Table 3. *Close intermolecular contacts between non-hydrogen atoms*

Symmetry operations listed were performed on the first atom. Distances are in Å.

F(3)···C(2)	$x, y-1, z-1$	3·416 (4)
C(1)···C(4)	$x-1, y, z$	3·427 (4)
F(2)···F(1)	$x-1, y, z$	3·414 (3)
F(2)···F(3)	$x-1, y, z$	3·300 (3)
N(9)···C(5a)	$x-1, y, z$	3·216 (3)
N(9)···C(6)	$x-1, y, z$	3·468 (4)
O(9*B*)···C(4a)	$x-1, y, z$	3·123 (3)
O(9*B*)···N(5)	$x-1, y, z$	3·192 (3)
O(9*B*)···C(5a)	$x-1, y, z$	3·161 (4)
O(9*B*)···C(9a)	$x-1, y, z$	3·155 (4)
O(9*B*)···N(10)	$x-1, y, z$	3·122 (3)
C(2)···N(9)	$1-x, 1-y, 1-z$	3·265 (4)
C(2)···O(9*A*)	$1-x, 1-y, 1-z$	3·499 (4)
C(2)···O(9*B*)	$1-x, 1-y, 1-z$	3·440 (3)
C(3)···O(9*A*)	$1-x, 1-y, 1-z$	3·348 (4)
C(1)···O(9*B*)	$2-x, 1-y, 1-z$	3·270 (4)
O(9*B*)···O(9*B*)	$2-x, 1-y, 1-z$	3·076 (2)
C(4)···F(1)	$-x, 1-y, 2-z$	3·315 (4)
N(5)···F(1)	$-x, 1-y, 2-z$	3·401 (3)
C(4)···F(1)	$1-x, 1-y, 2-z$	3·377 (4)
C(4)···F(2)	$1-x, 1-y, 2-z$	3·318 (3)
C(4a)···F(1)	$1-x, 1-y, 2-z$	3·202 (3)
C(5a)···C(6)	$1-x, 1-y, 2-z$	3·426 (4)
F(1)···N(10)	$1-x, 1-y, 2-z$	3·484 (2)
F(3)···C(8)	$1-x, 2-y, 2-z$	3·389 (2)
F(3)···O(9*A*)	$1-x, 2-y, 2-z$	3·304 (3)
F(2)···O(9*A*)	$2-x, 2-y, 2-z$	2·989 (2)
F(3)···O(9*A*)	$2-x, 2-y, 2-z$	3·429 (3)

displaced 0·05–0·06 Å in the opposite direction. If the molecules were not bowed, O(9*B*)–diazole ring contacts would be even shorter.

Fluorines of trifluoromethyl groups frequently have high thermal parameters (Chu & Napoleone, 1981; Allen, March & Nowell, 1984), and are sometimes disordered (Valle, Toniolo & Bonora, 1985). Consequently, the C—F bond distances reported are usually shorter than the distances reported here. In the packing environment for this molecule, the trifluoromethyl cannot rotate freely without incurring unfavorable short contacts with O(9*A*) (2·45 Å) and with C(2) (2·63 Å). The C—F distances of 1·34 Å for this structure are closer to the C—F single-bond length, 1·37 Å, calculated by Pauling (1960).

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N-n-Butylpyridinium Chloride

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Abstract. $C_9H_{14}N^+.Cl^-$, $M_r = 171·67$, orthorhombic, $P2_12_12_1$, $a = 8·095\ (3)$, $b = 10·621\ (4)$, $c = 11·419\ (5)$ Å, $V = 981·8\ (6)$ Å^3, $Z = 4$, $D_x = 1·16$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 3·3$ cm^{-1}, $F(000) = 368$, $T = 298\ (1)$ K, $R = 0·053$ for 1307 $[I > 3\sigma(I)]$ of 1676 unique reflections. *N-n*-butylpyridinium chloride (BPCl) was crystallized from a BPCl–$AlCl_3$ molten salt. The *n*-butyl chain of the cation is in extended staggered form and is approximately perpendicular to the plane of the pyridinium ring.

Introduction. Mixtures of *N-n*-butylpyridinium chloride (BPCl) and aluminium(III) chloride constitute an interesting continuum of ambient-temperature molten salt media which behave as somewhat unusual 'nonaqueous' solvents. These melts have been studied rather intensively by a number of investigators not only

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because of their intrinsic interest but also since they have a number of important industrial applications. (An excellent discussion of these media can be found in Gale & Osteryoung, 1983.)

In the course of a detailed study of complexation reactions (Taulelle & Popov, 1983; Rhinebarger, Rovang & Popov, 1984; Rhinebarger & Popov, 1986) we obtained single crystals of *N-n*-butylpyridinium chloride. Its structure is of interest to us and to others in the field.

Experimental. Crystals of BPCl were obtained by diluting a 45:55 mol% BPCl–$AlCl_3$ mixture with benzene and allowing the mixture to stand overnight. Resulting crystals were washed with benzene to remove the adhering melt and dried at $1\cdot3\times10^{-3}$ Pa and room temperature for one week to remove the benzene.

Irregular shape, colorless, 0·6 × 0·6 × 0·6 mm; Nicolet *P*3*F*; 15 reflections, $10 < \theta < 12\cdot5°$; empirical absorption correction based on ψ scans, 0·971 to 1·00 relative factors; $\sin\theta/\lambda < 0\cdot7049$ Å^{-1}; $0 \le h \le 11$, $0 \le k \le 14$, $0 \le l \le 16$; 400 292 250 to 314 600, 040 798 082 to 825 692, 004 1 380 978 to 1 430 714; 1683 measured; 1676 unique; $R_{\rm int} = 0\cdot003$, 12 data; 369 unobserved; $I < 3\sigma(I)$; direct methods; refinement using F; H atoms at calculated positions, those on C(8), C(9) and C(10) riding on attached atoms; anisotropic non-H atoms, isotropic H = 129 parameters; $R = 0\cdot053$, $wR = 0\cdot049$, $S = 0\cdot59$; $w = 1\cdot0$; $\Delta/\sigma < 0\cdot20$; $-0\cdot60\ (5) < \Delta\rho < 0\cdot56\ (5)$ e Å^{-3}; extinction $3\cdot21\times10^{-6}$; scattering factors (Cromer & Waber, 1974), f' and f'' (Cromer, 1974); *SDP* (Frenz, 1978).

Discussion. The refined atomic coordinates and isotropic temperature factors are presented in Table 1; bond distances and bond angles are given in Table 2; the molecular structure and the numbering of the atoms are shown in Fig. 1 and the stereographic packing diagram is shown in Fig. 2.*

Table 1. *Refined positional parameters (e.s.d.'s in parentheses) and thermal parameters*

	x	y	z	U(Å^2)
Cl(1)	0·4606 (1)	0·21108 (9)	0·10652 (8)	0·0503 (1)
N(1)	0·5985 (4)	0·4998 (3)	0·7124 (3)	0·0494 (8)
C(2)	0·5739 (5)	0·4242 (4)	0·8060 (3)	0·0512 (10)
C(3)	0·4216 (6)	0·4147 (4)	0·8551 (3)	0·0583 (11)
C(4)	0·2925 (6)	0·4827 (4)	0·8101 (4)	0·0659 (13)
C(5)	0·3194 (6)	0·5600 (4)	0·7155 (4)	0·0645 (11)
C(6)	0·4725 (6)	0·5670 (3)	0·6671 (3)	0·0576 (10)
C(7)	0·7661 (6)	0·5055 (6)	0·6573 (5)	0·0785 (13)
C(8)	0·7948 (6)	0·3980 (6)	0·5757 (4)	0·0963 (13)
C(9)	0·6848 (8)	0·3552 (6)	0·4952 (5)	0·1368 (25)
C(10)	0·7315 (7)	0·2536 (5)	0·4122 (5)	0·0912 (13)
H(2)	0·673 (5)	0·382 (4)	0·836 (3)	0·070 (13)*
H(3)	0·412 (5)	0·350 (4)	0·919 (4)	0·086 (15)*
H(4)	0·183 (5)	0·472 (4)	0·842 (3)	0·071 (14)*
H(5)	0·232 (5)	0·599 (4)	0·686 (4)	0·082 (15)
H(6)	0·495 (5)	0·622 (3)	0·599 (3)	0·075 (13)*
H(7*a*)	0·843 (6)	0·497 (4)	0·716 (4)	0·096 (16)*
H(7*b*)	0·761 (5)	0·584 (4)	0·616 (4)	0·081 (14)*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $(1/6\pi^2)\,[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

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

N(1)–C(2)	1·352 (5)	C(4)–C(5)	1·374 (7)
N(1)–C(6)	1·348 (6)	C(5)–C(6)	1·359 (7)
N(1)–C(7)	1·497 (6)	C(7)–C(8)	1·491 (8)
C(2)–C(3)	1·358 (6)	C(8)–C(9)	1·358 (8)
C(3)–C(4)	1·371 (6)	C(9)–C(10)	1·485 (8)
C(2)–N(1)–C(6)	120·4 (3)	C(4)–C(5)–C(6)	119·8 (4)
C(2)–N(1)–C(7)	119·3 (4)	N(1)–C(6)–C(5)	120·3 (4)
C(6)–N(1)–C(7)	120·2 (4)	N(1)–C(7)–C(8)	111·9 (4)
N(1)–C(2)–C(3)	120·3 (4)	C(7)–C(8)–C(9)	125·3 (5)
C(2)–C(3)–C(4)	119·9 (4)	C(8)–C(9)–C(10)	120·6 (5)
C(3)–C(4)–C(5)	119·2 (4)		

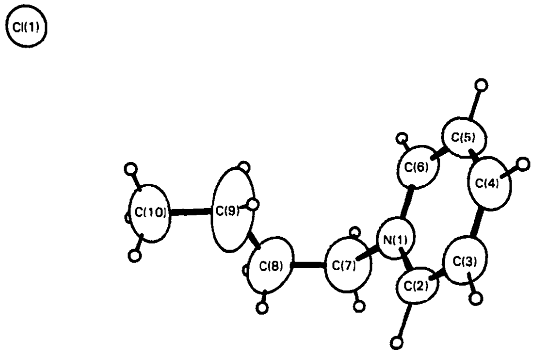

Fig. 1. The molecular structure and the numbering of the atoms. [*ORTEP* (Johnson, 1965), 50% probability ellipsoids.]

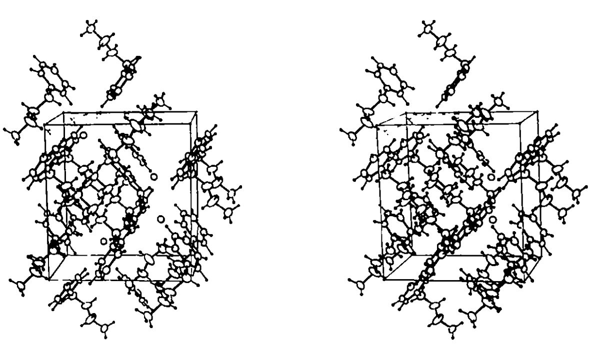

Fig. 2. Stereographic packing diagram. [*ORTEP* (Johnson, 1965), 20% probability ellipsoids.] The *c* axis is vertical, the *b* axis is horizontal and the *a** axis is normal to the page.

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* Lists of structure factors and tables of anisotropic thermal parameters, least-squares planes, torsion angles, root-mean-square amplitudes of thermal vibration, and atomic parameters and bond angles and distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43186 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The bond angles and distances within the pyridinium ring seem normal with angles ranging from 119·2 (4) to 120·4 (3)° and distances ranging from 1·352 (5) to 1·374 (7) Å; the ring is planar to within ±0·005 (5) Å. The *n*-butyl chain exhibits some signs of disorder involving C(9) and C(8) as shown by shortened bonds among the chain carbon atoms and by the instability during least-squares refinement of the H atoms bonded to C(8), C(9) and C(10). The H atoms bonded to C(8), C(9) and C(10) were finally placed in ideal positions and constrained to ride on their attached C atom to enable the least-squares refinement to converge with a reasonable model of the molecular structure.

The *n*-butyl chain is in the extended staggered form with a torsion angle C(7)—C(8)—C(9)—C(10) of 175·3 (5)°. The *n*-butyl chain junction with the pyridinium ring is of non-extended staggered form with the torsion angle N(1)—C(7)—C(8)—C(9) of 44·7 (7)°; therefore, the *n*-butyl chain lies approximately normal to the plane of the pyridinium ring with torsion angles C(2)—N(1)—C(7)—C(8) 81·5 (5) and C(6)—N(1)—C(7)—C(8) −96·9 (5)°.

The chloride ion lies in a hole bounded by the edges of pyridinium rings with five Cl···H contacts in the range 2·553 to 2·720 Å; four *n*-butyl chains give six Cl···H contacts in the range 3·133 to 3·389 Å. The shortest non-H contact with Cl is 3·516 Å and the shortest Cl···Cl distance is 4·794 Å to Cl^- ions at $\pm0\cdot5 + x, 0\cdot5 - y, -z$.

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Structures of Three Diels–Alder Adducts of Hexacyclo[10.2.1.0^{2,11}.0^{4,9}.0^{4,14}.0^{9,13}]-pentadeca-5,7-diene-3,10-dione

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Abstract. (V) Octacyclo[10.6.2.1^{5,8}.0^{2,6}.0^{2,11}.0^{4,9}.0^{7,11}.0^{13,18}]henicosa-15,19-diene-3,10,14,17-tetraone, $C_{21}H_{16}O_4$, $M_r = 332\cdot4$, monoclinic, $P2_1/m$, $a = 7\cdot175$ (1), $b = 11\cdot920$ (3), $c = 8\cdot622$ (1) Å, $\beta = 100\cdot18$ (1)°, $U = 725\cdot8$ Å^3, $Z = 2$, $D_m = 1\cdot54$, $D_x = 1\cdot52$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 9\cdot8$ mm^{-1}, $F(000) = 348$, $T = 130$ K, $R = 0\cdot042$ for 808 independent observed reflections. (VI) 15-Oxaoctacyclo[10.5.2.1^{5,8}.0^{2,6}.0^{2,11}.0^{4,9}.0^{7,11}.0^{13,17}]icos-18-ene-3,10,14,16-tetraone, $C_{19}H_{14}O_5$, $M_r = 322\cdot3$, monoclinic, $P2_1/n$, $a = 12\cdot521$ (3), $b = 6\cdot369$ (2), $c = 17\cdot604$ (4) Å, $\beta = 107\cdot09$ (2)°, $U = 1342$ Å^3, $Z = 4$, $D_m = 1\cdot59$, $D_x = 1\cdot60$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 10\cdot8$ mm^{-1}, $F(000) = 672$, $T = 130$ K, $R = 0\cdot052$ for 1578 independent observed reflections. (VII) 13,14-Bis(methoxycarbonyl)heptacyclo[10.2.2.1^{5,8}.0^{2,6}.0^{2,11}.0^{4,9}.0^{7,11}]heptadeca-13,15-diene-3,10-dione, $C_{21}H_{18}O_6$, $M_r = 366\cdot4$, monoclinic, $P2_1/c$, $a = 12\cdot257$ (4), $b = 6\cdot858$ (2), $c = 19\cdot610$ (5) Å, $\beta = 90\cdot09$ (2)°, $U = 1648$ Å^3, $Z = 4$, $D_m = 1\cdot44$, $D_x = 1\cdot48$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 10\cdot1$ mm^{-1}, $F(000) = 768$, $T = 130$ K, $R = 0\cdot038$ for 2104 independent observed reflections. The benzoquinone and maleic anhydride adducts, (V) and (VI), result from Alder attack of the dienophile on the carbonyl-bearing face of the diene, whereas the dimethyl acetylene

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