alkylamines by alcoholysis of the corresponding N-chloro-N-alkoxyamines.[5] To prepare trialkoxyamines by an analogous scheme, we have synthesized N,N-dialkoxyamines $(RO)_2NH$.[11] In the present work, chlorination of these compounds and properties of previously unknown N-chloro-N,N-dialkoxyamines thereby formed

* For Parts 43 and 69 see ref. 1.

have been investigated. These compounds were found to be highly unstable, so attempts to record their ^1H NMR spectra at -30 °C met with failure. In chlorination of N-benzyloxy-N-methoxyamine under the action of tert-BuOCl there were obtained products of fragmentation of intermediate N-chloro-N-benzyloxy-N-methoxyamine, benzyl chloride and methyl nitrite (scheme 1).

$$PhCH_2ONHOMe \xrightarrow[CCl_4]{t\text{-}BuOCl} \left[PhCH_2ON(Cl)OMe\right] \longrightarrow PhCH_2Cl + MeONO \qquad (1)$$

Nevertheless, N-chloro-N,N-dialkoxyamines react with nucleophiles in situ in solution at -78 °C. It was found that they interact with pyridine and trimethylamine as alkylating rather than dialkoxyaminating reagents (scheme 2).

$$(MeO)_2NH \xrightarrow[-78\ °C]{t\text{-}BuOCl/Et_2O} \left[(MeO)_2NCl\right] \xrightarrow{Me_3N} Me_4\overset{+}{N}\ Cl^- + Me_3N{\cdot}HCl$$
$$\Big\downarrow C_5H_5N \qquad (2)$$
$$C_5H_5\overset{+}{N}Me\ Cl^- \xrightarrow{AgClO_4} C_5H_5\overset{+}{N}Me\ ClO_4^-$$

The product of first reaction was identified as its own perchlorate. The formation of trimethylamine hydrochloride in the second case is apparently due to partial hydrolysis of intermediate N-chloro-N,N-dimethoxyamine under the action of water traces in the solvent to form HCl (scheme 3).

$$\left[(MeO)_2NCl\right] \xrightarrow{H_2O} HCl + MeOH + MeONO \qquad (3)$$

Under the action of the alkoxide, however, N-chloro-N,N-dialkoxyamines undergo the nucleophilic substitution of Cl with the formation of trialkoxyamines (scheme 4).

$$i\text{-}BuONHOR \xrightarrow[-78\ °C]{t\text{-}BuOCl/DME} \left[i\text{-}BuON(Cl)OR\right] \xrightarrow{MeONa} i\text{-}BuON(OMe)OR \qquad (4)$$

DME = dimethoxyethane — 1 R=Me, 2 R=Et

The products were characterized by ^1H NMR spectra and elemental analysis. In order to confirm their structures unequivocally, the unsymmetrically substituted trialkoxyamine-^{15}N was prepared (scheme 5). The starting N,N-dialkoxyamine-^{15}N was synthesized according to ref. 11.

$$^{15}NH_2Cl \xrightarrow{i\text{-}BuONa} {}^{15}NH_2OBu\text{-}i \xrightarrow{Me_2NCOCl} Me_2NCO{}^{15}NHOBu\text{-}i \xrightarrow{t\text{-}BuOCl}$$
$$\longrightarrow \left[Me_2NCO{}^{15}N(Cl)OBu\text{-}i\right] \xrightarrow{EtONa} Me_2NCO{}^{15}N(OEt)OBu\text{-}i \xrightarrow{KOH/H_2O} \qquad (5)$$
$$\longrightarrow EtO{}^{15}NHOBu\text{-}i \xrightarrow[2.\ MeONa]{1.\ t\text{-}BuOCl} EtO{}^{15}N(OMe)OBu\text{-}i \quad \underline{2}\text{-}{}^{15}N$$

^1H and ^{13}C NMR spectra of the N-mono-, N,N-di-, and N,N,N-trialkoxyamines obtained contain specific coupling constants of ^{15}N nucleus with protons($J\ ^{15}NOCH$) and carbon atoms ($J\ ^{15}NO^{13}C$) of all substituents (Table), which serve as a structural test for these types of compounds.

The ^1H NMR spectrum of the unsimmetrically substituted trialkoxyamine 2 (Fi-

Table. Coupling constants (J, Hz) of the ^{15}N nucleus in N-alkoxyamines

Compound	$J_{^{15}NOCH}$	$J_{^{15}NO^{13}C}$	$J_{^{15}NH}$
$^{15}NH_2OBu\text{-}i$	2.9	*)	60.3
$Me_2NCO^{15}NHOBu\text{-}i$	2.4	*)	86.7
$Me_2NCO^{15}N(OEt)OBu\text{-}i$	2.1(Et)	1.6(Et)	-
	2.2(Bu-i)	2.4(Bu-i)	
$EtO^{15}NHOBu\text{-}i$	2.4(Et)	2.7(Et)	64.5
	2.4(Bu-i)	2.6(Bu-i)	
$EtO^{15}N(OMe)OBu\text{-}i$	3.4(Me)	2.1(Me)	-
	2.8(Et)	3.7(Et)	
	2.4(Bu-i)	3.7(Bu-i)	

*) ^{13}C NMR spectra were not recorded

gure) reveals the geminal anisochronism of the diastereotopic groups and methylene protons. This is the evidence of a high pyramidal stability of the N atom in the trialkoxyamines. However, the attempts of determination of the inversion barrier for N atom in 2 by the coalescence of diastereotopic group signals failed because of decomposition of the sample during the heating.

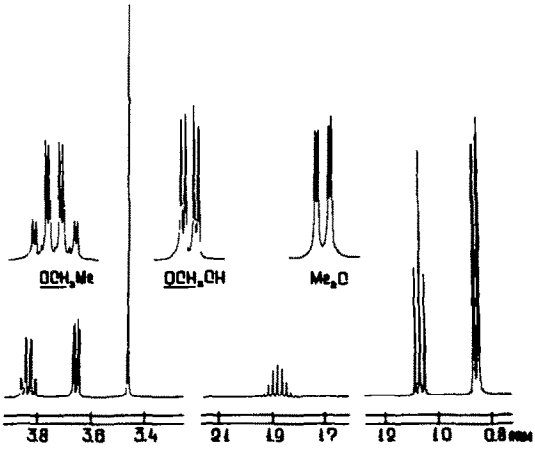
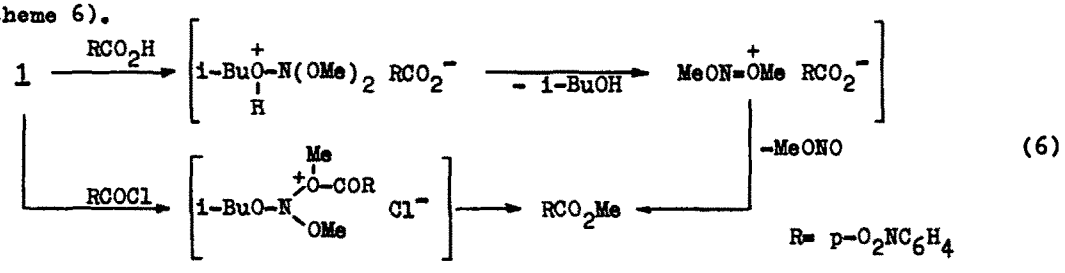


Figure. ^1H NMR spectrum (400 MHz, C_6D_6) of trialkoxyamine 2.

The trialkoxyamines are relatively unstable: they decomposes both when distilled (50 °C, 1 torr) and when chromatographed on column (Al_2O_3 - neutral by Brockmann or silica gel, elution with Et_2O). The product 1 decomposes by ca.50% on heating in CCl_4 in a sealed ampule at 100 °C for 4 hours.

Like the orthoesters of carboxylic acids, the trialkoxyamine 1 produces methyl p-nitrobenzoate on interaction with p-nitrobenzoic acid or its chloride (scheme 6).



(6)

The examples of the trialkoxyamine reactions presented above proved their alkylating properties under the acid catalysis conditions and the possibility of

extending the principles of chemistry of orthoesters[3] to these amines.

It should be noted that the properties of N-chloro-N,N-dialkoxyamines are to a great extent similar to those of well studied carbon analogues, dialkoxyalkyl chlorides.[20] It is known that these compounds are thermally unstable and their reactions with nucleophiles take place via intermediate dialkoxycarbenium ions in two directions: either by addition of nucleophiles to the carbenium centre or by dealkylation of the oxonium centre. The first route represents the kinetically controlled reaction pathway, which may be reversible, and second route yields thermodynamically stable products. It was shown that the use of strong nucleophiles (RO^-, CN^-) enables to the isolation of products being formed by the kinetically controlled route. The properties of dialkoxycarbenium ions has been accounted for on the basis of energetic considerations.[20,21]

By analogy with the dialkoxyalkyl chlorides the properties of the N-chloro-N,N-dialkoxyamines find a good explanation in easy dissociation of the N-Cl bond and in the formation of resonance-stabilized dialkoxynitrenium ions (the chemistry of other nitrenium cations has been reviewed elsewhere[22,23]). Thus, the thermal fragmentation of N-chloro-N,N-dialkoxyamines (scheme 1) is conditioned by dealkylation of these ions under the action of the inner nucleophile, the Cl^- anion, by a thermodynamically preferable irreversible route (scheme 7).

$$RON(Cl)OR' \rightleftharpoons RO\overset{+}{N}OR'\ Cl^- \longleftrightarrow RON{=}\overset{+}{O}R'\ Cl^- \longrightarrow RONO + R'Cl \qquad (7)$$

As like as dialkoxycarbenium ions,[20,21] dialkoxynitrenium ions are ambident cations (scheme 8), that causes the proceeding of the reactions of N-chloro-N,N-dialkoxyamines with nucleophiles in two directions (schemes 2, 4).

$$\begin{array}{ccc} & \overset{:Nu}{\curvearrowright} & \\ RO\overset{+}{N}OR'\ Cl^- & \xrightleftharpoons{\text{route 1}} & RON(Nu)OR' + Cl^- \\ \updownarrow & & \\ RON{=}\overset{+}{O}R'\ Cl^- & \xrightarrow{\text{route 2}} & RONO + NuR' + Cl^- \\ & \underset{:Nu}{\curvearrowleft} & \end{array} \qquad (8)$$

Thus, a new class of configurationally stable acyclic amines - trialkoxyamines has been synthesized, these are the first representatives of orthonitrites.

EXPERIMENTAL

^1H NMR spectra were recorded on JNM-C-60 HL (60 MHz) and BRUKER WM-400 (400 MHz) spectrometers. ^{15}N NMR (40.53 MHz) and ^{13}C NMR (100.61 MHz) spectra were obtained on a BRUKER WM-400 instrument. Chemical shifts are guoted in p.p.m. downfield from TMS (δ ^{15}N relative to external standard, $H^{15}NO_3$). Coupling constants values are given in Hzs.

N,N-Dimethoxyamine (61.5% yield, b.p. 83.5 °C), N-benzyloxy-N-methoxyamine (66.7% yield, purified by chromatography), N-isobuthoxy-N-methoxyamine (72.2% yield, b.p. 65 °C/40 torr), N-isobuthoxy-N-ethoxyamine (74.4% yield, b.p. 74 °C/30 torr) were prepared by known methods.[11]

Reaction of N-benzyloxy-N-methoxyamine with t-BuOCl. To a solution of 0.36 g (3.3 mmol) of t-BuOCl in 5 ml of CCl_4 at -20 °C was added a solution of 0.46 g (3.3 mmol) of N-benzyloxy-N-methoxyamine in 5 ml of CCl_4. The mixture was kept at -8 °C for 1 h and at 20 °C for 1 h. Methyl nitrite was identified by ^1H NMR (60 MHz) spectrum of the mixture after an addition of the standard MeONO in CCl_4 to the mixture. ^1H NMR: 3.98 (MeO). Removal of the solvent in vacuo and distil-

lation of the residue gave 0.33 g (78.9%) of benzyl chloride, b.p. 71-72 °C/20 torr, which was identified with the standard by ^{1}H NMR spectrum (60 MHz, CCl_4): 4.50 (CH_2), 7.28 (Ph). Found: C, 66.07; H, 5.47. Calc. for C_7H_7Cl: C, 66.42; H, 5.57%.

Reaction of N-chloro-N,N-dimethoxyamine with trimethylamine. To a solution of 0.15 g (2 mmol) of N,N-dimethoxyamine and 0.16 g (2.71 mmol) of trimethylamine in 15 ml of abs. ether at -78 °C was added a solution of 0.22 g (2 mmol) of t-BuOCl in 2 ml of abs. ether. The mixture was kept at -78 °C for 0.5 h and for 24 h at -8 °C. The precipitate was separated and extracted with CH_2Cl_2. Evaporation of the extract in vacuo and crystallization of the residue from abs. MeCN afforded 0.03 g (15.7%) of trimethylamine hydrochloride, m.p. 277 °C (dec.) which was identified by ^{1}H NMR spectrum (60 MHz, CD_3OD): 2.78 (Me). The residue insoluble in CH_2Cl_2 was crystallized from a EtOH-ether mixture to give 0.05 g (22.8%) of tetramethylammonium chloride, m.p. 230 °C (dec.). ^{1}H NMR spectrum (60 MHz, CD_3OD); 3.25 (Me). Found: C, 43.48; H, 11.12; N, 12.95. Calc. for $C_4H_{12}NCl$: C, 43.84; H, 11.04; N, 12.78%. Melting points of the products did not change when mixed with authentic samples.

Reaction of N-chloro-N,N-dimethoxyamine with pyridine. To a solution of 0.15 g (2 mmol) of N,N-dimethoxyamine and 0.32 g (4 mmol) of pyridine in 11 ml of abs. ether at -78 °C was added a solution of 0.24 g (2.2 mmol) of t-BuOCl in 2 ml of abs. ether. The mixture was kept for a night at -8 °C. The precipitate was separated, washed with abs. ether and dried in vacuo to yield 0.06 g (21%) of N-methylpyridinium chloride. ^{1}H NMR spectrum (60 MHz, CD_3OD): 4.40 (MeN), 8.08 and 8.90 (m, C_5H_5). Pyridinium perchlorate was prepared as follows. To a solution of 0.09 g (0.42 mmol) of $AgClO_4$ in 4 ml of water at 20 °C was added a solution of 0.06 g (0.42 mmol) of N-methylpyridinium chloride in 4 ml of water. The precipitate was separated, the water was vacuum removed from the filtrate, and the residue was crystallized from a MeOH-ether mixture to give 0.03 g (36.5%) of N-methylpyridinium perchlorate, m.p. 147-148 °C. ^{1}H NMR spectrum (60 MHz, CD_3OD): 4.35 (MeN), 8.03 and 8.85 (m, C_5H_5). Found: C, 37.11; H, 4.22; N, 7.37. Calc. for $C_6H_8NO_4Cl$: C, 37.23; H, 4.17; N, 7.24%.

N,N-Dimethoxy-N-isobuthoxyamine 1. To a stirred suspension of MeONa (obtained by dissolving 0.092 g of Na in 5 ml of abs. MeOH, evaporating the solution in vacuo, washing the residue with abs. ether, and drying it in vacuo) in 10 ml of abs. dimethoxyethane (DME) at -78 °C was added a solution of 0.28 g (2 mmol) of N-methoxy-N-isobuthoxyamine in 10 ml abs. DME and, immediately after, a solution of 0.43 g (4 mmol) of t-BuOCl in 10 ml of abs. DME. The mixture was kept for 1 h at -78 °C, then for 3 h at -8 °C and saturated with CO_2. The precipitate was separated, the solvent was removed from the filtrate in vacuo (10 torr), and the residue was extracted with ether. The extract was concentrated in vacuo (10 torr) and the residue was recondensed at 20 °C (ca. 10^{-3} torr) to afford 0.19 g(63.5%) of 1. ^{1}H NMR spectrum (400 MHz, toluene-d_8): 0.84 (Me_2C, ^3J=6.8), 1.84 (CH), 3.41 (MeO), 3.56 (CH_2, ^3J=6.6). Found: C, 48.11; H, 10.32; N, 9.41. Calc. for $C_6H_{15}NO_3$: C, 48.31; H, 10.13; N, 9.39%.

N-Isobuthoxy-N-ethoxy-N-methoxyamine 2. As described above, a reaction of 0.27 g (2 mmol) of N-isobuthoxy-N-ethoxyamine, 4 mmol of MeONa and 0.43 g (4 mmol) of t-BuOCl in 30 ml of abs. DME gave , after recondensation at 20 °C (ca. 10^{-3} torr), 0.16 g (50%) of 2. ^{1}H NMR spectrum (400 MHz, C_6D_6): 0.84 (Me_2C, $\Delta\nu$ =1.5 Hz, ^3J=6.8), 1.05 ($\underline{Me}CH_2$, ^3J=7.1), 1.86 (CH), 3.46 (MeO), 3.64 ($\underline{CH_2}CH$, $\Delta\nu$ =2.4 Hz, ^3J=6.8), 3.82 ($Me\underline{CH_2}$, $\Delta\nu$ =1.7 Hz). Found: C, 51.30; H, 10.52; N, 8.49. Calc. for $C_7H_{17}NO_3$: C, 51.51; H, 10.49; N, 8.58%.

Isobuthoxyamine-^{15}N was synthesized as described in ref. 24 from $^{15}NH_2Cl$ pre-

pared from $(^{15}NH_4)_2SO_4$ (enriched to 95.2%). The yield was 30% calc. on $^{15}NH_2Cl$. ^1H NMR spectrum (400 MHz, $CDCl_3$): 0.84 (Me_2C, ^3J=6.8), 1.89 (CH), 3.37 (CH_2, ^3J=6.8, J ^{15}NOCH =2.9), 4.81 (NH_2, J ^{15}NH=60.3). ^{15}N NMR spectrum (C_6D_6): 223.8 (J ^{15}NH =60.3).

1,1-Dimethyl-3-isobuthoxyurea-$^{15}N(3)$ was prepared according to ref. 10 in 80% yield, b.p. 109-110 °C/1 torr. ^1H NMR spectrum (400 MHz, C_6D_6): 0.92 (Me_2C, ^3J=6.8), 1.99 (CH), 2.44(Me_2N), 3.68 (CH_2, ^3J=6.8, J ^{15}NOCH =2.4), 6.4 (NH, J ^{15}NH=86.7).

1,1-Dimethyl-3-isobuthoxy-3-ethoxyurea-$^{15}N(3)$ was synthesized as described in ref. 10 in 75% yield, b.p. 83 °C/1 torr. ^1H NMR spectrum (400 MHz, C_6D_6): 0.87 (Me_2C, ^3J=6.8), 1.06 ($MeCH_2$, ^3J=7.1), 1.86 (CH), 2.55 (Me_2N), 3.70 (CH_2CH, ^3J=6.8, J ^{15}NOCH =2.2), 3.88 ($MeCH_2$, J ^{15}NOCH =2.1). ^{13}C NMR spectrum (C_6D_6): 13.9 ($MeCH_2$, ^1J=125.7, ^2J=2.4), 19.51 (Me_2C, ^1J=125.7), 27.98 (CH, ^1J=128.2), 36.81 (Me_2N, ^1J=137.9), 67.31 (CH_2CH, ^1J=144.0, J ^{15}NO^{13}C =2.4), 78.23 ($MeCH_2$, ^1J=142.8, J ^{15}NO^{13}C =1.6), 159.56 (CO, J ^{15}N^{13}C =2.4).

N-Isobuthoxy-N-ethoxyamine-^{15}N was obtained according to ref. 11 in 75% yield, b.p. 74 °C/30 torr. ^1H NMR spectrum (400 MHz, C_6D_6): 0.84 (Me_2C, $\Delta\nu$ =1.9, ^3J=6.8), 1.05 ($MeCH_2$, ^3J=7.1), 1.83 (CH), 3.46 and 3.68 (CH_2CH, J_{AB}=9.3, ^3J=6.8, J ^{15}NOCH =2.4), 3.68 and 3.86 ($MeCH_2$, J_{AB}=9.3, J ^{15}NOCH =2.4), 7.55 (NH, J ^{15}NH=64.5). ^{13}C NMR spectrum (neat): 14.51 ($MeCH_2$, ^1J=125.8, ^2J=2.0), 28.41 (CH, ^1J=128.5), 68.29 (CH_2CH, ^1J=143.4, ^2J=4.8, J ^{15}NO^{13}C =2.6, J_{CONH}=5.0). ^{15}N NMR (neat): 127.8 (J ^{15}NH =64.5, J ^{15}NOCH =2.4).

N-Isobuthoxy-N-ethoxy-N-methoxyamine-^{15}N 2-^{15}N was prepared similarly to the synthesis of 2. Its ^1H NMR spectrum is similar to that of 2 but, besides, there was additional signal splitting conditioned by the coupling of ^{15}N nucleus with protons of substituents (Table). ^{13}C NMR spectrum (C_6D_6): 13.99 ($MeCH_2$, ^1J=126.7, ^2J=1.9), 19.44 (Me_2C, ^1J=124.9, ^2J=3.7), 27.85 (CH, ^1J=127.6, ^2J=2.8), 53.92 (MeO, ^1J=144.3, J ^{15}NO^{13}C =2.1), 64.39 (CH_2CH, ^1J=144.3, ^2J=3.7, J ^{15}NO^{13}C =3.7), 75.12 ($MeCH_2$, ^1J=144.4, ^2J=4.5, J ^{15}NO^{13}C =3.7). ^{15}N NMR spectrum (C_6D_6): 56.1.

Reaction of 1 with p-nitrobenzoic acid. To a suspension of 0.15 g (0.89 mmol) of p-nitrobenzoic acid in 7 ml of abs. ether at -78 °C was added a solution of 0.15 g (0.99 mmol) of 1 in 1 ml of abs. ether. The mixture was stirred at this temperature for 20 min. and then was kept for 24 h at -8 °C and for 24 h at 20 °C. The solvent was removed in vacuo, and the residue was crystallized from CCl_4 to yield 0.12 g (73.7%) of methyl p-nitrobenzoate, m.p. 91-92 °C (cf. ref. 25: m.p. 91-92 °C). ^1H NMR spectrum (400 MHz, $CDCl_3$): 3.99 (MeO), 8.22, 8.30 (C_6H_4, J_{AB}=8.8). Found: C, 53.33; H, 3.99; N, 7.54. Calc. for $C_8H_7NO_4$: C, 53.04; H, 3.89; N, 7.73%.

Reaction of 1 with p-nitrobenzoyl chloride. To a solution of 0.16 g (0.85 mmol) of p-nitrobenzoyl chloride in 3 ml of CCl_4 was added at -20 °C a solution of 0.14 g (0.94 mmol) of 1 in 1 ml of CCl_4. The mixture was kept for 24 h at 20 °C, afterwards the solvent was removed in vacuo and the residue was crystallized from CCl_4 to give 0.14 g (90.6%) of methyl p-nitrobenzoate (see the preceeding experiment).

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