

- BECKER, P. J. & COPPENS, P. (1974). *Acta Cryst.* A30, 129–147.
- BECKER, P. J. & COPPENS, P. (1975). *Acta Cryst.* A31, 417–425.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.
- GREENHOUGH, T. J. & TROTTER, J. (1980). *Acta Cryst.* In the press.
- SCHEFFER, J. R., BHANDARI, K. S., GAYLER, R. E. & WOSTRADOWSKI, R. A. (1975). *J. Am. Chem. Soc.* 97, 2178–2189.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

Acta Cryst. (1979). B35, 3087–3089

Hexachloroquinoxaline

By A. J. W. A. VERMEULEN AND C. HUISZON*

Chemical Physics Laboratory, Twente University of Technology, PO Box 217, Enschede, The Netherlands

(Received 2 July 1979; accepted 13 August 1979)

Abstract. C₈Cl₆N₂, monoclinic, *P*2₁/*c*, *a* = 13.168 (1), *b* = 3.9178 (4), *c* = 21.622 (3) Å, β = 101.22 (2)° at 293 K; *D*_x = 2.045 (1) Mg m⁻³ with *Z* = 4; Mo *K* α radiation (λ = 0.7107 Å); *R*_w = 0.068. The molecule is planar despite close intramolecular contacts between Cl atoms.

Introduction. A crystal of the title compound was obtained from Dr D. M. W. van den Ham of our laboratory. It was prepared by the method of Allison, Chambers, MacBride & Musgrave (1971). The crystal was needle-shaped with *b* as the long axis. The diameter was about 0.2 mm and the length 0.7 mm. Data were collected at room temperature, 293 K, with a computer-controlled diffractometer (Philips PW 1100), and graphite-monochromated Mo *K* α radiation. The built-in program for the determination of lattice constants (Hornstra & Vossers, 1973) was used.

Each set of the four equivalent reflexions *hkl*, *h \bar{k} l*, *h \bar{k} l* and *hk \bar{l}* was recorded in the θ range 2.5 to 30°, with the ω -2 θ scan mode, scan width 1.2°, scan speed 0.1° s⁻¹, horizontal detector aperture 2.0° and vertical detector aperture 1.5°. Background counting was for one half of the total scan time at each side of the reflexion.

As μ = 1.55 mm⁻¹, absorption corrections were applied. The resulting correction factors for the intensities ranged from 0.75 to 0.82.

The intensities of the non-equivalent reflexions were obtained as weighted means of the intensities of equivalent reflexions.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). From the *E* map all atom

positions were found. The refinement was carried out with a modified version of *ORFLS* (Busing, Martin & Levy, 1962). The function minimized was $\sum w(|F_o| - k|F_c|)^2$. The weight, *w*, for each reflexion was taken to be $w = \sigma^{-2}$ with $\sigma = s + 0.01|F_o|$, where *s* denotes the e.s.d. in $|F_o|$ due to counting statistics. To calculate *s* from the statistical error in the measured intensities, a normal distribution in $|F_o|$ was assumed (Rees, 1976). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The parameters varied in the last cycle were: positional parameters and anisotropic thermal parameters of all atoms, an isotropic extinction factor (Larson, 1969)

Table 1. *Fractional coordinates for hexachloroquinoxaline*

E.s.d.'s are shown in parentheses and refer to the last digit.

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	0.6752 (2)	0.6571 (9)	0.0008 (1)
C(2)	0.7348 (3)	0.566 (1)	-0.0366 (2)
C(3)	0.8299 (3)	0.391 (1)	-0.0151 (2)
N(4)	0.8606 (2)	0.3086 (9)	0.0437 (1)
C(5)	0.8296 (3)	0.322 (1)	0.1494 (2)
C(6)	0.7664 (3)	0.404 (1)	0.1904 (2)
C(7)	0.6712 (3)	0.574 (1)	0.1689 (2)
C(8)	0.6409 (2)	0.657 (1)	0.1061 (2)
C(9)	0.7051 (3)	0.572 (1)	0.0631 (2)
C(10)	0.7987 (3)	0.402 (1)	0.0848 (2)
Cl(11)	0.69707 (9)	0.6649 (3)	-0.11521 (5)
Cl(12)	0.90803 (8)	0.2680 (4)	-0.06640 (5)
Cl(13)	0.94702 (7)	0.1299 (3)	0.17513 (5)
Cl(14)	0.80247 (8)	0.3113 (3)	0.26904 (5)
Cl(15)	0.59167 (8)	0.6686 (3)	0.22067 (5)
Cl(16)	0.52662 (7)	0.8682 (3)	0.07883 (5)

* To whom correspondence should be addressed.

and the scale factor. The final R was 0.095 ($R_w = 0.068$).^{*} The number of reflexions used for the refinement was 3288, of which 2261 had $I > \sigma(I)$.

Discussion. Atomic positions are given in Table 1, bond lengths and angles in Fig. 1.

Usually, C_{2v} symmetry would be assumed for the molecule. This symmetry is indeed found for all C—C and C—Cl distances; the difference between the two N—C bond lengths may be significant but is still $< 3\sigma$.

The equation of the best plane through all atoms of the molecule is $0.9429x + 0.5619y + 0.2544z = 1$,

^{*} Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34681 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

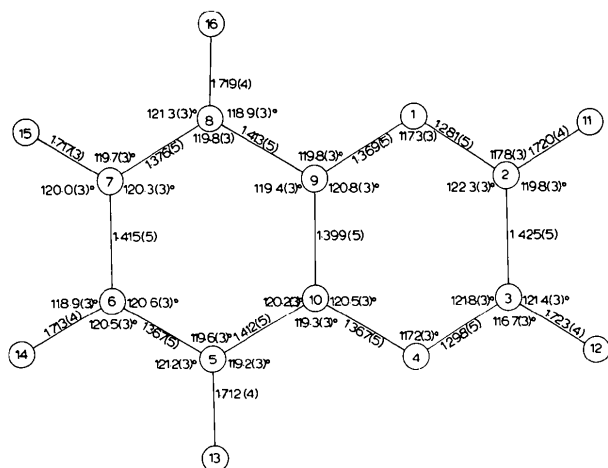


Fig. 1. Numbering of atoms, bond lengths (Å) and angles (°). E.s.d.'s are shown in parentheses and refer to the last digit.

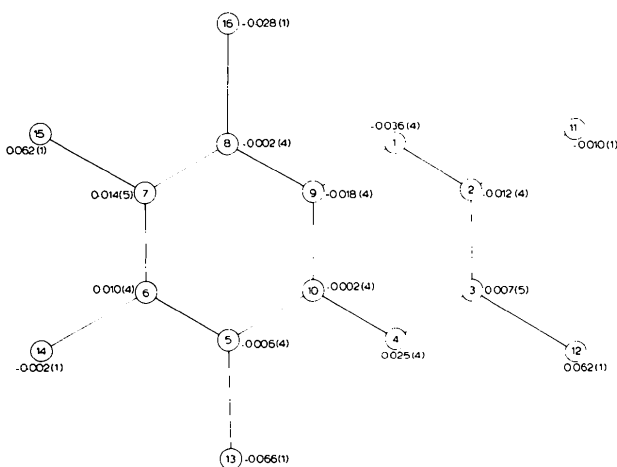


Fig. 2. Atomic deviations (Å) from the best plane through all the atoms of the molecule. A minus sign indicates a deviation away from the reader. E.s.d.'s are shown in parentheses and refer to the last digit.

Table 2. Intermolecular contacts between Cl atoms $< 3.7 \text{ \AA}$

E.s.d.'s are in parentheses.

	Symmetry operation applied to second atom	Distance (Å)
Cl(11)···Cl(12)	$x, 1 + y, z$	3.643 (4)
Cl(11)···Cl(14)	$x, 0.5 - y, -0.5 + z$	3.609 (4)
Cl(11)···Cl(15)	$x, 1.5 - y, -0.5 + z$	3.609 (4)
Cl(11)···Cl(16)	$1 - x, 2 - y, -z$	3.680 (4)
Cl(12)···Cl(13)	$2 - x, -y, -z$	3.653 (4)
Cl(12)···Cl(14)	$x, 0.5 - y, -0.5 + z$	3.573 (4)
Cl(13)···Cl(14)	$2 - x, -0.5 + y, 0.5 - z$	3.514 (4)
Cl(15)···Cl(15)	$1 - x, 0.5 + y, 0.5 - z$	3.531 (4)
Cl(15)···Cl(15)	$1 - x, -0.5 + y, 0.5 - z$	3.531 (4)
Cl(16)···Cl(16)	$1 - x, 2 - y, -z$	3.500 (4)

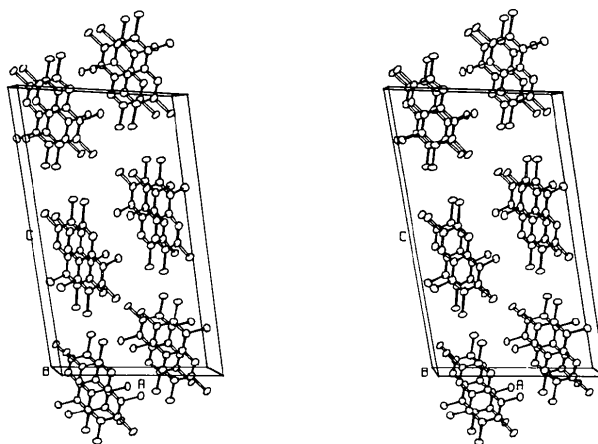


Fig. 3. Stereoscopic pair showing the molecular packing.

where x , y and z are fractional coordinates. The atomic deviations from this plane are given in Fig. 2. These deviations are very small, which is surprising because distances of the adjacent Cl atoms are all close to 3.12 \AA , which is considerably less than twice the van der Waals radius (1.8 \AA , Pauling, 1960).

Apparently, this does not introduce any appreciable deviation from the plane, in agreement with a phenomenon found in the related octachloronaphthalene (Gafner & Herbststein, 1963). In the latter compound, adjacent α - and β -Cl atoms are displaced in the same direction, although their mutual distances are only about 3.0 \AA , which is even less than in the present molecule. The intermolecular contacts between Cl atoms within 3.7 \AA (twice the van der Waals radius of Cl plus 0.1 \AA) have been listed in Table 2. It can be seen that in the present compound there are no such contacts between Cl atoms $< 3.5 \text{ \AA}$.

Fig. 3 is an ORTEP (Johnson, 1965) stereoview, showing the molecular packing. Owing to space-group symmetry, two sets of parallel molecules can be distinguished.

We thank Dr D. M. W. van den Ham for preparing the crystal and Mr G. J. van Hummel for technical assistance.

References

- ALLISON, C. G., CHAMBERS, R. D., MACBRIDE, J. A. H. & MUSGRAVE, W. K. R. (1971). *J. Fluorine Chem.* **1**, 59–67.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
- GAFNER, G. & HERBSTEIN, H. (1963). *Nature (London)*, **200**, 130–132.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- HORNSTRA, J. & VOSSERS, H. (1973). *Philips Tech. Rev.* **33**, 61–73.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- LARSON, A. C. (1969). *Crystallographic Computing*, edited by F. R. AHMED, pp. 291–294. Copenhagen: Munksgaard.
- PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., p. 260. Ithaca: Cornell Univ. Press.
- REES, B. (1976). *Acta Cryst.* **A32**, 483–488.

Acta Cryst. (1979). **B35**, 3089–3091

Refinement of the Structure of 5-Aminotetrazole Monohydrate

BY D. D. BRAY* AND J. G. WHITE

Chemistry Department, Fordham University, Bronx, New York 10458, USA

(Received 19 June 1979; accepted 13 August 1979)

Abstract. $\text{CH}_3\text{N}_5 \cdot \text{H}_2\text{O}$, monoclinic, $P2_1/c$, $a = 6.392$ (1), $b = 7.277$ (2), $c = 9.803$ (2) Å, $\beta = 90.24$ (2)°, $V = 456.1$ Å³, $Z = 4$, $D_x = 1.502$ Mg m⁻³. The structure has been refined by block-diagonal least-squares methods, using 828 independent diffractometer-measured intensity data to $R = 0.035$, $R_w = 0.030$ (observed reflections only). The ring H atom is ordered, and the refined positions of the water-molecule H atoms give a more acceptable H–O–H bond angle than that previously given.

Introduction. Crystals of the title compound were obtained during an attempted recrystallization of 2,4-dimethyltetrazolo[1,5-*a*]pyrimidine from water. Complete diffractometer X-ray data had been measured before it was realized that hydrolysis had occurred, and that the compound crystallized was, in fact, one of known crystal structure (Britts & Karle, 1967). Since the earlier work had been carried out using visual intensity estimates from film, and there had been some uncertainty in the indexing, it was decided to refine the structure using our diffractometer data.

The X-ray data were measured on an Enraf–Nonius CAD-4 automated diffractometer using Ni-filtered Cu $K\alpha$ radiation and a crystal measuring $0.10 \times 0.18 \times 0.33$ mm. The unit-cell parameters were determined by a least-squares fit of the angular settings for 15 high-angle reflections. The ω – 2θ scanning mode was used

with $\theta < 75^\circ$. The intensities were corrected for Lorentz–polarization effects, but not for absorption [$\mu(\text{Cu } K\alpha) = 1.12$ mm⁻¹]. Of 897 possible independent reflections 828 were accepted as observed on the criterion $I \geq 1.5\sigma(I)$.

Refinement was carried out by block-diagonal least-squares methods starting from the non-hydrogen coordinates of Britts & Karle (1967). A difference electron density map clearly showed the positions of all the H atoms. Refinement was then continued on all positional parameters, anisotropic thermal parameters for the non-hydrogen atoms and isotropic thermal parameters for the H atoms. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where w was obtained from the counting statistics for the observed reflections, while the unobserved reflections were weighted zero. In the final cycle $R = 0.035$, $R_w = 0.030$ for observed reflections only, and $R = 0.037$ for all reflections; the mean and maximum shift/e.s.d. were 0.08 and 0.33 respectively. The coordinates of all atoms and the isotropic thermal parameters for the H atoms are given in Table 1.† The scattering factors used for the nonhydrogen atoms are those given in *International Tables for X-ray Crystallography* (1962), and that for H is from Stewart, Davidson & Simpson (1965). All structural

† Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34677 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* To whom correspondence should be addressed.