Table 3. *Short intermolecular contacts* (Å,°)

$\mathrm{O—H}\cdots A$	O—H	$\mathrm{H}\cdots A$	$\mathrm{O}\cdots A$	$\mathrm{O—H}\cdots A$
$\mathrm{O}(1A^{i})\text{—}\mathrm{H}(1A)\cdots\mathrm{N}(2B^{ii})$	0·87 (3)	2·02 (3)	2·869 (3)	164 (2)
$\mathrm{O}(1B^{i})\text{—}\mathrm{H}(1B)\cdots\mathrm{N}(2A^{iii})$	0·86 (3)	2·00 (3)	2·846 (3)	166 (3)
$\mathrm{O}(1^{i})\cdots\mathrm{N}(1A^{iv})$			2·756 (3)	
$\mathrm{O}(2^{i})\cdots\mathrm{N}(1B^{iv})$			2·775 (4)	
$\mathrm{O}(1^{i})\cdots\mathrm{O}(2^{i})$			2·839 (5)	
$\mathrm{O}(2^{i})\cdots\mathrm{O}(3^{i})$			2·832 (7)	

Symmetry code: (i) x, y, z; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}+1-z$; (iii) $-1-x, \frac{1}{2}+y, \frac{1}{2}+1-z$; (iv) $\frac{1}{2}-x, 1-y, -\frac{1}{2}+z$.

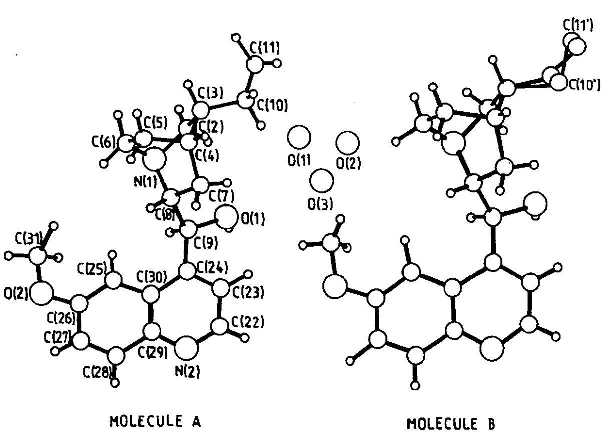

Fig. 1. A view of the molecules with the atomic labelling.

Related literature. For structures of quinidine and its derivatives, see Carter, McPhail & Sim (1967); Doherty, Benson, Maienthal & Stewart (1978); Karle & Karle (1981); Kashino & Haisa (1983); Suszko-Purzycka, Lipińska, Karczmarzyk & Pniewska (1987, 1990).

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Diethylammonium Tetrafluoro-4-pyridinolate

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Abstract. $\mathrm{C_4H_{12}N^+.C_5F_4NO^-}$, $M_r = 240·2$, triclinic, $P\bar{1}$, $a = 9·268$ (6), $b = 9·637$ (7), $c = 7·209$ (4) Å, $\alpha = 107·06$ (5), $\beta = 95·22$ (5), $\gamma = 63·70$ (4)°, $V = 551·3$ (7) Å^3, $Z = 2$, $D_x = 1·45$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·13$ mm^{-1}, $F(000) = 248$, $T = 293$ K, $R = 0·049$ for 1455 unique reflexions [$F \geq 3\sigma(F)$]. The title compound, $\mathrm{H_2NEt_2^+.NC_5F_4O^-}$, forms as an unexpected by-product during the synthesis of *N*,*N*-diethyl-*O*-(tetrafluoro-4-pyridyl)-hydroxylamine. Strong hydrogen bonds link two anion/cation pairs into a cyclic centrosymmetric dimer [$\mathrm{N}\cdots\mathrm{O}$ 2·741 (8), 2·765 (7); $\mathrm{H}\cdots\mathrm{O}$ 1·60 (6), 2·08 (6) Å; $\mathrm{N—H}\cdots\mathrm{O}$ 172 (5), 161 (7)°], which may in part explain the salt's stability and hence ease of formation.

Experimental. Reaction of sodium diethylnitroxide in benzene with pentafluoropyridine yielded several products, including a white solid consisting predominantly of diethylammonium tetrafluoro-4-pyridinolate and sodium fluoride. Removal of the latter by boiling in ethyl acetate followed by repeated recrys-

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Table 1. *Positional parameters and B_{eq}*

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$ (Å^2)
F(1)	0·8298 (5)	0·4341 (5)	0·8943 (6)	6·2 (3)
F(2)	0·5665 (5)	0·6512 (5)	0·7758 (7)	7·0 (4)
F(3)	0·3209 (5)	0·3229 (5)	0·6395 (7)	7·4 (4)
F(4)	0·5738 (4)	0·0871 (5)	0·7501 (6)	6·1 (3)
O(1)	0·8376 (5)	0·1402 (5)	0·8839 (7)	5·0 (3)
N(1)	0·4438 (7)	0·4858 (8)	0·7055 (9)	5·2 (5)
N(2)	0·8886 (7)	−0·1483 (8)	0·9361 (1)	4·4 (4)
C(1)	0·7167 (8)	0·2510 (9)	0·8279 (9)	3·5 (5)
C(2)	0·7045 (8)	0·3968 (9)	0·8285 (10)	3·9 (5)
C(3)	0·5704 (9)	0·5090 (1)	0·7675 (10)	5·0 (6)
C(4)	0·4516 (8)	0·3493 (1)	0·7025 (10)	4·7 (6)
C(5)	0·5781 (8)	0·2261 (9)	0·7574 (10)	4·2 (5)
C(6)	0·9170 (10)	−0·2797 (11)	0·7499 (14)	5·6 (6)
C(7)	1·0243 (12)	−0·2814 (13)	0·6101 (16)	6·9 (8)
C(8)	0·7763 (9)	−0·1368 (11)	1·0812 (14)	5·6 (6)
C(9)	0·7526 (11)	−0·0059 (12)	1·2611 (15)	6·5 (7)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

F(1)—C(2)	1·368 (7)	N(2)—C(8)	1·49 (1)
F(2)—C(3)	1·339 (8)	C(1)—C(2)	1·358 (8)
F(3)—C(4)	1·358 (7)	C(1)—C(5)	1·431 (8)
F(4)—C(5)	1·343 (7)	C(2)—C(3)	1·377 (9)
O(1)—C(1)	1·292 (7)	C(4)—C(5)	1·377 (9)
N(1)—C(3)	1·305 (8)	C(6)—C(7)	1·47 (1)
N(1)—C(4)	1·280 (8)	C(8)—C(9)	1·47 (1)
N(2)—C(6)	1·50 (1)		
C(3)—N(1)—C(4)	115·6 (7)	N(1)—C(3)—C(2)	123·4 (7)
C(6)—N(2)—C(8)	114·9 (6)	O(1)—C(1)—C(2)	126·2 (6)
O(1)—C(1)—C(5)	120·3 (7)	N(1)—C(4)—C(5)	127·0 (7)
C(2)—C(1)—C(5)	113·6 (7)	C(1)—C(5)—C(4)	117·8 (7)
C(1)—C(2)—C(3)	122·5 (7)	N(2)—C(6)—C(7)	112·4 (7)
N(2)—C(8)—C(9)	112·7 (7)		

tallization from the same solvent produced crystals suitable for structure determination.

Crystal dimensions 0·3 × 0·2 × 0·2 mm, Rigaku AFC6S diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Unit-cell dimensions from setting angles of 20 accurately centered reflexions ($12{\cdot}7 \leq 2\theta \leq 22{\cdot}6°$), ω–2θ scan mode, ω-scan width ($0{\cdot}84 + 0{\cdot}30\tan\theta$)° and scan speed of 8° min^{-1} with rescan (max. 2) and accumulation of counts of weak reflexions [$I < 10\sigma(I)$]. $0 \leq h \leq 9$, $-10 \leq k \leq 10$, $-7 \leq l \leq 7$, $0 \leq \theta \leq 24°$, 1920 reflexions measured, 1455 unique ($R_{\rm int} = 0{\cdot}095$), 691 observed [$F \geq 3\sigma(F)$]. Intensity standards ($\bar{2}0\bar{2}$; $\bar{3}10$; $\bar{2}\bar{2}\bar{1}$) measured every 150 reflexions, 9% decomposition, Lp and decomposition corrections applied, absorption ignored. *MITHRIL* (Gilmore, 1984) used to solve the phase problem, all non-H-atom positions found in Fourier map, H atoms from ΔF synthesis. Full-matrix least-squares refinement based on F using *TEXSAN* crystallographic software (Molecular Structure Corporation, 1985), anisotropic thermal parameters for heavier atoms, fixed isotropic (0·0633 Å^2) for H atoms, final $R = 0{\cdot}050$, $wR = 0{\cdot}056$, $S = 1{\cdot}90$, $w = 1/[\sigma^2(F_o) + (0{\cdot}03F_o)^2]$. Maximum fluctuation in final ΔF map in range $-0{\cdot}19$ to $0{\cdot}19$ e Å^{-3}, maximum Δ/σ 0·04. Scattering factors from Cromer & Waber (1974), computation carried out on a Digital VAX station 3520. Literature survey performed *via* the Cambridge Structural Database using the *Crystal Structure Search and Retrieval* interactive system (CSSR, 1984). Fractional atomic coordinates and vibrational parameters for non-H atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The hydrogen-bonded cluster, including atomic labelling, is displayed in Fig. 1.

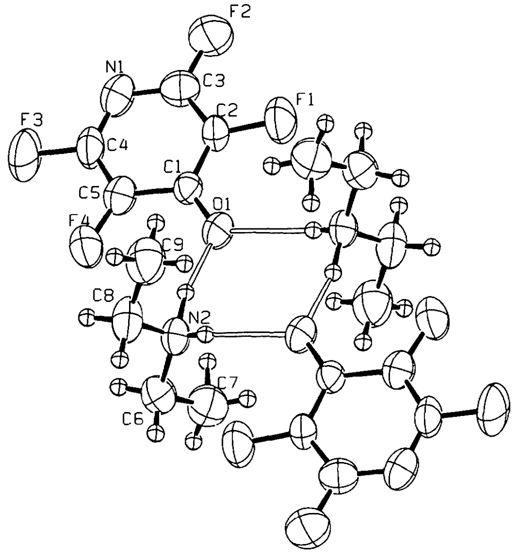

Fig. 1. The title molecular ions and associated hydrogen-bonding scheme using *ORTEP*II (Johnson, 1976).

Related literature. Further details of the sample preparation technique have been published by Banks, Jondi & Tipping (1989). The diethylammonium dimensions show good agreement with those of an adduct of pentane-2,4-dione and diethylamine reported by Emsley, Freeman, Parker, Dawes & Hursthouse (1986).

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* Lists of structure factors, intramolecular and intermolecular distances and angles, torsion angles, H-atom coordinates and anisotropic vibrational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53706 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2,3-Dichloro-4-formylphenoxy)acetic Acid

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Abstract. $C_9H_6Cl_2O_4$, $M_r = 249{\cdot}05$, orthorhombic, *Pnam*, $a = 11{\cdot}8480\,(5)$, $b = 12{\cdot}7840\,(6)$, $c = 6{\cdot}4678\,(6)$ Å, $V = 979{\cdot}7\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}69$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 5{\cdot}9$ cm^{-1}, $F(000) = 504$, $T = 296$ K, final $R(F) = 0{\cdot}047$ for 700 unique observed reflections. The molecule is planar and lies on the mirror plane of space group *Pnam*. The terminal oxyacetic acid moiety is oriented in a synperiplanar–synperiplanar manner. The molecules form chains along the *a* axis *via* hydrogen bonds with distance O(3)···O(1) being 2·668 (5) Å and an O(3)—H···O(O1) angle of 168·5°.

Experimental. The title compound was crystallized from ethanol at room temperature. Data were collected on a Rigaku AFC-5 diffractometer equipped with a rotating-anode X-ray source. Data collection at 32° min^{-1}, 2θ–ω scans. Crystal dimensions 0·2 × 0·3 × 0·3 mm. Cell parameters from 20 reflections in the 2θ range 68–78°. Range of indices: h 0 to 13, k 0 to 14, l 0 to 7 ($\theta < 60°$). Three standard reflections were measured after every 150 reflections and showed no decrease in intensity ($1\bar{4}1$, $1\bar{4}0$, $2\bar{4}0$; with a maximum reduction in intensity of 0·4% at the end of data collection). Empirical absorption correction was applied based on ψ scans of three reflections ($0\bar{1}1$, $0\bar{2}2$, $1\bar{2}2$: with minimum and maximum transmission coefficients of 0·7 and 1·0 respectively). Of the 893 reflections measured, 700 had $I > 2{\cdot}0\sigma(I)$. Direct methods (*MITHRIL*; Gilmore, 1983) were used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. Function minimized was $\sum[w(k|F_o| - |F_c|)^2]$, where $w = 1/\sigma(|F_o|)^2$ (*UPALS*; Lundgren, 1978). $R(F) = 0{\cdot}043$, $wR(F) = 0{\cdot}065$, $S = 1{\cdot}78$, for 700 reflections and 159 refined parameters in space group $Pna2_1$. Refinement also carried out in the non-isomorphic supergroup *Pnam*, with all atoms on the mirror plane along **c**. The z coordinate of all atoms was fixed and the B_{13} and B_{23} components of their anisotropic temperature factor tensors were set at zero. $R(F) = 0{\cdot}047$, $wR(F) = 0{\cdot}070$, $S = 1{\cdot}84$, max.

Table 1. *Fractional atomic coordinates and* $B_{\rm eq}$ (Å^2) *with their e.s.d.'s in parentheses*

$$B_{\rm eq} = (4/3)\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Cl(1)	0·2781 (1)	0·2022 (1)	0·75000	5·09 (5)
Cl(2)	0·0185 (1)	0·1916 (1)	0·75000	4·92 (5)
O(1)	−0·1315 (3)	−0·1139 (3)	0·75000	5·0 (1)
O(2)	0·3897 (3)	0·0054 (3)	0·75000	4·0 (1)
O(3)	0·6444 (3)	−0·1373 (3)	0·75000	5·1 (1)
O(4)	0·6144 (3)	0·0353 (3)	0·75000	5·0 (1)
C(1)	0·2767 (4)	−0·0085 (4)	0·75000	3·5 (1)
C(2)	0·2120 (4)	0·0828 (4)	0·75000	3·6 (1)
C(3)	0·0955 (4)	0·0771 (4)	0·75000	3·6 (1)
C(4)	0·0400 (4)	−0·0194 (4)	0·75000	3·7 (1)
C(5)	0·1069 (4)	−0·1096 (4)	0·75000	4·0 (1)
C(6)	0·2227 (4)	−0·1053 (4)	0·75000	4·2 (2)
C(7)	−0·0826 (4)	−0·0301 (5)	0·75000	4·1 (1)
C(8)	0·4590 (4)	−0·0866 (4)	0·75000	3·8 (1)
C(9)	0·5800 (4)	−0·0529 (4)	0·75000	3·7 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with their e.s.d.'s in parentheses*

C(2)—Cl(1)	1·714 (5)	C(3)—Cl(2)	1·723 (5)
C(1)—O(2)	1·349 (6)	C(1)—C(2)	1·395 (7)
C(1)—C(6)	1·392 (7)	C(2)—C(3)	1·380 (7)
C(3)—C(4)	1·396 (7)	C(4)—C(5)	1·398 (7)
C(4)—C(7)	1·458 (8)	C(5)—C(6)	1·370 (7)
C(7)—O(1)	1·216 (7)	C(8)—O(2)	1·432 (6)
C(8)—C(9)	1·495 (7)	C(9)—O(3)	1·320 (6)
C(9)—O(4)	1·198 (6)		
O(2)—C(1)—C(6)	124·9 (4)	O(2)—C(1)—C(2)	115·8 (4)
C(2)—C(1)—C(6)	119·3 (4)	C(1)—C(2)—C(3)	120·3 (4)
Cl(1)—C(2)—C(1)	119·6 (4)	C(2)—C(3)—C(4)	121·1 (5)
Cl(2)—C(3)—C(4)	120·0 (4)	C(3)—C(4)—C(5)	117·4 (5)
C(4)—C(5)—C(6)	122·2 (5)	C(1)—C(6)—C(5)	119·7 (5)
O(1)—C(7)—C(4)	123·8 (5)	O(2)—C(8)—C(9)	108·2 (4)
O(3)—C(9)—O(4)	124·8 (5)	C(8)—C(9)—O(4)	126·6 (5)
C(8)—C(9)—O(3)	108·6 (4)	C(1)—O(2)—C(8)	117·4 (4)

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