146. Crystal and Molecular Structure of 7-(4-chloro-phenyl)-8-phenyl-2,3-dihydro-imidazo[1,2-*a*]pyrimidin-5(8*H*)-on

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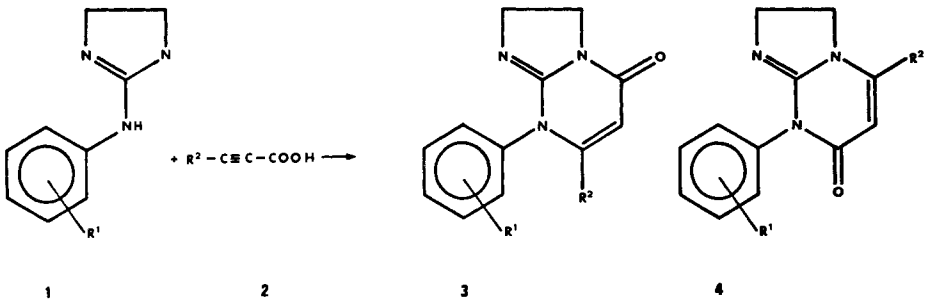
Zusammenfassung. Bei der Kondensation von 4-Chlorphenyl-propiolsäuremethylester mit 2-Anilino-2-imidazol entsteht die Titelverbindung, deren Struktur durch eine Röntgenstrukturanalyse bewiesen wurde. Die Kristalle gehören in die azentrische, orthorhombische Raumgruppe $Pna2_1$, $a = 13{,}49$, $b = 10{,}52$ und $c = 11{,}36$ Å, mit $Z = 4$ Molekeln pro Zelle. Die Struktur wurde nach der Schweratommethode gelöst und bis $R = 0{,}052$ verfeinert.

The 1,4-addition of propionic acid derivatives **2** to 2-anilino-2-imidazolines (**1**) leads to either of the isomeric pyrimidinones **3** or **4**. It was shown in the preceding paper [1] that the reaction is regiospecific, and that chemical and spectroscopic data indicated **3** to be the addition product.



1 **2** **3** **4**

For a final proof of the constitution of the product an X-ray analysis was undertaken with crystals of **3** ($R^1 = H$, $R^2 = p$–Cl–C_6H_4), obtained by reacting **1** (R = H) with **2** ($R^2 = p$–Cl–C_6H_4). The results confirm the earlier conclusions about the structure of **3**. They show, further, that the fused imidazoline and pyrimidine rings are approximately co-planar and that the C=N double bond is localized in the imidazoline ring as indicated in **3**, which explains the low pK (about 5.0) of the guanidine-like structure.

Crystallographic Data. – Well shaped prisms of the title compound (**3**) were obtained by crystallisation from methanol/water. The compound crystallises as the monohydrate $C_{18}H_{14}N_3OCl \cdot H_2O$, $M = 342.8$, in the orthorhombic system $Pna2_1$ (No. 33), with $a = 13.49(1)$, $b = 10.52(1)$, $c = 11.36(1)$ Å, $V = 1612$ Å^3, $Z = 4$, $d_c = 1.41$ g cm^{-3}.

Intensity data were collected on a CAD-4 diffractometer using graphite monochromatised Mo$K\alpha$ radiation, ω: 2θ-scan with $\Delta\omega = 0.8° + 0.3\ \mathrm{tg}\theta$, 6000 counts per reflexion with $t_{max} = 120$ s. A total of 1225 measurements of 1122 unique reflexions in the range $1.5 \leqslant \theta \leqslant 45°$ gave 803 reflexions with significant intensities ($I \geqslant 3\sqrt{P + B}$). Data reduction to absolute values of F by the method of *Wilson* [2] yielded the following statistics: $\bar{B} = 4.7$ Å^2, $<|E|> = 0.836$, $<|E^2 - 1|> = 0.829$, $<|E^2|> = 1.003$. No absorption correction was applied.

The structure was solved by the heavy-atom method and refined by least-squares procedures to a final $R = 0.052$ with 273 parameters (in addition to coordinates and anisotropic temperature factors for the heavier atoms, isotropic hydrogen atoms, a scale factor and an isotropic extinction coefficient – final value $G = 1.1(9)$ assuming $T = 0.02$ cm^{-1} [3] – were included). One molecule of water was found in a $(F_o - F_c)$ *Fourier* synthesis, but not the associated hydrogen atoms. Hydrogen atoms were otherwise introduced in calculated positions and included in the refinement. Final positions and standard deviations are presented in Table 1; a full list of parameters and structure factor tables may be obtained on request (HPW).

Results. – Fig. 1 shows a stereoscopic projection of the molecule with anisotropic vibrational ellipsoids. Fig. 2 gives the numbering of atoms, bond lengths, angles and some torsion angles. Standard deviations in atomic positions for C, N and O atoms are 0.008 to 0.010 Å (see Table 1), corresponding to standard deviations of 0.012 to 0.014 Å for bonds, 1.0 to 1.2° for angles and $\approx 1.6°$ for torsion angles. This compares quite well with the r.m.s. deviation of 0.015 Å calculated for the twelve bond lengths in the two benzene rings. C–H distances calculated from the final atomic positions (Table 1) vary between 0.86 and 1.27 Å, and no significance can be attached to them.

The imidazo[1,2-a]pyrimidine system shows small but significant deviations from planarity, $<d> = 0.036$ Å, $d_{max} = 0.09$ Å. The planes of the *p*-chlorophenyl and phenyl substituents make angles of 49° and 73° respectively with the mean plane of this system.

The packing of the molecules in the crystal is shown in Fig. 3. The water molecule makes only one hydrogen bond to N(4), the O...N distance being 2.95 Å; other

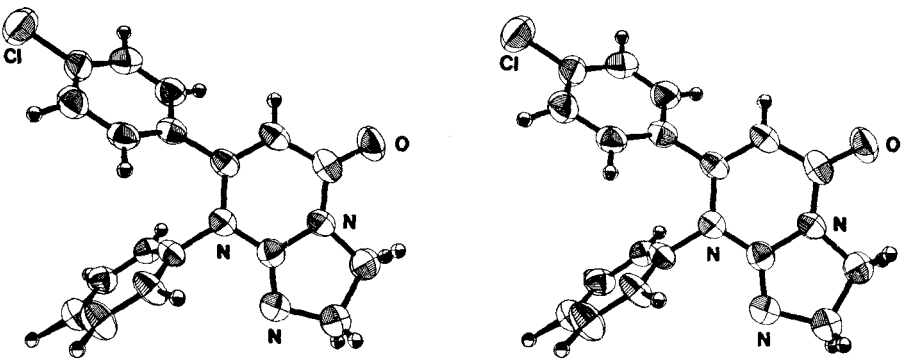


Fig. 1. *Stereoscopic projection of the molecule with anisotropic vibrational ellipsoids.* Hydrogen atoms have been given uniform isotropic B's of 1 Å^2 for the purpose of this drawing

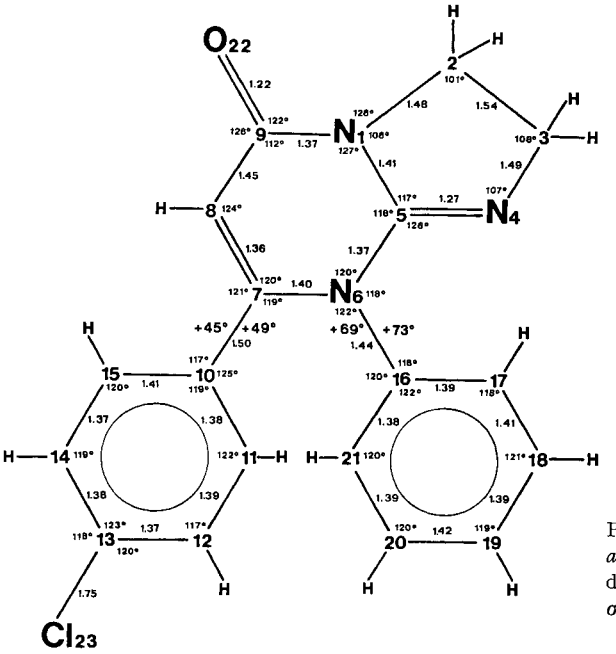


Fig. 2. *Bond lengths, bond angles and some torsion angles.* Standard deviations are σ(bond) $\approx$ 0.015 Å, σ(angles) $\approx$ 1.2°, σ(torsion angles) $\approx$ 1.6°

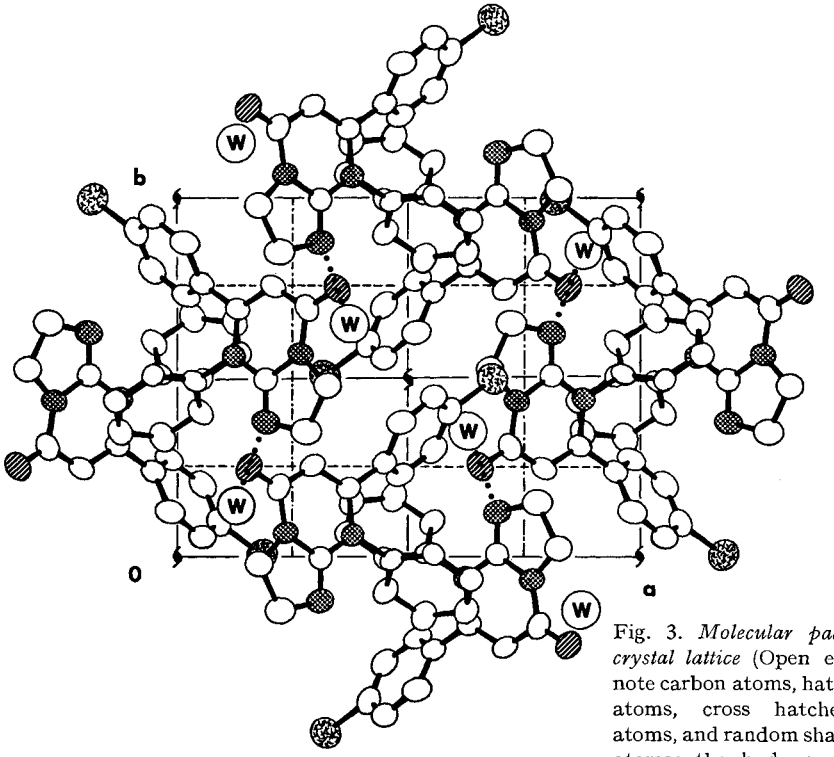


Fig. 3. *Molecular packing in the crystal lattice* (Open ellipsoids denote carbon atoms, hatched oxygen atoms, cross hatched nitrogen atoms, and random shaded chlorine atoms; the hydrogen bonds are marked by dotted lines)

Table. *Coordinates and LS-standard deviations* (in brackets). The values for C-, N- and O-atoms are multiplied by 10^4, those for H-atoms by 10^3

	x	y	z
N (1)	2337 (5)	650 (6)	3702 (7)
C (2)	1759 (7)	– 228 (9)	4452 (9)
C (3)	2215 (8)	–1517 (10)	4101 (11)
N (4)	3081 (6)	–1250 (7)	3322 (8)
C (5)	3089 (6)	– 59 (8)	3146 (8)
N (6)	3778 (4)	591 (6)	2496 (7)
C (7)	3695 (6)	1906 (8)	2371 (9)
C (8)	2921 (6)	2537 (8)	2867 (8)
C (9)	2192 (6)	1935 (9)	3618 (9)
C(10)	4475 (6)	2615 (8)	1700 (8)
C (11)	4816 (7)	2275 (8)	601 (8)
C (12)	5538 (7)	2982 (9)	16 (10)
C (13)	5881 (7)	4055 (10)	566 (10)
C (14)	5537 (7)	4457 (9)	1645 (10)
C (15)	4837 (8)	3742 (9)	2223 (10)
C (16)	4629 (6)	– 103 (8)	2085 (8)
C (17)	4496 (7)	– 965 (9)	1170 (9)
C (18)	5324 (7)	–1668 (11)	787 (10)
C (19)	6240 (6)	–1523 (9)	1322 (10)
C (20)	6341 (6)	– 654 (8)	2270 (10)
C (21)	5530 (5)	53 (8)	2640 (8)
O (22)	1546 (4)	2479 (7)	4175 (6)
Cl (23)	6818 (2)	4956 (3)	– 99 (0)
H (2A)	163 (8)	2 (13)	525 (12)
H (2B)	106 (6)	1 (9)	423 (9)
H (3A)	244 (9)	– 197 (14)	482 (14)
H (3B)	168 (5)	– 195 (7)	371 (8)
H (8)	286 (7)	333 (10)	272 (9)
H (11)	456 (7)	144 (10)	33 (11)
H (12)	586 (6)	259 (10)	– 64 (9)
H (14)	573 (6)	525 (9)	181 (9)
H (15)	461 (8)	393 (11)	296 (11)
H (17)	374 (4)	– 119 (5)	85 (5)
H (18)	517 (6)	– 229 (8)	0 (9)
H (19)	700 (6)	– 211 (9)	94 (9)
H (20)	698 (5)	– 45 (8)	267 (8)
H (21)	565 (5)	67 (8)	331 (8)
O (H_2O)	3725 (8)	6491 (9)	1992 (12)

potential hydrogen-bond acceptors (0(22), Cl) are not engaged in hydrogen bonding. There are no other unusually short intermolecular contacts.

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[1] *H. P. Härter, H. Lichti, U. Stauss, O. Schindler,* Helv. *59,* 1203 (1976).
[2] *A. J. C. Wilson,* Nature *150,* 151 (1942).
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