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Structure of 2,4,6-Trichloroacetanilide

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Abstract. $C_8H_6Cl_3NO$, $M_r = 238{\cdot}5$, monoclinic, Pn (general equivalent positions: x, y, z; $\frac{1}{2}+x, -y, \frac{1}{2}+z$), $a = 8{\cdot}224\,(3)$, $b = 8{\cdot}237\,(3)$, $c = 7{\cdot}966\,(3)$ Å, $\beta = 113{\cdot}01\,(5)°$, $V = 496{\cdot}7\,(3)$ Å^3, $Z = 2$, $D_m = 1{\cdot}59\,(2)$, $D_x = 1{\cdot}595$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu(\text{Cu } K\alpha) = 80{\cdot}28$ cm^{-1}, $F(000) = 240$, $T = 295$ K, $R = 0{\cdot}055$, $wR = 0{\cdot}073$, 907 significant reflections. The amide group and phenyl ring are each planar (maximum deviations 0·028 and 0·043 Å respectively) but, to eliminate conflict between the amide oxygen atom and an adjacent chlorine substituent, the normals to these planes are at 69·4°. The molecules are N—H···O hydrogen bonded with H···O 1·91 Å and the angle at H 159·0°.

Experimental. Colourless crystal of unmeasured dimensions; 1268 unique reflections measured with Ni-filtered Cu $K\alpha$ radiation on a Picker automated four-circle diffractometer; 907 reflections observed with $I > 2\sigma(I)$. Cell dimensions from 16 centred reflections in the range $40 < 2\theta < 65°$. During data collection three standard reflections were measured every 25 reflections. Standard deviation 0·06%. Range of h, k, l: $-7 \le h \le 9$, $0 \le k \le 9$, $-7 \le l \le 9$; $(\sin\theta)/\lambda < 0{\cdot}59$ Å^{-1}. φ-sweep absorption corrections were made (North, Phillips & Mathews, 1968). The structure was solved from the Patterson function and refined on F using program *XRFLS* (Busing, Martin & Levy, 1962). $\sigma(I)$ was based on counting statistics and $\sigma(F_o)$ as $\{[\sigma(I)/(\text{Lp})A]^2 + 0{\cdot}02F_o^4\}^{1/2}/2F_o$ where A is the transmission factor. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962) with corrections for anomalous scattering of Cl atoms included. Hydrogen atomic positions were all indicated on ΔF maps but, on refinement, gave C—H and N—H bond lengths in the range 0·74–1·20 Å. They were thus placed in their best theoretical positions with C—H and N—H each 1 Å, and given isotropic temperature factors. $R = 0{\cdot}055$, $wR = 0{\cdot}073$ [$w = 1/\sigma^2(F)$], $S = 1{\cdot}36$. In the final refinement cycle the largest parameter change was for β_{22} of atom Cl(1), 0·76σ.§

The two molecules per cell and atomic numbering are given in y-projection in Fig. 1. Atomic fractional coordinates and equivalent isotropic temperature factors are given in Table 1.

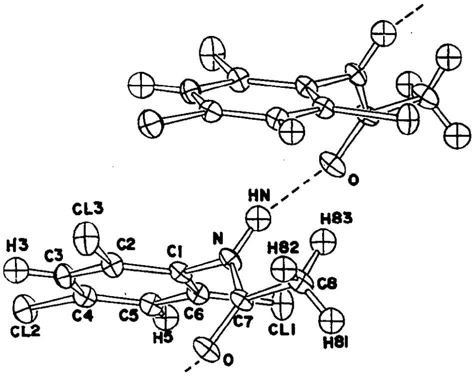

Fig. 1. *ORTEP* plot (Johnson, 1965) of cell content in y-projection with 50% probability ellipsoids.

Table 1. *Fractional atomic coordinates* (× 10^4), *e.s.d.'s and* B_{eq} *values* [*x,z of* Cl(1) *fixed arbitrarily*]

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Cl(1)	0	−1388 (2)	0	4·7 (1)
Cl(2)	−6299 (3)	468 (2)	−136 (4)	4·8 (1)
Cl(3)	−4075 (3)	−5633 (2)	1762 (4)	5·6 (1)
C(1)	−2136 (8)	−3411 (7)	861 (8)	2·9 (1)
C(2)	−3713 (9)	−3743 (7)	1053 (9)	3·5 (2)
C(3)	−4998 (9)	−2577 (8)	746 (10)	3·6 (2)
C(4)	−4714 (9)	−1018 (8)	206 (9)	3·5 (2)
C(5)	−3173 (9)	−638 (8)	4 (8)	3·3 (2)
C(6)	−1932 (8)	−1841 (7)	297 (8)	3·1 (1)
C(7)	−944 (8)	−5885 (7)	106 (8)	5·1 (1)
C(8)	657 (11)	−6914 (9)	616 (11)	4·3 (2)
N	−797 (7)	−4580 (6)	1202 (8)	3·3 (1)
O	−2311 (7)	−6183 (6)	−1228 (8)	4·4 (1)

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§ Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44286 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Related literature. The closest packing between chlorine atoms is Cl(1)···Cl(2), 3·88, Cl(1)···Cl(3), 3·85 and Cl(2)···Cl(3), 3·71 Å, in each case longer than the smallest possible distances calculated from the effective van der Waals radii of the Cl atom, 3·54, 3·36 and 3·40 Å respectively (Nyburg & Faerman, 1985).

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Structure of 2,6-Pyrido-18-crown-6*–Guanidinium Perchlorate–Deuterochloroform

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Abstract. $C_{15}H_{23}NO_5.CH_6N_3^+.ClO_4^-.CDCl_3$, $M_r = 577·27$, orthorhombic, $Pna2_1$, $a = 10·799$ (1), $b = 22·671$ (5), $c = 10·561$ (2) Å, $V = 2586$ (1) Å^3, $Z = 4$, $D_x = 1·48$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 5·1 cm^{-1}, $F(000) = 1200$, $T = 168$ K, final $R = 4·7$% for 1762 observed reflections. Each guanidinium cation is hydrogen-bonded to two 2,6-pyrido-18-crown-6 molecules, as the macrocyclic cavity of one crown molecule cannot encapsulate the cation completely. Similarly, each crown molecule is hydrogen-bonded with two cations. As a result, the structure consists of chains with an alternating sequence of crown molecules and cations. The perchlorate anion is involved in short contacts with two 2,6-pyrido-18-crown-6 molecules and one solvent molecule of deuterochloroform.

Experimental. The title compound was obtained in an extraction experiment. A solution of 1 mmol of 2,6-pyrido-18-crown-6 in 2 ml $CDCl_3$ was equilibrated with a solution of 2 mmol of guanidinium sulfate and 2 mmol $LiClO_4$ in 2 ml H_2O. The organic layer was separated off and the amount of guanidinium perchlorate that was extracted into the organic phase was determined from the intensities in the ^{1}H NMR spectrum. Only 0·32 mmol of guanidinium perchlorate proved to be transferred. Upon addition of 0·5 ml of diethyl ether the complex crystallized and was filtered off; m.p. 353–356 K (Uiterwijk, van Staveren, Reinhoudt, den Hertog, Kruise & Harkema, 1986).

Intensities were measured at 168 K on a Philips PW 1100 diffractometer (Mo $K\alpha$ radiation, graphite monochromator). .Lattice parameters determined by least squares from 25 centered reflections ($4·5 < \theta < 9·5°$). A total of 2404 independent reflections up to $\theta = 25°$ ($0 \leq h \leq 12$, $0 \leq k \leq 26$, $0 \leq l \leq 12$) were measured in the $\theta/2\theta$ scan mode (scan speed 0·05° s^{-1}, scan width 1·4°); 1762 reflections considered observed [$F_o^2 > 3\sigma(F_o^2)$]. The intensity variation of three standard reflections, measured every hour, was less than 3%. No absorption correction.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by full-matrix least squares. Weights for each reflection in the refinement (on F) were calculated from $w = 4F_o^2/\sigma^2(F_o^2)$, $\sigma^2(F_o^2) = \sigma^2(I) + (pF_o^2)^2$; the value of the instability factor p was determined as 0·06. All H atoms were located on difference Fourier maps; they were placed in calculated positions and treated as riding on their parent atoms [bond distance 0·96 Å, B_{iso}(H) = 1·2 B_{eq}(parent)]. The number of parameters refined was 308: scale factor, isotropic extinction parameter [final value 1·1 (6) × 10^{-7}], positional and anisotropic thermal parameters for the non-H atoms.

Refinement converged at $R = 4·7$%, $wR = 6·0$%, $(\Delta/\sigma)_{max} = 0·11$. Largest peak on final difference

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* IUPAC name: 3,6,9,12,15-pentaoxa-21-azabicyclo[15.3.1]-henicosa-1(21),17,19-triene.

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