main, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least-squares methods minimized $\sum w(F_o - F_c)^2$ with $w = 4F_o^2[\sigma^2(I) + (0{\cdot}04F_o^2)^2]^{-1}$. Non-H atoms refined with anisotropic thermal parameters, H atoms placed at calculated positions and not refined. Final $R = 0{\cdot}043$, $wR = 0{\cdot}059$, $S = 1{\cdot}94$, for observed data. Max. $\Delta/\sigma = 0{\cdot}02$ in final cycle, max. residual density $0{\cdot}2$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atom coordinates and molecular dimensions are given in Table 2.* Fig. 1 (*ORTEP*; Johnson, 1976) shows the molecular structure and numbering scheme.

Related literature. Prior syntheses to related compounds are described in Weyenberg, Toporcer & Bey (1965), Nefedov, Manalcov & Petrov (1961) and Gilman & Atwell (1964).

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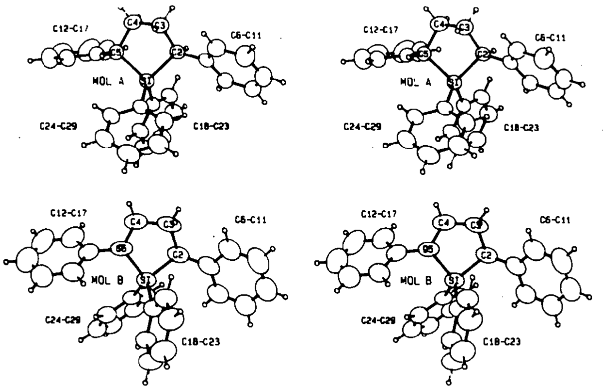

Fig. 1. Stereoviews of the two independent molecules. Thermal ellipsoids are drawn at the 50% probability level.

References

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Gilman, H. & Atwell, W. H. (1964). *J. Am. Chem. Soc.* **86**, 2687–2693.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Nefedov, O. M., Manalcov, M. N. & Petrov, A. D. (1961). *Izv. Akad. Nauk SSSR Otd. Khim. Nauk*, p. 1717.

Weyenberg, D. R., Toporcer, L. H. & Bey, A. E. (1965). *J. Org. Chem.* **30**, 4096–4101.

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* Lists of H-atom parameters, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44805 (75 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ethyl 1-Trifluoromethylindolizine-3-carboxylate

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Abstract. $C_{12}H_{10}F_3NO_2$, $M_r = 257{\cdot}2$, triclinic, $P\bar{1}$, $a = 4{\cdot}530\,(2)$, $b = 8{\cdot}834\,(4)$, $c = 14{\cdot}320\,(5)$ Å, $\alpha = 84{\cdot}85\,(2)$, $\beta = 87{\cdot}17\,(2)$, $\gamma = 84{\cdot}66\,(2)°$, $V = 567{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}50$ Mg m^{-3}, $F(000) = 264$, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}091$ mm^{-1}, $T = 293$ K, $R = 0{\cdot}074$ for 1434 unique reflexions [$F > 3\sigma(F)$]. The molecules are stacked within the crystal so that neighbouring five- and six-membered rings overlap [$C(2)\cdots C(8^i)$ 3·47(1) Å; (i) $1+x, y, z$]. A short C—C bond [1·437 (5) Å] linking the indolizine ring and the coplanar carboxy substituent suggests that the title compound has a dipole involving the ring N and carboxy O.

Experimental. The title compound was prepared by a 1,3-dipolar cycloaddition reaction of pyridinium ethoxycarbonylmethylide with 3,3,3-trifluoropropyne and recrystallized from a mixture of ethanol and methylene chloride.

Crystal dimensions 0·5 × 0·3 × 0·3 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo *Kα* radiation, unit-cell dimensions from

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Table 1. *Fractional atomic coordinates* (× 10^4) *and vibrational parameters* (Å^2 × 10^4) *for non-hydrogen atoms*

	x	y	z	U_{eq}*
C(1)	3375 (9)	299 (4)	7073 (3)	625
C(2)	4979 (9)	693 (4)	7797 (3)	622
C(3)	4407 (9)	2224 (4)	7890 (2)	621
N(4)	2362 (7)	2800 (3)	7210 (2)	609
C(5)	1091 (11)	4259 (4)	7019 (3)	748
C(6)	−864 (12)	4539 (5)	6318 (3)	815
C(7)	−1514 (12)	3370 (5)	5786 (3)	805
C(8)	−280 (10)	1938 (5)	5974 (3)	703
C(9)	1774 (9)	1615 (4)	6697 (2)	620
C(10)	3438 (11)	−1219 (5)	6740 (3)	777
F(1)	761 (7)	−1642 (3)	6642 (3)	1481
F(2)	4724 (7)	−1343 (3)	5897 (2)	1099
F(3)	4819 (7)	−2297 (3)	7293 (2)	1036
C(11)	5447 (11)	3165 (4)	8551 (2)	710
O(1)	4819 (8)	4513 (3)	8587 (2)	896
O(2)	7314 (7)	2330 (3)	9135 (2)	757
C(12)	8485 (16)	3145 (6)	9849 (4)	892
C(13)	10228 (18)	2022 (8)	10477 (4)	1028

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\mathbf{a}_i.\mathbf{a}_j a_i^* a_j^*$.

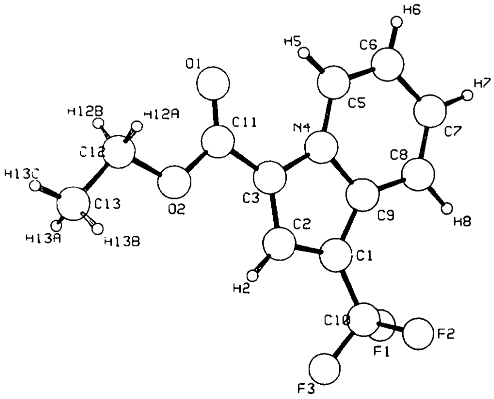

Fig. 1. The title molecule including atomic labelling scheme produced using *PLUTO* (Motherwell & Clegg, 1978).

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

C(1)—C(2)	1·382 (5)	C(1)—C(9)	1·392 (5)
C(1)—C(10)	1·460 (5)	C(2)—C(3)	1·370 (5)
C(3)—N(4)	1·407 (5)	C(3)—C(11)	1·437 (5)
N(4)—C(5)	1·372 (5)	N(4)—C(9)	1·385 (4)
C(5)—C(6)	1·362 (6)	C(6)—C(7)	1·398 (6)
C(7)—C(8)	1·344 (5)	C(8)—C(9)	1·418 (5)
C(10)—F(1)	1·319 (5)	C(10)—F(2)	1·323 (5)
C(10)—F(3)	1·314 (4)	C(11)—O(1)	1·204 (4)
C(11)—O(2)	1·342 (4)	O(2)—C(12)	1·449 (5)
C(12)—C(13)	1·477 (7)		
N(4)—C(3)—C(2)	107·0 (3)	C(5)—N(4)—C(3)	129·7 (3)
C(9)—N(4)—C(3)	108·3 (3)	C(9)—N(4)—C(5)	122·0 (3)
N(4)—C(9)—C(1)	107·5 (3)	C(8)—C(9)—C(1)	134·5 (3)
C(8)—C(9)—N(4)	117·9 (3)		

setting angles of 25 accurately centred reflexions (9·9 < θ < 15·9°), ω–2θ scan mode used to measure 2325 reflexions with $I > 0$, ω-scan width (°) of 0·90 + 0·35 tanθ and scan speed ranging from 0·6 to 5° min^{-1} according to the intensity gathered in a pre-scan, $-5 \leq h \leq 5$, $-10 \leq k \leq 10$, $0 \leq l \leq 16$, $0 < \theta < 25°$, 1434 unique structure amplitudes with $F > 3\sigma(F)$, $R_{\rm int} = 0{\cdot}028$, negligible drift in three intensity standards ($\bar{1}43$, $\bar{2}1\bar{3}$, $\bar{3}2\bar{4}$) measured every 3 h, Lorentz and polarization corrections but absorption ignored, *MULTAN*80 (Main, Hull, Fiske, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem, all non-hydrogen atoms found in a Fourier map, hydrogens from difference Fourier map, full-matrix least-squares refinement based on F using *SHELX*76 (Sheldrick, 1976), final $R = 0{\cdot}074$ $\{wR = 0{\cdot}081$, $w = 10{\cdot}8745/[\sigma^2(F_o) + 0{\cdot}00006F_o^2]\}$, anisotropic thermal parameters for heavier atoms, isotropic for hydrogen, empirical extinction correction applied [$F^* = F(1-1{\cdot}347 \times 10^{-5}F^2)/\sin\theta$]. Maximum fluctuation in final difference map in range −0·36 to 0·32 e Å^{-3}, $(\Delta/\sigma)_{\max} = 0{\cdot}022$ [U, H(13A)]. Scattering factors from *International Tables for X-ray Crystallography* (1974), computations carried out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre. Cambridge Crystallographic Database surveyed using the *Crystal Structure Search and Retrieval* interactive system (*CSSR*, 1984). The molecular structure is depicted in Fig. 1; atomic coordinates are listed in Table 1 and molecular geometry in Table 2.†

Related literature. Related structures are described by Aslanov, Ionov, Achmed Farah, Tafeenko, Vinogradova, Kost & Terent'ev (1976) and by Oeser (1972).

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† Lists of structure factors, hydrogen coordinates and anisotropic vibrational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44782 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Aslanov, L. A., Ionov, V. M., Achmed Farah, I. S., Tafeenko, V. A., Vinogradova, S. M., Kost, A. N. & Terent'ev, P. B. (1976). *Zh. Strukt. Khim.* **17**, 746–748.

CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual.* SERC Daresbury Laboratory, Warrington, England.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & **Clegg**, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Oeser, E. (1972). *Chem. Ber.* **105**, 2351–2357.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of 1,7-Heptanediamine Dihydrochloride

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Abstract. 1,7-Heptanediyldiammonium dichloride, $C_7H_{20}N_2^{2+}.2Cl^-$, $M_r = 203{\cdot}16$, monoclinic, $P2_1/c$, $a = 4{\cdot}648\,(2)$, $b = 16{\cdot}079\,(6)$, $c = 15{\cdot}027\,(4)$ Å, $\beta = 95{\cdot}52\,(3)°$, $V = 1117{\cdot}8$ Å^3, $Z = 4$, $D_m = 1{\cdot}20$, $D_x = 1{\cdot}207$ Mg m^{-3}, $\lambda(\text{Cu }K\bar{\alpha}) = 1{\cdot}54178$ Å, $\mu = 4{\cdot}94$ mm^{-1}, $F(000) = 440$, $T = 173$ K, final $R = 0{\cdot}031$ for 1562 unique observed reflections. The compound is isomorphous with the corresponding dibromide. The $[NH_3C_7H_{14}NH_3]^{2+}$ cation has an all-*trans* conformation. The NH_3^+ groups form N—H···Cl hydrogen bonds with the Cl^- ions with N—Cl distances ranging from 3·123 (2) to 3·339 (2) Å.

Experimental. Compound prepared by the same method as 1,3-diaminopropane dihydrochloride (Brisson & Brisse, 1982). Crystals grown from ethanol, 0·10 (010–0$\bar{1}$0) × 0·33 (001–00$\bar{1}$) × 0·46 (100–$\bar{1}$00) mm, density by flotation in chlorobenzene/bromobenzene. Enraf–Nonius CAD-4 diffractometer, cell dimensions and Laue symmetry from 25 centered reflections ($25 \le \theta \le 30°$) checked with oscillation photographs, data-collection procedure described elsewhere (Bélanger-Gariépy & Beauchamp, 1980), Cu $K\bar{\alpha}$ graphite-monochromatized radiation, $2\theta_{\max} = 120°$, $0 \le h \le 5$, $0 \le k \le 18$, $-17 \le l \le 17$; orientation checked every 100 measurements, intensity of seven standards checked every hour, max. fluctuation 3·1%, 1656 independent reflections measured, 1562 with $I > 3{\cdot}0\sigma(I)$. Data corrected for Lp and absorption, transmission range 0·23–0·60. Cl located from Patterson map, other atoms from ΔF map; structure refined on F by full-matrix least squares. H refined isotropically, other atoms anisotropically. Final $R = 0{\cdot}031$, $wR = 0{\cdot}043$ (weights based on counting statistics), $S = 1{\cdot}97$, mean shift/$\sigma = 0{\cdot}04$, max. $= 0{\cdot}10$. Residual electron density on final ΔF map: $-0{\cdot}23$ and $0{\cdot}21$ e Å^{-3}. Scattering factors for Cl, N and C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion f' and f'' terms for Cl from Cromer & Liberman (1970). Programs used listed elsewhere (Authier-Martin & Beauchamp, 1977). Final positional and isotropic thermal parameters are listed in Table 1,† and selected bond distances and angles in Table 2.

Table 1. *Refined coordinates* (× 10^4, Cl × 10^5, H × 10^3), U_{eq} (Å^2 × 10^3) *for* C, N, Cl and U_{iso} (Å^2 × 10^3) *for* H

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	11262 (8)	60510 (2)	45077 (3)	27
Cl(2)	5720 (8)	27511 (3)	128849 (3)	31
N(1)	6373 (3)	5766 (1)	6010 (1)	27
N(7)	5216 (3)	2316 (1)	11550 (1)	29
C(1)	4608 (3)	5421 (1)	6696 (1)	27
C(2)	6455 (3)	5030 (1)	7470 (1)	27
C(3)	4618 (3)	4665 (1)	8157 (1)	28
C(4)	6325 (4)	4168 (1)	8886 (1)	28
C(5)	4415 (4)	3813 (1)	9558 (1)	27
C(6)	5990 (3)	3251 (1)	10260 (1)	26
C(7)	3878 (4)	2902 (1)	10867 (1)	29
H(N1)1	736 (4)	540 (1)	582 (1)	35 (5)
H(N1)2	753 (4)	620 (1)	622 (1)	32 (5)
H(N1)3	502 (5)	597 (1)	551 (1)	48 (6)
H(N7)1	583 (4)	193 (1)	1131 (1)	35 (5)
H(N7)2	679 (4)	257 (1)	1191 (1)	42 (5)
H(N7)3	402 (4)	217 (1)	1194 (1)	42 (6)
H(11)	355 (4)	588 (1)	686 (1)	31 (5)
H(12)	333 (4)	503 (1)	641 (1)	33 (5)
H(21)	762 (4)	462 (1)	724 (1)	28 (5)
H(22)	784 (4)	546 (1)	774 (1)	33 (5)
H(31)	351 (4)	512 (1)	843 (1)	41 (5)
H(32)	316 (4)	433 (1)	785 (1)	37 (5)
H(41)	730 (4)	374 (1)	865 (1)	42 (6)
H(42)	787 (4)	453 (1)	920 (1)	32 (5)
H(51)	359 (4)	425 (1)	988 (1)	32 (5)
H(52)	294 (4)	348 (1)	921 (1)	33 (5)
H(61)	684 (4)	282 (1)	996 (1)	32 (5)
H(62)	754 (4)	354 (1)	1061 (1)	27 (5)
H(71)	313 (4)	337 (1)	1121 (1)	30 (5)
H(72)	232 (4)	260 (1)	1054 (1)	41 (5)

Related literature. 1,7-Heptanediamine dihydrochloride is isomorphous with the corresponding di-

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† Lists of observed and calculated structure-factor amplitudes, anisotropic thermal parameters, distances and angles involving H atoms, and data on the geometry of the hydrogen bonds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44727 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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