Fig. 1. Numbering scheme and hydrogen bonding between carboxyl groups across a twofold axis. A second independent twofold axis passes through C(2).

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Structure of N-(4-Amino-3-furazanyl)-2,2,2-trichloro-N'-methoxyacetamidine

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Abstract. $C_4H_6Cl_3N_4O_2$, $M_r = 248.48$, monoclinic, $P2_1/c$, $a = 9.287$ (1), $b = 9.493$ (1), $c = 13.208$ (2) Å, $\beta = 108.47$ (1)°, $V = 1104.5$ (3) Å^3, $Z = 4$, $D_x = 1.494$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 7.653$ mm^{-1}, $F(000) = 500$, $T = 295$ K, final $R = 0.047$, $wR = 0.060$ for 1400 independent observed reflections. The furazan ring is planar, while the adjacent primary and secondary amine groups deviate from the ring-atoms least squares plane by only 0.10 and 0.13 Å, respectively. In addition the acetamidine moiety is planar to within 0.04 Å, except for its three chlorine substituents. Intermolecular hydrogen bonding occurs between the secondary amine and ring nitrogen N(2).

Experimental. A clear 0.28 × 0.18 × 0.43 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident-beam graphite monochromator, 25 centered reflections within $40 \leq 2\theta \leq 60°$ used for determining lattice parameters. (Sin$\theta/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl: $0 \leq h \leq 10$, $0 \leq k \leq 10$, $-13 \leq l \leq 14$. Standards 406, 040, 006, monitored every 60 reflections with linear variation of 3.1% over data collection, $\theta/2\theta$ mode, scan width $(2.0 + \Delta_{\alpha_1\alpha_2})°$,

Table 1. Atom coordinates (×10⁴) and temperature factors (Å² × 10³)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cl(1)	10972 (1)	−3638 (1)	3684 (1)	74 (1)
Cl(2)	8459 (1)	−3377 (1)	1751 (1)	75 (1)
Cl(3)	8518 (1)	−5596 (1)	3237 (1)	81 (1)
O(1)	7273 (3)	1809 (2)	4102 (2)	49 (1)
C(4)	6396 (3)	48 (3)	3053 (2)	37 (1)
O(9)	6413 (2)	−2326 (2)	4397 (2)	47 (1)
N(6)	8729 (3)	−1478 (3)	3820 (2)	42 (1)
N(5)	6038 (3)	1324 (3)	3254 (2)	46 (1)
N(2)	8359 (3)	763 (3)	4430 (2)	44 (1)
C(3)	7852 (3)	−301 (3)	3797 (2)	35 (1)
C(7)	8169 (3)	−2843 (3)	3667 (2)	35 (1)
N(8)	7022 (3)	−3334 (3)	3879 (2)	39 (1)
C(11)	8986 (4)	−3829 (3)	3118 (3)	45 (1)
N(12)	5530 (3)	−733 (3)	2218 (2)	47 (1)
C(10)	5113 (5)	−2859 (5)	4601 (4)	75 (2)

Table 2. Bond lengths (Å) and bond angles (°)

Cl(1)–C(11)	1.767 (3)	Cl(2)–C(11)	1.767 (4)
Cl(3)–C(11)	1.752 (3)	O(1)–N(5)	1.403 (3)
O(1)–N(2)	1.383 (3)	C(4)–N(5)	1.306 (4)
C(4)–C(3)	1.435 (4)	C(4)–N(12)	1.360 (4)
O(9)–N(8)	1.396 (4)	O(9)–C(10)	1.411 (5)
N(6)–C(3)	1.377 (4)	N(6)–C(7)	1.387 (4)
N(2)–C(3)	1.301 (4)	C(7)–N(8)	1.272 (4)
C(7)–C(11)	1.525 (5)		
N(5)–O(1)–N(2)	110.2 (2)	N(5)–C(4)–C(3)	108.7 (2)
N(5)–C(4)–N(12)	123.1 (3)	C(3)–C(4)–N(12)	128.0 (3)
N(8)–O(9)–C(10)	110.6 (3)	C(3)–N(6)–C(7)	124.3 (3)
O(1)–N(5)–C(4)	105.7 (2)	O(1)–N(2)–C(3)	106.0 (2)
C(4)–C(3)–N(6)	128.7 (3)	C(4)–C(3)–N(2)	109.4 (3)
N(6)–C(3)–N(2)	121.6 (2)	N(6)–C(7)–N(8)	127.7 (3)
N(6)–C(7)–C(11)	115.0 (3)	N(8)–C(7)–C(11)	117.2 (3)
O(9)–N(8)–C(7)	110.8 (2)	Cl(1)–C(11)–Cl(2)	108.7 (2)
Cl(1)–C(11)–Cl(3)	108.4 (2)	Cl(2)–C(11)–Cl(3)	109.1 (2)
Cl(1)–C(11)–C(7)	110.2 (2)	Cl(2)–C(11)–C(7)	108.6 (2)
Cl(3)–C(11)–C(7)	111.7 (3)		

Hydrogen bonds

N···H	2.29 (4)	N···N	3.03 (1)
N···H–N	152.4 (2.8)		

scan rate a function of count rate (4·0° min⁻¹ minimum, 30·0° min⁻¹ maximum), 1793 reflections measured, 1514 unique, $R_{int} = 0.013$, 1400 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization effects. Empirical ellipsoidal absorption correction, max. and min. transmission = 0·57, 0·22, respectively. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00030$. Secondary-extinction parameter $p = 0.023$ (2) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin 2\theta]^{0.25}$. 149 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms, methyl H atoms included using riding model, C–H = 0·96 Å, H–C–H = 109·5°, $U(H) = 1.2U_{eq}(C)$. $(\Delta/\sigma)_{max} = 0.003$, $R = 0.047$, $wR = 0.060$, $S = 2.468$. Final difference Fourier excursions 0·43 and −0·41 e Å⁻³. Atomic scattering factors from International Tables for X-ray Crystallography (1974).† Table 1 gives atom coordinates, and Table 2 bond distances and angles. Atom numbering follows that shown in Fig. 1.

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† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43001 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. Thermal-ellipsoid plot of title compound with ellipsoids drawn at 20% probability level. Bonds between C(7)–N(8), N(2)–C(3) and C(4)–N(5) are formally double bonds.

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Structure of a 1,2-Diazepine Analogue of a Diaza[2₄](1,2,4,5)cyclophane*

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Abstract. Ethyl 1,2,5,6,9,10,13,14-octahydro-4,11-metheno-3,12-nitrilo-7H-dicycloocta[c,f][1,2]di-azepine-7-carboxylate, $C_{21}H_{23}N_3O_2$, $M_r = 349.4$, monoclinic, $P2_1$, $a = 10.727$ (1), $b = 9.581$ (1), $c = 8.498$ (1) Å, $\beta = 106.40$ (1)°, $V = 837.9$ Å³, $Z = 2$, $D_m = 1.37$, $D_x = 1.384$ g cm⁻³, Cu $K\alpha_1$, $\lambda = 1.54056$ Å, $\mu = 7.3$ cm⁻¹, $F(000) = 372$, $T = 296$ K, final $R = 0.051$ for 1267 unique observed reflections. The analysis confirms the proposed formulation. Inter-ring repulsion is inferred from bridging bond lengths as long as 1·578 (7) Å. The diazepine ring is a distorted boat; the four bridgehead atoms are roughly coplanar, and the three remaining atoms are bent away from the opposing pyridine ring.
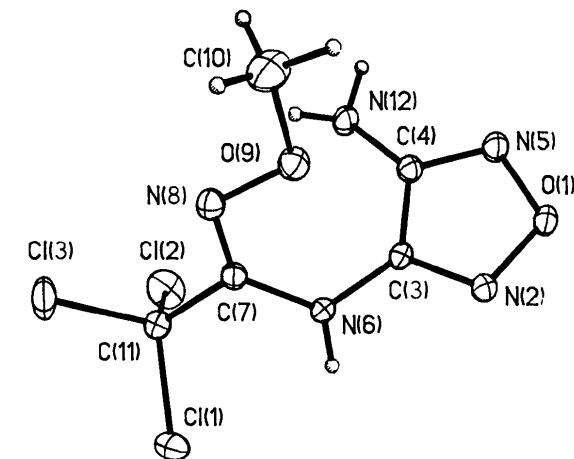
Experimental. Glassy, orange crystal of irregular shape, 0·2 × 0·3 × 0·4 mm, D_m by flotation in aqueous potassium iodide solution. Picker four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation, θ–2θ scan, $2\theta \le 130°$. Cell constants from 30 reflections, $50 \le \theta \le 65°$. Max. $(\sin\theta)/\lambda = 0.588$ Å⁻¹, $h = -12$ to 12, $k = 0$ to 11, $l = 0$ to 9. No absorption corrections. One standard reflection (040) measured every 60 reflections, mean intensity 100 466 (671) counts; no indication of specimen decay. 1523 unique reflections scanned, of which 256 unobserved $[I_{net} \le 3\sigma(I_{net})]$; σ

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* NRCC No. 25907.