# 2-[3-(7-Chloro-2-methoxy-10-[benzo[*b*]-1,5-naphthyridinyl]amino)propylamino] ethanol (ICR-372-OH)

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The crystal structure of 2-[3-(7-chloro-2-methoxy-10-[benzo[*b*]-1,5-naphthyridinyl]amino)propylamino]-ethanol (ICR-372-OH), a weak mutagen for *Salmonella*, has been studied by X-ray diffraction using three-dimensional data. When the terminal hydroxyl group of the side chain is replaced by a chlorine atom the product (ICR 372) is a very powerful bacterial mutagen. The crystals are triclinic, space group $P\bar{1}$, with $Z=2$, F.W. 360·84, cell dimensions $a=9\cdot473$ (1), $b=9\cdot280$ (1), $c=10\cdot603$ (1) Å, $\alpha=99\cdot64$ (1), $\beta=101\cdot67$ (1), $\gamma=101\cdot80$ (1)°, $V=872\cdot1$ (2) Å³. The calculated and measured densities are 1·36 and 1·35 g cm⁻³ respectively. The structural formula is:

$$NH\text{-}(CH_2)_3\text{-}NH\text{-}(CH_2)_2\text{-}OH$$
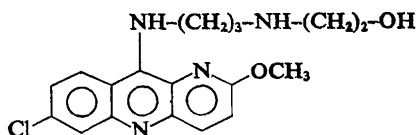
Three-dimensional diffractometer data were collected with Cu $K\alpha$ radiation. Of the 3266 reflections scanned, 788 were below the threshold of measurement. The structure was solved from the Patterson map and refined by a full-matrix least-squares procedure to the final residual $R=0\cdot054$. All hydrogen atoms were located from a difference map and were refined isotropically. The azaacridine ring system is approximately planar, the dihedral angle between the two outer rings being 4·2°. The nitrogen atom in the central ring forms a hydrogen bond (2·794 Å) to the side chain hydroxyl group of another molecule. The nitrogen atom in the ring with methoxy substituent does not take part in hydrogen bonding. There is an internal hydrogen bond between the two nitrogen atoms of the side chain, as found in other ICR compounds studied to date. The molecules stack in pairs with extensive overlap of the ring systems.

Experimental

Flat yellow crystalline plates of the compound were provided by Drs R. M. Peck and R. K. Preston of this Institute (Creech, Preston, Peck, O'Connell & Ames. 1972). A crystal of dimensions 0·03 × 0·03 × 0·02 cm was used to collect three-dimensional data on a Syntex automated diffractometer with Cu $K\alpha$ radiation (monochromatized with a highly oriented graphite crystal) using the θ–2θ scan technique. Intensities were measured for 3266 independent reflections. The raw data were corrected for intensity loss from X-rays (18% intensity loss for the standard reflections over 87 h of exposure). Values for $\sigma(F)$ were derived from counting statistics and measured instrumental uncertainties. The formula used was $\sigma(F)=(F/2)\{\sigma^2(I)/I^2+\delta^2\}^{1/2}$ where $\sigma(I)$ is derived from counting statistics alone and $\delta=0\cdot021$ is the measured instrumental uncertainty. There were 788 reflections for which the measured intensity, I_{obs}, was less than 3 $\sigma(I)$ and these were considered to be below the threshold of measurement. The intensity data were converted to structure amplitudes by application of Lorentz and polarization factors and an ellipsoidal absorption correction (Johnson, 1963), and

placed on an absolute scale with a Wilson plot. Crystal data are given in Table 1.

Table 1. *Crystal data for* 2-[3-(7-*chloro*-2-*methoxy*-10-[*benzo*[*b*]-1,5-*naphthyridinyl*]*amino*)*propylamino*]-*ethanol* (*ICR*-372-OH)

Formula: $C_{18}H_{21}ClN_3O_2$	F.W. 360·84
Crystal system: triclinic	
$a=$ 9·473 (1) Å	$\alpha=$ 99·64 (1)°
$b=$ 9·280 (1)	$\beta=101\cdot67$ (1)
$c=$ 10·603 (1)	$\gamma=101\cdot80$ (1)
(from 15 reflections measured on a diffractometer with Cu α_1, α_2 peaks resolved, Cu $K\alpha$ radiation, $\lambda=1\cdot5405$ Å)	
$V=872\cdot1$ (2) Å³	$F(000)=380$
$D_x=1\cdot36$ g cm⁻³	$Z=2$
$D_m=1\cdot35$ g cm⁻³ (in aqueous KI)	
μ(Cu $K\alpha$)=20·94 cm⁻¹	Max. sin $\theta/\lambda=0\cdot607$ Å⁻¹
Space group: $P\bar{1}$	Systematic absences: none
$|E|=0\cdot75$, $|\overline{E^2}|=1\cdot00$, $|\overline{E^2-1}|=1\cdot07$	

Structure determination and refinement

The structure was solved from the Patterson map with the aid of vector superposition maps. For the initial trial structure the residual was 0·487. This was reduced

to 0·386 by two cycles of isotropic full-matrix least-squares refinement. The chlorine atom was moved slightly, as indicated by a difference map, and then refined anisotropically, with all other atoms refined isotropically, by two more cycles of full-matrix least-squares calculations. The R value was reduced from 0·405 to 0·238 and then to 0·114 when all heavy atoms were refined anisotropically in two cycles. All hydrogen atoms were located from a difference Fourier synthesis. An extinction correction $\alpha = 6·5 \times 10^{-6}$ was applied to the data (Zachariasen, 1963). After four cycles of least-squares refinement with heavy atoms refined anisotropically and hydrogen atoms isotropically, the R value was 0·054.* The weights used in the refinement were $1/[\sigma^2(F_o)]$ with unobserved reflections assigned zero weight. The quantity minimized was $\sum\{||F_o| - |F_c||\}^2$. The weighted R value was 0·061. The maximum value of a parameter shift/e.s.d. was 0·15 for the heavy atoms in the last cycle of refinement.

The atomic scattering factors used for chlorine, oxygen, nitrogen and carbon atoms were those in *International Tables for X-ray Crystallography* (1962) and for hydrogen atoms those of Stewart, Davidson & Simpson (1965). The anomalous dispersion correction for the chlorine atom ($\Delta f'$) is listed by Cromer & Liberman (1970). Computer programs used in this determination were the X-RAY 70 System (Stewart, Kundell & Baldwin, 1970), and *UCLALS*4 (full-matrix least-squares) (Gantzel, Sparks, Long & Trueblood, 1969), modified by H.L.C.

The final atomic parameters are given in Table 2.

Discussion

The conformation of the molecule is shown in Fig. 1 in which the thermal ellipsoids are illustrated. Interatomic distances and interbond angles are given in

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30728 (26 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.
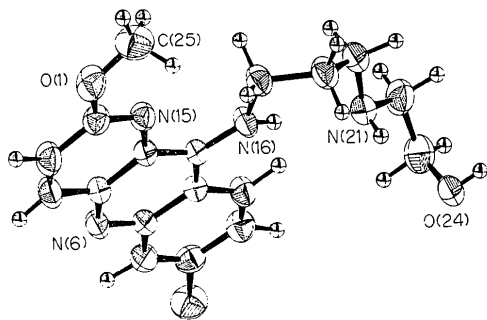

Fig. 1. Thermal ellipsoids for ICR-372-OH drawn with the computer program *ORTEP* (Johnson, 1965). The internal hydrogen bond is formed between N(16)-H···N(21).

Fig. 2, together with estimated standard deviations. As shown in Fig. 1, the molecule consists of a ring system that is nearly planar and a long side chain. The deviations from planes through portions of the molecule are listed in Table 3. These calculations indicate that there is an angle of 4·2° between the best planes through the outer rings of the ring system. This value may be compared with values of 8° for quinacrine (Courseille, Busetta & Hospital, 1973), 4·9° for ICR-191-OH (Carrell, 1972), 7·5° for ICR-171-OH (Glusker, Minkin & Orehowsky, 1972) 10·7° for ICR-170-OH (Berman &

Table 2. *Final atomic parameters*

Positional parameters are given as fractions of cell edges. Anisotropic temperature factors are expressed as:
exp $[-\frac{1}{4}(h^2a^{*2}B_{11}+k^2b^{*2}B_{22}+l^2c^{*2}B_{33}+2hka^*b^*B_{12}+2hla^*c^*B_{13}+2klb^*c^*B_{23})]$ and isotropic temperature factors as: exp $(-B \sin^2 \theta/\lambda^2)$ with B values given in Å². The standard deviation for each parameter, determined from the inverted full matrix, is given in parentheses and applies to the last specified digits. Fractional coordinates are $\times 10^3$ for hydrogen atoms, $\times 10^4$ for other atoms.

	x	y	z	B
Cl(17)	−5463 (1)	2376 (1)	4156 (1)	
O(1)	4209 (2)	10552 (2)	8240 (2)	
C(2)	2972 (3)	9340 (3)	7879 (2)	
C(3)	2716 (3)	8583 (3)	8898 (3)	
C(4)	1482 (3)	7452 (3)	8625 (2)	
C(5)	491 (3)	7016 (3)	7336 (2)	
N(6)	−740 (2)	5892 (2)	7130 (2)	
C(7)	−1625 (3)	5476 (3)	5894 (2)	
C(8)	−2941 (3)	4303 (3)	5657 (3)	
C(9)	−3862 (3)	3810 (3)	4440 (3)	
C(10)	−3568 (3)	4431 (3)	3370 (3)	
C(11)	−2325 (3)	5562 (3)	3571 (2)	
C(12)	−1311 (3)	6130 (3)	4826 (2)	
C(13)	2 (3)	7323 (3)	5053 (2)	
C(14)	902 (3)	7796 (3)	6376 (2)	
N(15)	2146 (2)	8985 (2)	6685 (2)	
N(16)	316 (2)	7886 (2)	4028 (2)	
C(18)	1513 (3)	9116 (3)	3962 (3)	
C(19)	1625 (3)	8996 (3)	2530 (3)	
C(20)	226 (3)	8863 (3)	1532 (3)	
N(21)	−749 (3)	7330 (3)	1189 (2)	
C(22)	−2046 (4)	7126 (4)	101 (3)	
C(23)	−2785 (3)	5470 (4)	−453 (3)	
O(24)	−1829 (2)	4659 (2)	−923 (2)	
C(25)	4432 (4)	11368 (4)	7303 (4)	
H(3)	355 (3)	895 (3)	981 (3)	4·8 (7)
H(4)	129 (3)	691 (3)	928 (3)	4·6 (7)
H(8)	−315 (3)	390 (4)	644 (3)	5·5 (8)
H(10)	−436 (3)	396 (3)	241 (3)	4·0 (7)
H(11)	−219 (3)	606 (3)	279 (3)	5·1 (8)
H(16)	−33 (3)	759 (3)	327 (3)	5·3 (8)
H(18)	239 (3)	905 (3)	443 (2)	3·3 (6)
H(18')	135 (3)	1014 (3)	432 (3)	5·3 (8)
H(19)	206 (4)	808 (4)	228 (3)	7·8 (9)
H(19')	233 (3)	980 (3)	239 (3)	3·9 (6)
H(20)	42 (4)	912 (4)	68 (3)	7·2 (10)
H(20')	−36 (3)	956 (3)	195 (3)	5·7 (8)
H(21)	−12 (3)	677 (3)	96 (3)	5·5 (8)
H(22)	−177 (3)	756 (3)	−67 (3)	5·3 (7)
H(22')	−281 (3)	766 (4)	35 (3)	7·1 (9)
H(23)	−315 (3)	494 (3)	27 (3)	5·5 (8)
H(23')	−362 (3)	535 (4)	−119 (3)	7·7 (9)
H(24)	−153 (3)	508 (4)	−152 (3)	6·7 (9)
H(25)	472 (3)	1070 (4)	655 (3)	6·7 (8)
H(25')	513 (4)	1220 (5)	770 (4)	10·9 (13)
H(25'')	352 (3)	1170 (3)	691 (3)	5·1 (7)

Table 2 (cont.)

|	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cl(17)	4·25 (2)	4·11 (3)	6·10 (3)	−0·65 (2)	1·14 (2)	0·59 (2)
O(1)	4·35 (8)	4·80 (9)	3·27 (8)	0·07 (7)	−0·25 (6)	−0·02 (7)
C(2)	3·55 (10)	3·56 (10)	3·11 (10)	0·44 (9)	0·57 (8)	0·29 (8)
C(3)	4·54 (12)	4·70 (12)	2·69 (10)	0·75 (10)	0·32 (9)	0·58 (9)
C(4)	4·74 (11)	4·20 (11)	2·51 (9)	0·67 (10)	0·70 (9)	1·05 (9)
C(5)	3·82 (10)	3·16 (9)	2·61 (9)	0·87 (8)	0·93 (8)	0·74 (8)
N(6)	4·07 (9)	3·37 (8)	2·60 (7)	0·41 (7)	0·85 (7)	0·94 (7)
C(7)	3·56 (9)	2·88 (9)	2·88 (9)	0·68 (8)	0·97 (8)	0·81 (8)
C(8)	4·02 (10)	3·27 (10)	3·65 (10)	0·51 (8)	1·38 (8)	0·87 (9)
C(9)	3·28 (9)	2·97 (10)	4·24 (11)	0·35 (8)	0·96 (9)	0·43 (9)
C(10)	3·49 (10)	4·19 (11)	3·61 (11)	0·50 (9)	0·35 (9)	0·57 (10)
C(11)	3·56 (10)	3·85 (11)	2·83 (9)	0·49 (9)	0·47 (8)	0·94 (8)
C(12)	3·16 (9)	3·04 (9)	2·71 (9)	0·78 (7)	0·84 (8)	0·70 (8)
C(13)	3·54 (9)	2·76 (8)	2·47 (8)	0·76 (7)	0·94 (8)	0·80 (7)
C(14)	3·34 (9)	2·94 (9)	2·58 (8)	0·64 (8)	0·83 (8)	0·68 (8)
N(15)	3·47 (8)	3·21 (8)	2·77 (8)	0·26 (7)	0·62 (7)	0·59 (7)
N(16)	4·08 (9)	3·77 (9)	2·42 (7)	−0·11 (7)	0·54 (7)	1·09 (7)
C(18)	4·55 (11)	3·65 (11)	2·95 (9)	−0·26 (9)	0·78 (9)	0·93 (9)
C(19)	5·29 (12)	3·99 (11)	3·35 (10)	−0·24 (10)	1·41 (10)	1·12 (10)
C(20)	6·56 (15)	3·66 (11)	3·16 (10)	0·66 (11)	1·35 (11)	1·10 (9)
N(21)	5·04 (10)	3·75 (9)	2·94 (8)	1·04 (8)	0·94 (8)	0·82 (7)
C(22)	5·89 (14)	5·69 (15)	3·36 (11)	2·23 (13)	0·57 (11)	0·73 (11)
C(23)	4·57 (13)	6·04 (16)	3·67 (12)	0·91 (12)	0·25 (10)	0·74 (12)
O(24)	6·08 (9)	4·04 (8)	3·06 (7)	0·76 (7)	1·22 (7)	1·25 (6)
C(25)	4·86 (14)	4·35 (14)	6·57 (18)	−0·43 (12)	0·93 (14)	0·77 (14)
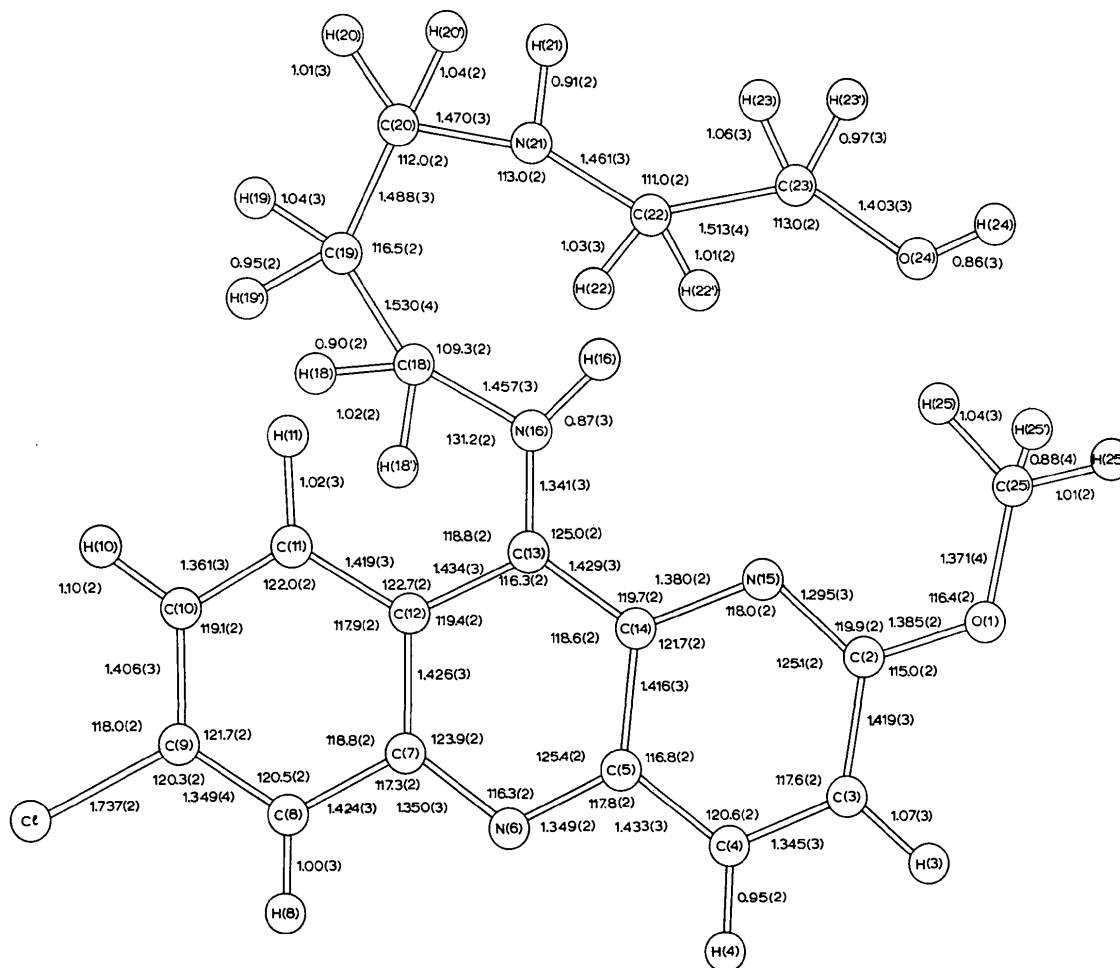
Fig. 2. Angles and distances in ICR-372-OH. Estimated standard deviations with respect to the last digit listed are given in parentheses.

Glusker, 1972) and 12·5° for ICR 449-OH (Glusker, Gallen & Carrell, 1973). In each of the ICR compounds, for which the formulae are shown in Fig. 3, there is an internal hydrogen bond between the two nitrogen atoms of the side chain. In this study of ICR 372-OH this internal hydrogen bond involves nitrogen atoms [N(16) and N(21)] which are separated by three methylene groups. In quinacrine these nitrogen atoms are separated by a chain of four carbon atoms and no such hydrogen bond forms.

It is interesting that N(16) and C(18) lie almost in the plane of the central ring (Plane 4, Table 3). The hydrogen atom on N(16) lies near H(11) [at 1·94 (3) Å] while the hydrogen atoms on C(18) lie nearer to N(15) [at 2·46 (2) and 2·91 (2) Å] than to C(11). Thus N(15) tends to lie in a hydrophobic environment. This tendency may influence the initial direction of the side chain. This is the opposite (with respect to the Cl and OCH₃ ring substituents) of that found in the other ICR compounds (which are not azacridines but are simple acridines) studied to date (see Fig. 3).

There is a strong hydrogen bond [2·794 (2) Å] (Table 4) between the nitrogen atom N(6) of the central ring and the hydroxyl group at the end of the side chain
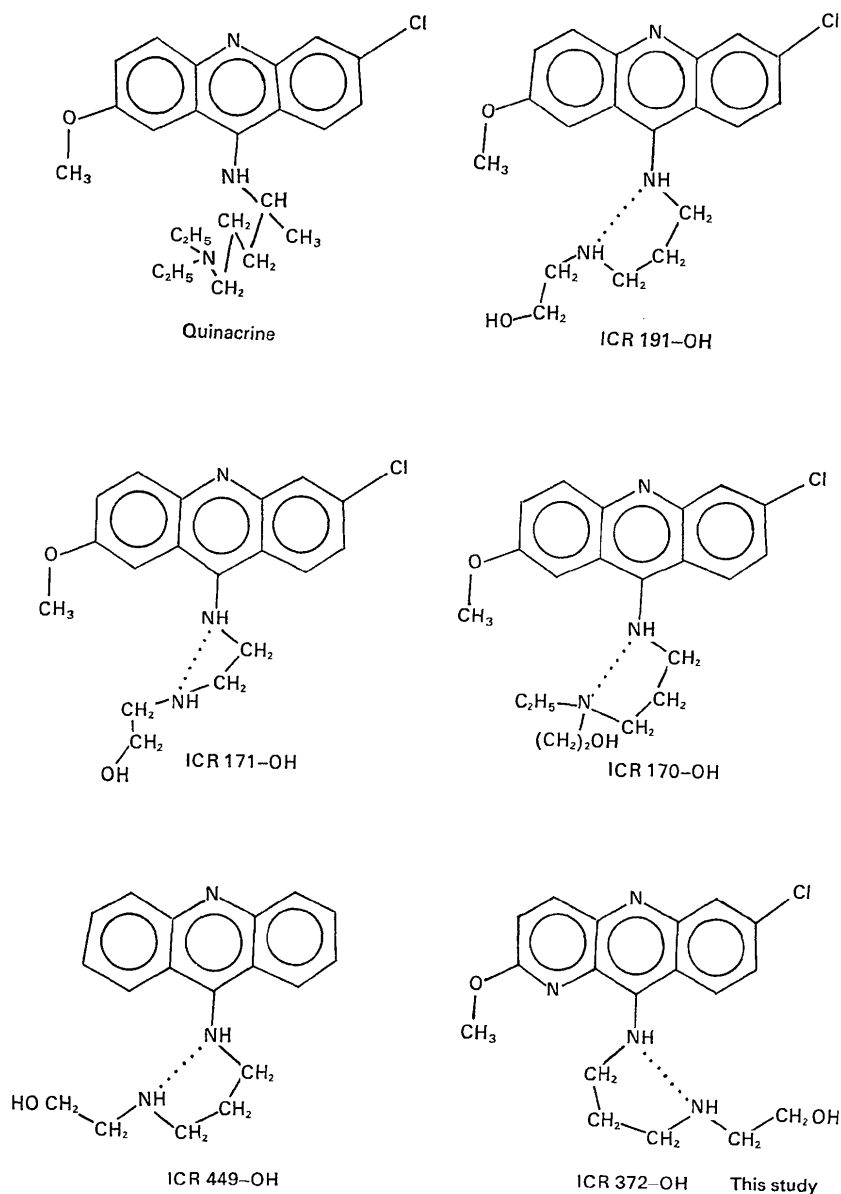

Fig. 3. Formulae of quinacrine and ICR compounds. The conformations of the ring systems and of the initial parts of the side chains are those found in X-ray crystallographic studies.

Table 3. *Some least-squares planes through parts of the molecule*

An asterisk denotes atoms used to calculate the planes. All deviations from the planes are in Å.

Deviations from the planes.

	Plane 1	Plane 2	Plane 3	Plane 4
Cl	−0·057	−0·111	−0·004	−0·055
O(1)	−0·130	−0·079	−0·176	−0·134
C(2)	*−0·046	*−0·017	−0·072	−0·046
C(3)	*0·047	*0·018	0·062	0·065
C(4)	*0·054	*0·000	0·090	0·077
C(5)	*0·003	*−0·020	0·019	*0·013
N(6)	*−0·010	−0·060	0·028	*0·005
C(7)	*−0·015	−0·034	*0·005	*−0·013
C(8)	*−0·045	−0·092	*−0·003	−0·038
C(9)	*−0·025	−0·045	*0·000	−0·030
C(10)	*0·019	0·056	*0·003	−0·005
C(11)	*0·037	0·103	*−0·001	0·008
C(12)	*0·020	0·058	*−0·002	*0·003
C(13)	*0·036	0·103	−0·010	*0·013
C(14)	*−0·012	*0·022	−0·037	*−0·021
N(15)	*−0·062	*−0·003	−0·108	−0·075
N(16)	0·124	0·245	0·039	0·083
C(18)	0·108	0·269	−0·007	0·058
C(19)	0·645	0·859	0·491	0·575
C(20)	0·016	0·253	−0·150	−0·064
N(21)	0·509	0·718	0·365	0·434
C(22)	0·026	0·260	−0·133	−0·060
C(23)	0·827	1·041	0·685	0·744
O(24)	2·196	2·421	2·045	2·110
C(25)	−0·310	−0·203	−0·394	−0·331

Equations of the planes
Plane

1	0·7415X	−0·6266Y	−0·2397Z	= −6·5484
2	0·7517X	−0·6017Y	−0·2700Z	= −6·6585
3	0·7321X	−0·6450Y	−0·2191Z	= −6·4753
4	0·7397X	−0·6331Y	−0·2280Z	= −6·4969

where X, Y, and Z are coordinates in orthogonal ångström space with X measured parallel to a, Y perpendicular to a in the plane of a and c, and Z perpendicular to the plane of a and c.

of another molecule. However the nitrogen atom, N(15), in the ring with the methoxy substituent does not take part in any hydrogen bonding but behaves as part of the hydrophobic aromatic system.

A view down c* of the unit-cell contents is shown in Fig. 4. The molecules pack in pairs across centers of symmetry and the extent of overlap of azaacridine rings is shown in Fig. 5. The weakness of the interaction between N(21) and O(24), which is probably too long to be called a hydrogen bond (N⋯O distance of 3·376 (2) Å, see Table 4), suggests that the stacking of rings may be a more important feature than hydrogen bonding for the packing in the crystalline state.

Unlike the inactive hydroxy precursors of the ICR 170 and ICR 191 mutagens the molecule studied (ICR 372-OH) is a weak mutagen. The compound in which a chlorine atom replaces the hydroxyl group at the end of the side chain (ICR 372) is one of the strongest bacterial mutagens tested in *Salmonella* (Creech *et al.*,
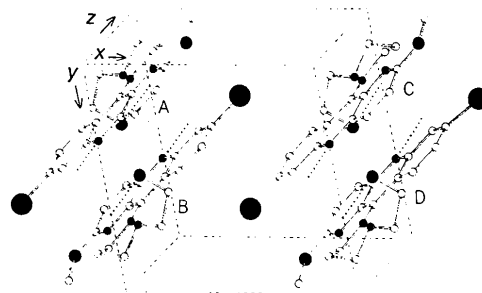

Fig. 4. View down c* of the unit-cell contents. Large black circles: chlorine. Medium black circles: oxygen. Small black circles: oxygen. Open circles: carbon. Molecules shown are A at $-x, 1-y, -z-1$; B at $x, y, z-1$; C at $1-x, 1-y, -z-1$; and D at $1+x, y, z-1$. The hydrogen bond between O(24) and N(6) is shown.
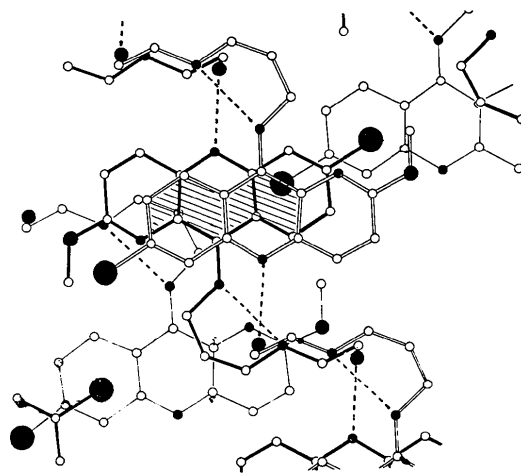

Fig. 5. Overlap of ring systems in the crystal. The direction of this projection is perpendicular to the least-squares plane (plane 1 in Table 3) through the three rings of the acridine portion of the molecule. Molecules drawn lie in planes 3·4 Å apart, the molecule with bonds drawn in black lying between the other two molecules. Areas of overlap of two molecules are shown by the shading. Bonds: — ring at −3·4 Å, — ring at 0 Å, = ring at +3·4 Å.

Table 4. *Distances (Å) and angles (°) in the hydrogen-bond system*

D–H⋯A	A⋯D	D–H	H⋯A	∠D–H⋯A	∠H–D⋯A
O(24)–H(24)⋯N(6^i)	2·794 (2)	0·86 (3)	1·94 (3)	175 (3)	4 (3)
N(16)–H(16)⋯N(21)	2·895 (3)	0·87 (3)	2·13 (3)	148 (3)	23 (3)
N(21)–H(21)⋯O(24^{ii})	3·376 (2)*	0·91 (2)	2·48 (2)	166 (3)	10 (3)

Code: i $x, y, z-1$; ii $-x, 1-y, -z$.

* This distance is probably too long to be designated a hydrogen bond.

1972). The acridine ring system of ICR-372-OH is fairly planar with an angle of 4·2° between outer rings compared with values of 4·9° for ICR-191-OH and 10·7° for ICR-170-OH. In this system of acridine derivatives it seems that the more powerful mutagens have the flatter ring systems and better stacking of ring systems.

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Two Crystal Structures of 2,4-Hexadiynylene Dibenzoate

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Crystals of one form ('*A*') of the title compound ($C_{20}H_{14}O_4$; F.W. 318·3) are monoclinic, $P2_1/c$, $a = 14·02$ (1), $b = 4·352$ (4), $c = 15·14$ (1) Å, $\beta = 118·9$ (1)°, $D_m = 1·306$ (2), $D_x = 1·306$ g cm^{-3}, $Z = 2$. Crystals of another form ('*B*') are similar except that $a = 9·665$ (9), $b = 8·479$ (8), $c = 11·03$ (1) Å, $\beta = 114·0$ (1)°, $D_m = 1·276$ (2), $D_x = 1·280$ g cm^{-3}. The crystal structures have been determined for both forms, and refined by block-diagonal least-squares analysis of diffractometrically measured intensities. Final R indices are 0·064 (1201 observed reflexions) for A and 0·085 (1326 observed reflexions) for B. In each structure the molecule was found to have essentially the same conformation, with the hexadiyne nucleus linear and the benzoate groups approximately planar. For A (which is polymerically reactive under high pressure) the closest approach between terminal atoms of the diacetylene nuclei of adjacent molecules is 4·02 Å. For the unreactive B form, the corresponding distance is 5·37 Å.

Introduction

Many substituted diacetylenes (I) undergo solid-state

$$R_1 \overset{1}{\underset{}{}}-\overset{2}{C}\equiv\overset{3}{C}-\overset{4}{C}\equiv C-R_2$$
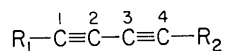
(I)

polymerization by 1:4 addition of adjacent nuclei (Wegner, 1972), while many do not; it appears that solid-state reactivity depends more on the crystal structure of the monomer than on the nature of the substituent, the latter being of importance only to the extent that it affects the former. The title compound

($R_1 = R_2 = -CH_2OCOC_6H_5$) is of interest because it crystallizes in two forms, one of which (A) is polymerically reactive under high pressure, while the other (B) is not (Baughman, 1972). The apparently crucial difference between the two crystal structures is that in the A form the smallest 1:4 distance for adjacent molecules is 4·02 Å, while for the B form it is 5·37 Å.

Experimental

The material crystallizes from methanol in the A form as transparent colourless prisms, elongated along **b**. The space group was determined from single-crystal photographs. The specimen used for intensity measure-