1-(N-ETHOXYCARBONYL-N-ISOPROPYLOXY)AMINO-4-DIMETHYLAMINOPYRIDINIUM CHLORIDE. SYNTHESIS AND STRUCTURE

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Stable 1-(N-alkoxy-N-alkoxycarbonyl)amino-4-dimethylaminopyridium salt, 1-(N-ethoxycarbonyl-N-isopropyloxy)amino-4-dimethyl-aminopyridinium chloride, has been synthesized for the first time. Its structure has been studied by XRD method.

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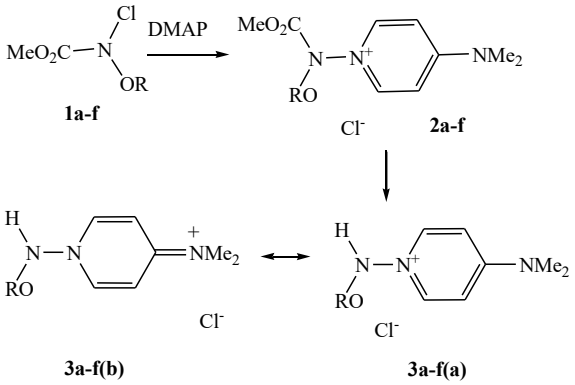
INTRODUCTION

It has been found earlier[1,2] that an interaction of methyl N-alkoxy-N-chlorocarbamates (**1a-f**) with 4-dimethyl-aminopyridine (DMAP) is a route to unknown 1-N-alkoxyamino-4-dimethylaminopyridium chlorides (**3a-f**) which was presumably realized via initial formation of unstable 1-(N-alkoxy-N-methoxycarbonyl)amino-4-dimeth-ylaminopyridium chlorides (**2a-f**) as the reaction intermediates (Scheme 1). Evidently the decomposition of unstable compounds **2a-f** yields 1-N-alkoxyamino-4-dimethylaminopyridium chlorides (**3a-f**). Compounds **3a, b** exist as structure (**3a**,**b (b)**) and "quinonoid" deformation of the pyridine ring take place.[1]

In this article synthesis of stable 1-(N-alkoxy-N-alkoxycarbonyl)amino-4-dimethylaminopyridium chloride and XRD study of its structure have been described.

EXPERIMENTAL

^{1}H and ^{13}C NMR spectra were recorded on VARIAN JEMINI 400 spectrometer (400 and 100 MHz, respectively) in DMSO-d_6 and $CDCl_3$ as solvents with TMS as internal standard. Mass spectrum was recorded on VG 70-70EQ mass spectrometer in fast atom bombardment (FAB) mode. XRD structural study was performed on Xcalibur 3 automatic four-circle diffractometer (MoKα-radiation, graphite monochromator, Sapphire-3 CCD-detector, ω-scanning). DMAP was sublimated under vacuum (3 mm Hg). The solvents were purified and dried according to standard procedures.



R = Me(a), Et(b), i-Pr(c),n-Bu(d), Oct(e), Bn(f)

Scheme 1. Reported synthesis of 1-N-alkoxyamino-4-dimethylaminopyridium chlorides.

Synthesis of 1-(N-ethoxycarbonyl-N-isopropyloxy)amino-4-dimethylaminopyridinium chloride (5)

A solution of DMAP (166 mg, 1.357 mmol) in MeCN (13 mL) was added to a solution of ethyl N-chloro-N-isopropyloxycarbamate (**4**)[3] (245 mg, 1.347 mmol) in MeCN (6 mL) at 8 ºC. The reaction solution was maintained at 8º C for 46 h, then it was evaporated under vaccum (20 mm Hg), dried under vacuum (4 mm Hg), washed with benzene (12 mL), dried under vacuum (4 mm Hg), giving **5** as colorless hygroscopic crystals (399 mg, 97 %). m.p. 103-104 °C (with decomp.), after crystallization (CH_2Cl_2-EtOAc) m.p. 104-105 °C (with decomp.). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.18 (6H, d, ^3J = 6.0, NOCH<u>Me</u>$_2$), 1.26 (3H, t, ^3J = 7.2, CO_2CH_2<u>Me</u>), 3.36 (6H, s, NMe_2), 4.23 (H, sept, ^3J = 6.0, NO<u>CH</u>Me$_2$), 4.28 (2H, q, ^3J = 7.2, CO_2<u>CH_2</u>Me), 7.39 (2H, d, ^3J = 8.0, H Py), 8.17 (2H, d, ^3J =

8.0, H Py). ^{1}H (400 MHz, $(CD_3)_2SO$) δ = 1.23 (6H, d, ^3J = 6.4, NOCH<u>Me</u>$_2$), 1.26 (3H, t, ^3J = 7.0, CO_2CH_2<u>Me</u>), 3.29 (6H, s, NMe_2); 4.29 (H, sept, ^3J = 6.4, NO<u>C</u>HMe$_2$), 4.30 (2H, q, ^3J = 7.0, CO_2<u>C</u>H_2Me), 7.11 (2H, d, ^3J = 8.0, H Py), 8.68 (2H, d, ^3J = 8.0, H Py). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.9 (CO_2CH_2<u>Me</u>), 20.5 (NOCH<u>Me</u>$_2$), 41.2(NMe_2), 65.7(NO<u>C</u>HMe$_2$), 79.2 (CO_2<u>C</u>H_2Me), 155.6, 156.8 (C-3, C-5, C-2, C-6 Py), 166.7 (C-4 Py), 185.5 (C=O). Mass spectrum, *m/z*, (I_{rel} %): 268 M^+ (100). Anal. Calcd. for $C_{13}H_{22}ClN_3O_3$: N 13.83; Found: N 13.65.

XRD structural study of compound (5)

Crystals of **5** suitable for X-ray structural analysis were grown from a solution in CH_2Cl_2-EtOAc mixture at 6 ºC. Triclinic, $C_{13}H_{22}N_3O_3^+Cl^-•H_2O$, at 100 K, *a*= 9.2359(8) Å, *b* = 13.2566(8) Å, *c* = 13.8685(9)Å, *α* = 75.552(5)°, *β* = 89.534(7)°, *γ* = 89.291(6)°,*V* = 1644.2(2) Å^3, M_r = 321.80, *Z* = 4, space group $P\bar{1}$, d_{calc}= 1.30 g cm^{-3}, μ(MoKα) = 0.25 mm^{-1}, *F*(000) = 688. Cell parameters and intensities of 11451 reflections (5737 independent reflections, R_{int}= 0.068) were measured using «Xcalibur 3» diffractometer (graphite-monochromated MoKα radiatione, CCD detector, *ω*-scan, 2 θ_{max}= 50°).

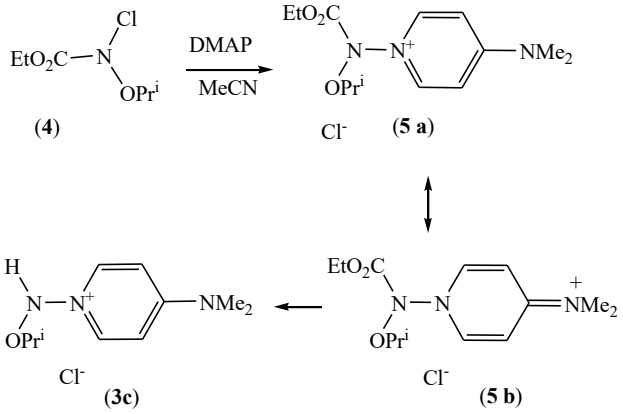
The structure was solved by direct method using SHELX-2016 program package.[4] Positions of hydrogen atoms were located geometrically and refined using the riding model with U_{iso}=nU_{eqv} of the carrier atom (*n*=1.5 for methyl moieties and *n*=1.2 for other hydrogen atoms). Full-matrix least-squares refinement against F^2 in anisotropic approximation for non-hydrogen atoms to wR_2 = 0.2844 for 5737 reflections (R_1 = 0.108 for 4016 reflections with F>4σ (F), S = 1.389). The final atomic coordinates, molecular geometry parameters, and crystallographic data of compound **5** were deposited in the Cambridge Crystallographic Data Center, 12 Union Road, CB2, 1EZ, UK (fax:+44-1223-336033, e-mail: deposit@ccdc.cam.ac.uk) and is available on request quoting the deposition number CCDC 1565981).

RESULTS AND DISCUSSION

We have found that ethyl N-chloro-N-isopropyloxycarbamate (**4**)[3] reacted with DMAP in acetonitril yielding relatively stable 1-(N-ethoxycarbonyl-N-isopropyloxy)amino-4-dimethylaminopyridinium chloride (**5**) (Scheme 2). The reaction must be carried out in mild condition at 8 ºC, because under certain conditions compound (**5**) may be spontaneously converted to 1-N-isopropyloxyamino-4-dimethylaminopyridium chloride (**3c**), for example if the reaction time is increased to 93 h.

Compound (**5**) is the first example of stable 1-(N-alkoxy-N-alkoxycarbonyl)amino-4-dimethylaminopyridium chlorides, a novel kind of N-alkoxyhydrazines.[1,2,5-10] Earlier it was regarded that 1-(N-alkoxy-N-alkoxycarbonyl)amino-4-dimethylaminopyridium chlorides were very labile and cannot exist, for example compounds **2a**-**2f**. The structure of 1-(N-ethoxycarbonyl-N-isopropyloxy)amino-4-dimethyl-aminopyridinium chloride (**5**) has been confirmed by data of ^{1}H and ^{13}C NMR spectra and mass spectrum. Also XRD study of N-alkoxyhydrazine (**5**) has been done (Figure 1, Tables 1 and 2).

It was found that in independent part of the unit cell of crystal of compound **5** there were two organic cations of forms **5A** and **5B**, two Cl^- anions and two molecules of water. Cations **5A** and **5B** differ by some geometric parameters.



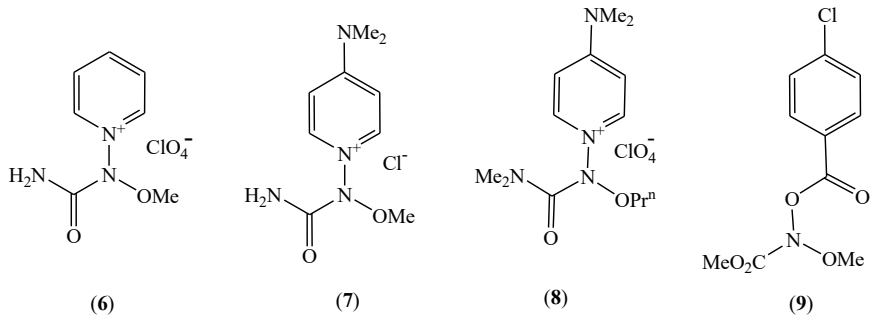
Scheme 2. Formation of (**5**).

Evidently, the positive charge is localized mainly on N(3) atom of Me_2N-group conjugate to the pyridine ring, which is confirmed by shortening of the N(3)–C(3) bond as in cation **5A** and **5B** (Table 1) in comparison with average value of 1.371 Å for N–C_{arom} bond.[11] The lengths of the N(3)–C(3) bonds are close to mean value of N=C bond (1.316 Å).[11] Thus 1-(N-ethoxycarbonyl-N-isopropyloxy)amino-4-di-3methylaminopyridinium chloride (**5**) structure is more closed to "quinonoid" structure **5b** (Scheme 2).

The observed altering of pyridine bonds conforms to this assumption: the C(2)–C(3) and C(3)–C(4) bonds are elongated in cation **5A** and **5B**, the N(2)–C(1) and N(2)–C(5) bond are elongated in both the cations **5A** and **5B** (Table 1), whereas the C(1)–C(2) and C(4)–C(5) bonds are shortened in both the cations in comparison with average bond lengths of pyridine[11] (Table 1).

In Table 1 the parameters of "quinonoid" deformation of pyridine ring are given in comparison[7] with those of 1-(N-methoxyamino)pyridinium perchlorate (**6**) with unsubstituted on 4-site as reference etalon and 1-(N-alkoxyamino)-4-dimethylaminopyridium salts (**3a**),[1] (**7**)[8] and (**8**)[8] (Scheme 3). The corresponding bond lengths of compounds **2a**, **3a**, **5A**, **5B**, **7** and **8** are similar in contrast to those in 4-unsubstituted **6**. Evidently, the nature of the substituent (H, CO_2Et, $C(O)NH_2$, $C(O)NMe_2$) at nitrogen atom, which are bound with alkoxy group, causes slight influence on "quinonoid" deformation of pyridine ring.

In **5** atom N(2) has planar configuration. The sum of bond angles centered at this nitrogen atom (∑β) is 359.9º as in **5A** as in **5B**. But amide nitrogen atom N(1), the central atom of geminal systems O-N-N^+, has pyramidal configuration. Its ∑β is equal 335.6º in (**5A**) and 335.4º in **5B**. Among anomeric amides[12,13] the existing in two or more forms differing by the pyramidality degree of the amide nitrogen atom is known for N-ethoxy-N-chlorourea[14] and N-chloro-N-methoxy-N'-(4-nitrophenyl)urea.[15] Also, N-[(benzo-yl)(hydroxyl)methyl]-N-benzyloxy-N'-(2-bromo-phenyl)-urea exists in two forms which vary in a different degree of pyramidality of the same nitrogen atom.[16]



Scheme 3. Structure of **6**, **7**, **8** and **9**.

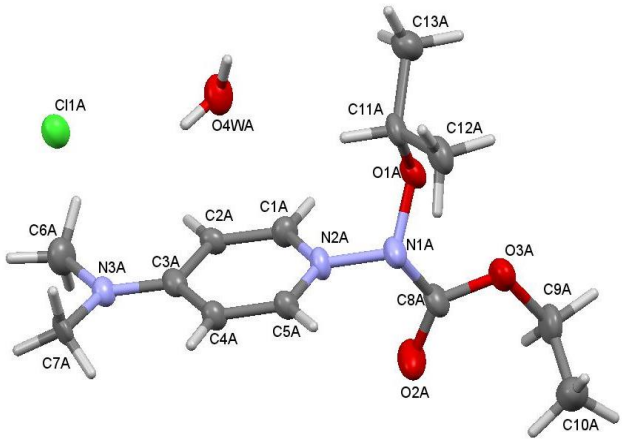


Figure 1. Molecular structure of **5A** monohydrate.

Table 1. Pyridine ring deformation in 1-(N-alkoxyamino) pyridinium salts (**3a**), (**5A**), (**5B**), (**6**), (**7**) and (**8**).

Compound	Bond lengths, Å			
	N2-C1, N2-C5	C1-C2, C4-C5	C2-C3, C3-C4	C3-NMe2
6[7]	1.341(2)	1.385(3)	1.349(5)	-
	1.341(2)	1.385(3)	1.387(4)	
5A	1.348(7)	1.360(7)	1.432(7)	1.337(7)
	1.368(7)	1.362(7)	1.411(8)	
5B	1.356(7)	1.352(7)	1.429(7)	1.326(7)
	1.349(8)	1.346(8)	1.427(8)	
3a	1.349(2)	1.351(2)	1.414(2)	1.337(2)
	1.354(2)	1.355(2)	1.416(2)	
7	1.361(2)	1.353(3)	1.425(2)	1.324(2)
	1.345(2)	1.341(3)	1.426(2)	
8	1.366(3)	1.349(3)	1.430(3)	1.333(3)
	1.346(3)	1.334(3)	1.413(3)	
Py	1.337	1.380	1.379	

Numbering of pyridine ring shown Figure 1 has been used

In compound **5** the ∑β is more in comparison to those in known 1-(N-alkoxyamino)pyridium salts (**3a**, **b**),[1] (**6**),[7] (**7**)[8] and (**8**)[8] (Table 1).

Probably it is caused by higher electron withdrawing capacity of EtO_2C-substituent compare to hydrogen (**3a**, **3b**), carbamoyl (**6**, **7**) and dimethylcarbamoyl (**8**) substituents. In anomeric amides the presence of electron withdrawing substituent at pyramidal amide nitrogen atom diminishes it pyramidality degree.[12,13]

The lone pair (Lp) of the N(1) atom is almost coplanar to the pyridine plane (the LpN(1)–N(1)-N(2)-C(1) torsion angle (TaLPPy) is -10.4º in cation **5A** and 14.6º in cation **5B**. The conjugation between LpN(1) and π-system of pyridine is impossible. The same type of LpN(1) orientation was revealed for other 1-(N-alkoxyamino)pyridium salts **3a**, 3**b**,[1] **6**,[7] **7**[8] and **8**[8] (Table 2).

In compound **5** the $N-N^+$ bond is elongated compare to the N–N bond in hydrazides of carboxylic acids (1.400 Å[17]). Probably, this $N-N^+$ bond elongation has been caused by $n_{O(Me)} \rightarrow \sigma^*_{N\text{-}N+}$ anomeric effect action as in cases of 1-(N-alkoxyamino)pyridium salts **3a**, **b**,[1] **6**,[7] 7,[8] and **8**.[8]

The lengths of the amide N–C bond in compound (**5**) is close to that in methyl N-(4-chlorobenzoyloxy)-N-methoxycarbamate **9** (1.423(2) Å[18]). The degrees of the nitrogen pyramidality in compounds **5** and **9** (∑β 334.1) are also close.[18] There is some elongation of the amide N–C bond in compound **5** compare to that in amides[19] (1.359 Å). It is caused by different degrees of C=O conjugation with sp^3 hybridized nitrogen atom in compounds **5**, **9** and sp^2 hybridized nitrogen atom in usual amides.[12,13,19]

In compound **5** ester moiety adopts +*sc*- and -*sc*-conformations relatively to the N(1)-N(2) bond, respectively. The C(11)–O(1)–N(1)–N(2) torsion angle is 77.8(5)º in **5A** and -81.4(5)º in **5B**. Ethoxy moiety has -*sc*-conformation toward to the C(8)–O(3) bond, the C(8)–O(3)–C(9)–C(10) torsion angle is -77.1(7)º in (**5A**) and -84.8(8)º in **5B**.

The isopropyloxy substituent is situated in *sp*-conformation toward to the N(1)–N(2) bond. The N(2)-N(1)-C(8)-O(2) torsion angle is-23.3(8)º in **5A** and 20.3(10)º in **5B**. This orientation of substituent is stabilized by attractive intramolecular shortening contact N(2)…H(11) 2.65 Å in **5A** and 2.63 Å in **5B**, the van der Waals radii sum is 2.66 Å.[20]

Table 2. Some structure parameters in 1-(N-alkoxyamino)pyridium salts

Compound	∑β, °	Bond lengths, Å			
		N−N+	N−OR	N−C(O)	TaLPP, °
5A	335.6	1.418(6)	1.411(6)	1.428(7)	-10.4
5B	335.4	1.421(6)	1.414(6)	1.419(8)	14.6
3a (ref.1)	312	1.428(2)	1.431(2)	-	4
3b (ref.1)	312	1.426(1)	1.440(1)	-	17
6 (ref.7)	333.9(3)	1.4254(18)	1.3999(17)	1.4515(19)	0.2
7 (ref.8)	332.7	1.413(2)	1.411(2)	1.450(2)	6
8 (ref.8)	324.22	1.425(3)	1.429(3)	1.465(3)	10.6

In crystal of **5** due to system of intramolecular hydrogen bonds with participating of chloride anions and bridging molecules of water three-dimensional network takes place:

O(4WA)−H(4WA)...Cl(1A)' (x,y,z) H…Cl' 2.50 Å, O−H….Cl' 142˚;

O(4WA)−H(4WB)...Cl(1A)' (2-x,-y,1-z) H…Cl' 2.46 Å, O−H….Cl' 157˚;

O(4WB)−H(4WC)...Cl(1B)' (x,y,z) H…Cl' 2.37 Å, O−H….Cl' 165˚;

O(4WB)−H(4WD)...Cl(1B)' (1-x,1-y,1-z) H…Cl' 2.58 Å, O−H….Cl' 143˚;

C(1A)−H(1A)...Cl(1B)' (1-x,1-y,1-z) H…Cl 2.90 Å, C−H…Cl 125˚;

C(1B)−H(1B)...Cl(1A)' (1-x,1-y,1-z)H…Cl 2.58 Å, C−H…Cl 143˚;

C(2A)−H(2A)...Cl(1B)' (1-x,1-y,1-z) H…Cl 2.87 Å, C−H…Cl 127˚;

C(4B)−H(4B)...Cl(1A)' (2-x,1-y,1-z) H…Cl 2.76 Å, C-H…Cl 134˚;

C(5A)−H(5A)...Cl(1B)' (2-x,1-y,1-z) H…Cl 2.68 Å, C−H…Cl 138˚;

C(7A)−H(7AC)...Cl(1A)' (x,y,z) H…Cl 2.81 Å, C−H…Cl 143˚;

C(7A)−H(7AC)...Cl(1A)' (x,y,z) H…Cl 2.81 Å, C−H…Cl 143˚;

C(7A)−H(7AB)...O(2A)' (2-x,1-y,1-z) H…O 2.40 Å, C−H…O 176˚;

C(7B)−H(7BC)...C(5A)' (π) (2-x,1-y,1-z) H…C 2.73 Å, C−H…C 148˚

Conclusion

The first stable 1-(N-alkoxy-N-alkoxycarbonyl)amino-4-dimethylaminopyridium salt, 1-(N-ethoxycarbonyl-N-isopropyloxy)amino-4-dimethylaminopyridinium chloride, has been synthesized. XRD study of it structure has been done.

Acknowledgements

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