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ASYMMETRICAL NITROGEN.

52.* NUCLEOPHILIC SUBSTITUTION AT NITROGEN IN N-CHLORO-N-ALKOXYAMINES

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UDC 541.127:542.91:547.435

The mechanism of nucleophilic substitution at tervalent nitrogen has received limited attention. For example, it has been shown by kinetic methods that substitution in the system H_2NX when X = Cl [2], $HOSO_3$ [3], and 2,4-$(O_2N)_2C_6H_3O$ [4] occurs by the S_N2 mechanism. We have previously observed that N-chloro-N-alkoxy-N-tert-alkylamines [5] readily undergo nucleophilic substitution of chlorine [5, 6]. In this case, it was suggested that, by analogy with α-chloroalkyl esters [7], nitrenium-oxonium ions are formed as intermediates. In order to check the validity of this suggestion, we have attempted to elucidate the mechanism of nucleophilic substitution in some reactions of N-chloro-N-alkoxyamines using kinetic and stereochemical data.

It has previously been shown that the alkoxyamination of pyridine takes place cleanly and irreversibly [6]:

$$\underset{(I)}{MeO_2CCMe_2N(OMe)Cl} + NC_5H_5 \rightarrow \underset{(II)}{MeO_2CCMe_2\overset{+}{N}(OMe)\overset{+}{N}C_5H_5Cl^-}$$

We have examined by PMR (80 MHz, CD_3CN) the kinetic features of this reaction at constant concentrations of (I) (~0.11 mole/liter) and pyridine concentrations of 1.37-2.43 mole/liter at 20°C, and using an equimolar ratio of reactants (concentration ~0.16 mole/liter) at temperatures of 15, 20, 25, and 35°C. The current concentrations of (I), a − x, and (II), x, were found by integrating the signals for the MeO groups. All the points on the kinetic plots

*For communication 51, see [1].

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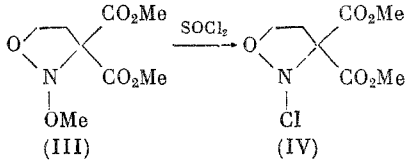
TABLE 1. Thermodynamic Data for the Reaction of (I) with Pyridine in CD_3CN

T., °C	$k_2 \cdot 10^4$, liter/mole·sec	Parameters
15	1.87±0.02	E_a=16.9 kcal/mole
20	3.57±0.03	$\Delta H_{20}^{\neq}$=16.3 kcal/mole
25	5.26±0.05	$\Delta S_{20}^{\neq}$=−18.5 cal/mole·deg
35	13.28±0.16	

were well described by a first-order equation when an excess of pyridine was used, and by a second-order equation when equimolar proportions of the reactants were used (Fig. 1). The second-order rate constants (k_2) obtained in experiments using equimolar amounts of the reactants and using an excess of pyridine (Fig. 2) were identical. The activation parameters of the reaction were obtained from the k_2–temperature plot (Table 1), and were similar to those obtained for the S_N2 amination of pyridine [4]. These findings lead to the conclusion that the alkoxyamination of pyridine under these conditions takes place via a transition state typical of the S_N2 reaction.

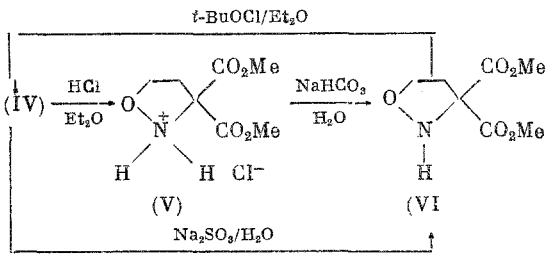
A general method for the study of reaction mechanisms is by establishing their stereochemistry using optically active reactants. Hitherto, however, this method has not been employed to examine nucleophilic substitution at nitrogen as a result of the restricted range of optically active compounds with an asymmetrical nitrogen atom, and the absence of model reactions in which the initial and final products possess high configurational stability of the nitrogen atom. We have recently studied a reaction suitable for this purpose, namely the N-alkoxyamination of alcohols by acyclic N-chloro-N-alkoxyamines to give NN-dialkoxyamines, these moreover being obtained in optically active forms [5]. However, attempts to optically activate acyclic N-chloro-N-alkoxyamines met with no success, apparently as a result of their insufficiently great configurational stability ($\Delta G^{\neq}$ 18.6-19.7 kcal/mole) [5]. The increased configurational stability of nitrogen when included in a ring [5, 8] led to the idea of using for this purpose cyclic N-chloro-N-alkoxyamines. The only instances of such compounds were the difficultly accessible perhalo-2-chloro-1,2-oxazetidines [9]. We have obtained the novel N-chloroisoxazolidine (IV) by reacting the N-methoxyisoxazolidine (III) with thionyl chloride (for preliminary communication, see [10]).



(III) (IV)

A similar replacement of an N-alkoxy group by chlorine has been observed in acyclic NN-dialkoxyamines [10, 11].

In order to find suitable reactions for nucleophilic substitution and optical activation, some chemical properties of (IV) were examined. It was found that like its acyclic analogs [11], (IV) was reduced smoothly by HCl and Na_2SO_3. This is a new method for the synthesis of NH-isoxazolidines (cf. [12]).



The nucleophilic substitution of Cl in (IV) takes place smoothly only on treatment with methanol in the presence of Et_3N to give the N-methoxyisoxazolidine (III). This is a new method for the preparation of N-alkoxyisoxazolidines (cf. [13]). When other nucleophiles are used, 1,2-elimination takes place exclusively or competitively to give 2-isoxazolines:

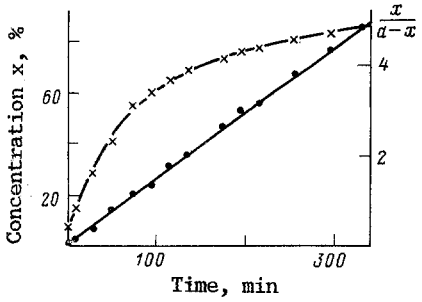


Fig. 1

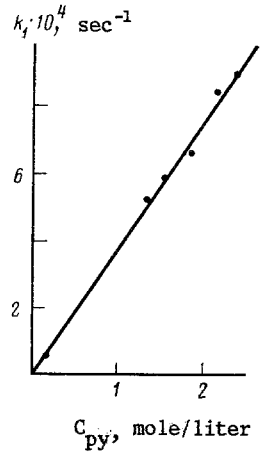
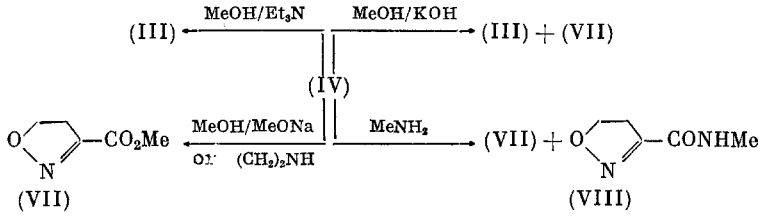


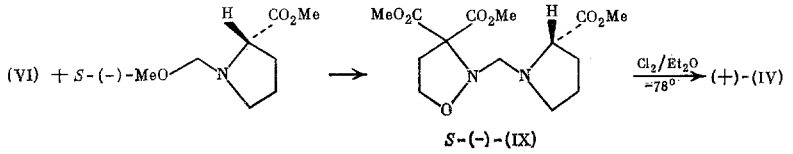
Fig. 2

Fig. 1. Kinetic plot for the reaction of (I) with an equimolar amount of pyridine (0.16 mole/liter) at 35°C, and its linear transformation.

Fig. 2. Plot of the pseudo-first-order rate constant against pyridine concentration in the reaction of (I) (0.11 mole/liter) with pyridine at 20°C.

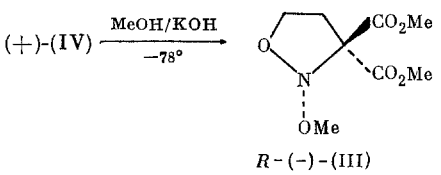


On the basis of these results, compound (VI) was used for the asymmetric synthesis of the first optically active N-chloro-N-alkoxyamine, namely N-chloroisoxazolidine (+)-(IV), by the optical activation of 1-chloro-2,2-dimethylaziridine [14]:



The kinetics of racemization of (+)-(IV) in acetonitrile at 13°C were used to obtain: $\Delta G_{inv}^{\neq}$ 20.80 ± 0.06 kcal/mole and $\tau_{1/2}$ 8 min, i.e., even in cyclic (five-membered) N-chloro-N-alkoxyamines the configurational stability of the nitrogen is relatively low, making it difficult to use them for stereochemical purposes. Attempts to determine the optical purity of (+)-(IV) by PMR (400 MHz) using the chiral shift reagent Eu(tfc)$_3$ were unsuccessful.

It was found that low-temperature methanolysis of (+)-(IV) resulted in the retention of optical activity. This is a new method for the optical activation of N-alkoxyisozazolidines (cf. [8]).



The absolute configuration and optical purity of the product (~3%) were found by comparing the sign and magnitude of the specific optical rotation with those given in [8]. The unknown optical purity and absolute configuration of the initial (+)-(IV) did not permit an unambiguous conclusion to be drawn as to the mechanism of nucleophilic substitution in

this reaction. However, the fact that optical activity is retained in the reaction product is evidence for the fact that it takes place at least to some extent stereospecifically. The mechanism of the alcoholysis of N-chloro-N-alkoxyamines requires further study. The experiments described here is important in principle, since it is the first instance of nucleophilic substitution at nitrogen in an optically active compound with an asymmetric center at the nitrogen atom only.

EXPERIMENTAL

PMR spectra were obtained on Jeol C-60-HL, Bruker WP-80-SY, and Bruker WM-400 spectrometers. Chemical shifts are given as ppm from HMDS, and coupling constants in Hz.

(I) was obtained as described in [5], in quantitative yield; (III) (cf. [8]) in 30% yield, bp 96-97°C (1.5 mm); and the S-(−)-methyl ester of N-methoxymethylproline as in [15], yield 45%, bp 48°C (1.5 mm), $[\alpha]_D$ −33.7° (C 2.3, n-pentane).

Dimethyl N-Chloroisoxazolidine-3,3-dicarboxylate (IV). A solution of 1.09 g (5 mmole) of (III) in 3 ml of $SOCl_2$ was kept for 1 h at 0°C, and the excess $SOCl_2$ removed under reduced pressure. The residue consisted of (IV), which was used without further purification for the synthesis of (V) and (VI) (method B).

Dimethyl Isoxazolidine-3,3-dicarboxylate Hydrochloride (V). A solution of 2.24 g (10 mmole) of (IV) in 50 ml of dry ether was saturated with gaseous HCl, and the mixture kept for 12 h at 0°C and one day at 20°C. The solid which separated (1.84 g, 81.6%) had mp 81-82°C. PMR spectrum (60 MHz, CD_3OD): 2.90 (CCH_2C, ^3J = 6.8), 3.80 (MeO_2C), 4.16 (CH_2O). Found, %: C 37.40, H 5.41, N 6.44. $C_7H_{12}NO_5Cl$. Calculated, %: C 37.26, H 5.36, N 6.21.

Dimethyl Isoxazolidine-3,3-dicarboxylate (VI). A). A solution of 2.76 g (12 mmole) of (V) in 5 ml of water was neutralized with a solution of 2.02 g (24 mmole) of $NaHCO_3$ in 10 ml of water, and the mixture extracted with ether. The extract was dried over $MgSO_4$, evaporated under reduced pressure, and the residue recrystallized from a mixture of hexane and CCl_4 to give 1.01 g (44.5%) of (VI), mp 53-54°C. PMR spectrum (60 MHz, CCl_4): 2.63 (CCH_2C, ^3J = 6.8), 3.76 (MeO_2C), 3.83 (CH_2O), 5.7 (NH). Found, %: C 44.63, H 5.90, N 7.40. $C_7H_{11}NO_5$. Calculated, %: C 44.45, H 5.86, N 7.40.

B). To a solution of 2.24 g (10 mmole) of (IV) in 50 ml of ether was added with stirring at 0°C a solution of 1.51 g (12 mmole) of Na_2SO_3 in 10 ml of water. After 2 h, the organic layer was separated, and the aqueous layer extracted with ether. The combined extracts were dried over $MgSO_4$, evaporated under reduced pressure, and the residue recrystallized to give 1.27 g (67.2%) of (VI).

Chlorination of (VI). To a solution of 1.89 g (10 mmole) of (VI) in 10 ml of dry ether was added at 0°C a solution of 1.19 g (11 mmole) of t-BuOCl in 10 ml of dry ether. The solvent was removed under reduced pressure to give a quantitative yield of (IV), mp 37°C (from ether–pentane at 0°C). PMR spectrum (400 MHz, C_6D_6, TMS): 2.46, 3.08 (CCH_2C, J_{AB} = −12.9, $^3J_{H_A,H}$ = 7.1 and 8.3, $^3J_{H_B,H}$ = 5.1 and 8.3), 3.32, 3.41 (MeO_2C), 3.69, 4.02 (CH_2O, J_{AB} = −10.5). ^{13}C NMR spectrum (400 MHz, C_6D_6, TMS): 31.3 ($C\underline{CH_2}C$, ^1J = 139.2), 53.3 (Me, ^1J = 148.9), 73.1 (CH_2O, ^1J = 153.8, ^2J = 3.7), 92.0 (CN, ^2J = 4.9), 163.9, 166.3 (CO, ^3J = 3.7). Found, %: C 37.92, H 4.52, N 6.26. $C_7H_{10}NO_5Cl$. Calculated, %: C 37.59, H 4.50, N 6.26.

Reaction of (IV) with Ethyleneimine. To a solution of 2.15 g (50 mmole) of ethyleneimine in 12 ml of dry ether was added dropwise with stirring at −78°C a solution of 1.12 g (5 mmole) of (IV) in 10 ml of dry ether. After 1 h, the mixture was kept for one day at −8°C. The solid was separated, the solvent removed from the filtrate under reduced pressure (1 mm) and the residue distilled to give 0.39 g (61%) of methyl isoxazoline-3-carboxylate (VII), bp 75°C (1 mm), bp 68°C (0.5 mm) [16]. It was identified by comparison of its PMR spectrum with that of an authentic sample.

Reaction of (IV) with MeONa/MeOH. To a solution of 0.32 g (6 mmole) of MeONa in 20 ml of dry methanol was added with stirring at −78°C a solution of 1.12 g (5 mmole) of (IV) in 5 ml of dry ether. After 1 h, the solvent was removed under reduced pressure, the residue extracted with ether, the extract evaporated under reduced pressure, and the residue distilled to give 0.52 g (81%) of (VII).

Reaction of (IV) with Methylamine. To a solution of 2 ml of methylamine in 10 ml of dry ether was added dropwise with stirring at −78°C a solution of 1.12 g (5 mmole) of (IV)

in 10 ml of dry ether. After 1 h, the mixture was warmed to 20°C, the solid filtered off, the solvent removed under reduced pressure, and the residue chromatographed on a column (neutral alumina, eluant chloroform). There was obtained 0.07 g (12%) of (VII) and 0.34 g (53%) of ixoazoline-3-carboxylic acid methylamide (VIII), mp 98°C (from CCl_4), mp 96-97°C [8], identified by comparison of its PMR spectrum with that of an authentic sample.

Reaction of (IV) with MeOH/Et_3N. To 1.12 g (5 mmole) of (IV) was added at −78°C a solution of 0.5 g (5 mmole) of Et_3N in 20 ml of dry methanol. The mixture was kept for 2 h at −8°C, the solvent removed under reduced pressure, and the residue extracted with ether. The extract was evaporated under reduced pressure, and the residue distilled to give 0.95 g (86.8%) of (III), bp 96-97°C (1.5 mm) (cf. [8]). Identified by comparison of its PMR spectrum with that of an authentic sample.

Reaction of (IV) with MeOH/KOH. To 1.12 g (5 mmole) of (IV) was added at −78°C a solution of 0.28 g (5 mmole) of KOH in 10 ml of dry methanol. The mixture was kept for 2 h at −8°C, the solvent removed under reduced pressure, and the residue extracted with ether. The extract was evaporated under reduced pressure, and the residue chromatographed on a column (silica, eluant chloroform) to give 0.56 g (51%) of (III) and 0.13 g (20%) of (VII), identified by comparison of their PMR spectra with those of authentic samples.

S-(−)-Methyl Ester of 3,3-Bismethoxycarbonylisoxazolidinomethylproline S-(−)-(IX). A solution of 0.57 g (3 mmole) of (VI) and 0.55 g (3 mmole) S-(−)-N-methoxymethylproline methyl ester in 10 ml of dry benzene was kept for 1 day at 20°C, and the solvent removed under reduced pressure to give 1.02 g (~100%) of S-(−)-(IX), which was used without further purification in the following syntheses. $[\alpha]_{546}$ −42.7° (C 0.79, MeOH), PMR spectrum (400 MHz, toluene-d_8): 0.78, 1.68 (CH_2CH_2), 1.86 (NCH_e), 3.01 ($NCH\alpha$, J_{AB} = −9.0, $^3J_{H\alpha,H_A}$ = 7.6, $^3J_{H_a,He}$ = 3.9), 2.45 (OCH_2CH_2, ^3J = 7.3), 3.17, 3.23, 3.29 (MeO_2C), 3.52 (CH_2O), 4.01 (CH, ^3J = 6.4 and 8.3), 4.24, 4.27 (NCH_2N, J_{AB} = −13.9).

(+)-Dimethyl N-Chloroisoxazolidine-3,3-dicarboxylate, (+)-(IV). A solution of 1.02 g (3 mmole) of S-(−)-(IX) in 80 ml of a mixture of ether and carbon tetrachloride (5:1) was saturated at −78°C with 0.28 g (4 mmole) of chlorine. The solid was separated at −10°C, and the solvent removed from the filtrate at 0 to −10°C under reduced pressure. The residue consisted of 0.67 g (~100%) of (+)-(IV), identical in its melting point and PMR spectrum with (IV). $[\alpha]_{546}$ 74° (13°, C 5.2, MeCN).

R-(−)-Dimethyl N-Methoxyisoxazolidine-3,3-dicarboxylate, R-(−)-(III). To 0.67 g (3 mmole) of (+)-(IV) was added with stirring at −78°C a solution of 0.17 g (3 mmole) of KOH in 70 ml of dry methanol. The mixture was kept for 3 h at −78°C and 12 h at −8°C, then saturated with CO_2, the solvent removed under reduced pressure, and the residue extracted with ether. The extract was evaporated under reduced pressure, and the residue chromatographed on a column (silica, eluant chloroform) to give 0.33 g (51.3%) of R-(−)-(III), $[\alpha]_{546}$ −18.6° (C 0.35, MeOH). The PMR spectrum was identical with that of (III).

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CONCLUSIONS

1. The reaction of methyl α-(N-chloro-N-methoxyamino)isobutyrate with pyridine in CD_3CN is bimolecular.

2. The dimethyl ester of an NH-isoxazolidine-3,3-dicarboxylic acid and an optically active N-chloro-N-alkoxyamine [(+)-dimethyl N-chloroisoxazolidine-3,3-dicarboxylate] have been obtained for the first time. It has been shown that low-temperature methanolysis of the latter takes place with retention of optical activity.

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EPR AND ^1H AND ^{13}C NMR STUDY OF NITROXYL RADICAL CONVERSIONS IN STRONG AND SUPERSTRONG ACIDS

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When strong acids react with nitroxyl radicals (I) the latter are protonated at the unshared electron pair of the oxygen atom to form a strong oxidant, viz., the radical cation (II) [1, 2]; this can react with nitroxyl radicals to form protonated hydroxylamino derivatives (III) and oxoammonium salts (IV) [3-6]. The most resistant to the action of acids are the nitroxyl radicals of the 3-imidazoline series [7-10]. In the present work we have studied the behavior of nitrosyl radicals in strong and superstrong acids by EPR, PMR, and ^{13}C NMR, in order to determine the limits of their stability and the structural factors that affect it.

EXPERIMENTAL

EPR spectra were recorded on a Bruker ER-200D EPR spectrometer; precision in determination of hyperfine coupling constant a_N ±0.02 Oe; g-factor ± 0.0001. PMR spectra were recorded on a Bruker WP-200SY spectrometer (200.13 MHz frequency) in a 5-mm ampul for 3-5% solutions in the respective acid at 300°K. Internal standard was tetramethylammonium borofluoride (3.20 ppm from TMS). ^{13}C NMR spectra were recorded on a Bruker HX-90 spectrometer (22.63 MHz frequency) and a Bruker WP-200SY (50.13 MHz frequency) in a 10-mm ampul for 10-20% solutions in the respective acid, using an external standard (capillary, 3 mm outside diameter, containing 1:1 CD_3COCD_3 and TMS by volume).

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