derivatives. For instance, (I) exhibits the $g^+aag^-aag^+ag^+g^+aa$ sequence that occurs in the sodium complex with 15-crown-5 or with N-phenyl-aza-15-crown-5 (Groth, 1981; Weber, Sheldrick, Dix & Vogtle, 1980). The same sequence is also exhibited by 15-crown-5 in an out-of-sphere complex with $CuBr_2^-$ $(OH_2)_2$ (Arte, Feneau-Dupont, Declercq, Germain & Van Meerssche, 1979). Moreover, (I) displays the smaller sequence $aag^-aag^+ag^+g^+a$ that is found in 15-crown-5 hydrogen bonded to an $Sm(ClO_4)_3$ complex (Lee, Sheu, Chiu & Chang, 1983). However, the analogy that one might draw from wedge representations is limited because of large differences (up to 39°) between the actual values of the torsion angles within the sequences that are apparently common to several rings. The exact geometrical arrangement of (I) is thus different from any other conformation reported to date including the structure suggested for uncomplexed 15-crown-5 in CS_2 on the basis of ^{13}C NMR studies (Dale, 1980).

The present crystallography analysis is entirely in keeping with earlier studies (Rubin *et al.*, 1984) indicating that odd-membered macrocyclic rings can adopt a variety of configurations.

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Structure of Arprinocid [9-(2-Chloro-6-fluorobenzyl)adenine], a Coccidiostat

BY S. K. ARORA*

Drug Dynamics Institute, The University of Texas at Austin, Austin, Texas 78712-1074, USA

AND S. B. LARSON

Department of Chemistry, The University of Texas at Austin, Austin, Texas 78712-1074, USA

Abstract. $C_{12}H_9ClFN_5$, $M_r = 277.69$, triclinic, $P\bar{1}$, $a = 8.715$ (2), $b = 11.210$ (2), $c = 7.435$ (1) Å, $\alpha = 92.5$ (1), $\beta = 121.8$ (1), $\gamma = 103.5$ (1)°, $V = 588.3$ Å^3, $Z = 2$, $D_m = 1.568$, $D_x = 1.567$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 3.2$ cm^{-1}, $F(000) = 284$, $T = 163$ K, $R = 0.045$ for 2038 observed reflections. The substituted benzyl moiety is disordered into two orientations. The adenine moiety is almost planar with N(6) displaced by 0.04 Å from the nine-atom least-squares plane. The dihedral angle between the least-squares planes of the adenine and substituted benzyl moieties has values of 86.2 and 87.8° (two orientations). The hydrogen bonding involves the usual N(6)—H$\cdots$N(1) and N(6)—H$\cdots$N(7) bonds.

Introduction. The veterinary use of drugs and antibiotics for the treatment of infectious diseases is based on similar principles to those in human medicine. Arprinocid, 9-(2-chloro-6-fluorobenzyl)adenine, is one of a class of 6-amino-9-(substituted)benzylpurines possessing broad-spectrum anticoccidiol activity (Miller *et al.*, 1977). There has also been great interest in the

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synthesis of (9-substituted)adenines because of their biological activity (Hartman, Buffar, Weinstock & Tull, 1978). Structural studies have been carried out on the above drug to study the orientation of the substituted benzyl with respect to the base and also the effect of 9-substitution on the geometry and stacking of the adenine ring system.

Experimental. The drug was kindly supplied by Merck, Sharp & Dohme Ltd and needle-shaped crystals were grown from ethanol, density measured by flotation (bromoform–benzene); crystal dimensions 0·54 × 0·30 × 0·11 mm; X-ray data from a Syntex $P2_1$ diffractometer using Mo $K\alpha$ radiation equipped with a graphite monochromator and a Syntex LT-1 inert-gas (N_2) low-temperature delivery system, lattice parameters refined using 60 reflections with 2θ values between 20·2 and 29·6°; 2718 unique reflections measured (h: 0 to 11, k: −14 to 14, l: −9 to 8) to the limit of $2\theta < 55\cdot0°$ using ω-scan technique; scan rate 3·0–6·0° min^{-1}; scan range 2·0° with a scan to background ratio of 1·0; 2038 reflections significant with $F_o > 4\sigma(F_o)$; $\sigma(F_o) = \sigma(F_o{}^2)/2F_o$, with $\sigma(F_o{}^2)$ based on counting statistics plus the term $0\cdot04F_o{}^2$. Lorentz and polarization corrections applied, absorption ignored. Three standard reflections, 2% intensity variation. Structure solved using direct-methods program *MULTAN* (Germain, Main & Woolfson, 1971); two orientations of the 2-chloro-6-fluorobenzyl ring were observed; occupancy factors for one orientation were refined (0·563); structure refined by least-squares method. $\Sigma[w(|F_o| − |F_c|)^2]$ minimized, where $w = 1/|\sigma(F_o)|^2$; H atoms located by difference electron-density maps; non-H atoms were refined with aniso-tropic temperature factors [except C(11)′ to C(16)′, which were refined with isotropic temperature factors], H atoms refined with isotropic temperature factors; final R for 2038 reflections 0·045 with $wR = 0\cdot0521$ and goodness-of-fit of 1·56; max. $|\Delta/\sigma| < 0\cdot01$; largest peak in $\Delta\rho$ map 0·36 e Å^{-3}, atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final positional and isotropic thermal parameters for the non-H atoms are given in Table 1.* The atoms with primes (′) refer to the second orientation of the 2-chloro-6-fluorobenzyl moiety, which is 180° from the first. The distances and angles in the adenine moiety agree well with the corresponding quantities found in other crystal structures containing adenine (Voet & Rich, 1970; McMullan, Benci &

* Lists of structure factors, bond distances and angles, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43543 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Craven, 1980; Kistenmacher & Rossi, 1977). The maximum deviations are 4σ in bond lengths and 5σ in bond angles. There has been no other benzyl-substituted-adenine structure studied, so it is hard to compare the geometry of the 2-chloro-6-flurobenzyl moiety. The N(9)—C(10) distance of 1·468 Å agrees with the similar distance (1·456 Å) observed in 9-[3-(3-indolyl)propyl]adenine (Bunick & Voet, 1982).

The adenine moiety of the molecule is approximately planar (Fig. 1). The torsion angles N(1)—C(6)—N(6)—HN(6)A and N(1)—C(6)—N(6)—HN(6)B have values of −175 and −5°, indicating the approximate co-planarity of the two H atoms at N(6) with N(1), C(6),

Table 1. *Atomic positions in fractional coordinates and U/U_{eq} for non-*H *atoms in arprinocid*

	x	y	z	U^*(Å^2)
N(1)	−0·0776 (2)	0·9086 (2)	0·7342 (3)	0·0283 (8)
N(3)	−0·1708 (2)	0·8012 (2)	0·3869 (3)	0·0281 (8)
N(6)	0·2347 (3)	0·9999 (2)	1·0189 (3)	0·0317 (9)
N(7)	0·3369 (2)	0·9135 (2)	0·7053 (3)	0·0331 (9)
N(9)	0·1023 (2)	0·8054 (2)	0·3702 (3)	0·0284 (8)
C(2)	−0·2014 (3)	0·8432 (2)	0·5311 (3)	0·0305 (10)
C(4)	0·0159 (3)	0·8326 (2)	0·4705 (3)	0·0247 (9)
C(5)	0·1613 (3)	0·8991 (2)	0·6764 (3)	0·0250 (9)
C(6)	0·1100 (3)	0·9383 (2)	0·8144 (3)	0·0252 (9)
C(8)	0·2918 (3)	0·8564 (2)	0·5185 (4)	0·0342 (11)
C(10)	0·0115 (3)	0·7404 (2)	0·1469 (3)	0·0293 (10)
C(11)	−0·1289 (4)	0·6058 (2)	0·0819 (6)	0·026 (2)
C(12)	−0·3178 (4)	0·5785 (2)	−0·0886 (6)	0·041 (2)
C(13)	−0·4413 (4)	0·4578 (2)	−0·1496 (6)	0·043 (2)
C(14)	−0·3757 (4)	0·3645 (2)	−0·0400 (6)	0·040 (3)
C(15)	−0·1867 (4)	0·3919 (2)	0·1305 (6)	0·034 (3)
C(16)	−0·0633 (4)	0·5126 (2)	0·1914 (6)	0·024 (2)
F	−0·3814 (12)	0·6589 (9)	−0·201 (2)	0·076 (5)
Cl	0·1670 (4)	0·5414 (4)	0·3913 (4)	0·0439 (8)
C(11)′	−0·0895 (7)	0·6074 (3)	0·1122 (10)	0·027 (2)
C(12)′	0·0048 (7)	0·5314 (3)	0·2477 (10)	0·044 (2)
C(13)′	−0·0917 (7)	0·4064 (3)	0·2174 (10)	0·044 (2)
C(14)′	−0·2827 (7)	0·3573 (3)	0·0515 (10)	0·041 (2)
C(15)′	−0·3770 (7)	0·4333 (3)	−0·0840 (10)	0·042 (2)
C(16)′	−0·2805 (7)	0·5583 (3)	−0·0536 (10)	0·0239 (13)
F′	0·1876 (15)	0·5725 (10)	0·401 (2)	0·084 (6)
Cl′	−0·3966 (7)	0·6536 (5)	−0·2229 (9)	0·0528 (14)

* For anisotropic atoms, the U value is U_{eq}, calculated as $U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.
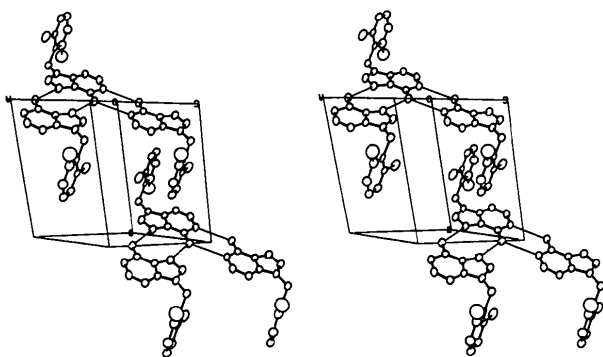

Fig. 1. Thermal-ellipsoid plot of the molecule showing the two rotamers.

Fig. 2. Stereoscopic view of the unit cell with hydrogen bonding.

and N(6). There is no folding (the dihedral angle being 0·2°) about the C(4)–C(5) bond in the adenine ring system. The r.m.s. deviation of the nine atoms of the purine group from their least-squares plane is 0·003 Å. The N(6) atom is displaced 0·04 Å from the least-squares plane of the purine ring system. The seven atoms of the benzyl moieties (two rotamers) have an r.m.s. deviation of 0·011 Å. The dihedral angle between the least-squares planes of the adenine and substituted benzyl moieties (two rotamers) has values of 86·2 and 87·8°.

Fig. 2 shows the packing of the molecules in the unit cell. The 2-chloro-6-fluorobenzyl rings stack over each other approximately perpendicular to the **b** direction of

the unit cell. There is no stacking observed between the adenine rings. The hydrogen-bonding interactions in the structure of arprinocid are also illustrated in Fig. 2. The adenine groups associate through N(6)–HN(6)A···N(7) and N(6)–HN(6)B···N(1) hydrogen bonds. These hydrogen-bond distances have values of 3·03 and 3·07 Å, respectively, with the proton-to-acceptor distances being 2·15 and 2·15 Å, respectively.

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Structure of [Bis(p-methoxyphenyl)](diethylaminocarbodithioato)iodine(III)

BY A. P. BOZOPOULOS AND P. J. RENTZEPERIS

Applied Physics Laboratory, Department of Physics, Aristotle University of Thessaloniki, Thessaloniki, Greece

Abstract. $C_{19}H_{24}INO_2S_2$, $M_r=489\cdot4$, monoclinic, $P2_1/c$, $a = 15\cdot296$ (1), $b = 14\cdot085$ (1), $c = 20\cdot766$ (1) Å, $\beta = 106\cdot813$ (5)°, $V = 4282\cdot67$ Å^3, $Z = 8$, $D_x = 1\cdot518$, $D_m = 1\cdot517$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot71$ mm^{-1}, $F(000) = 1968$, $T = 298$ K. Final $R = 0\cdot067$ for 3317 independent non-zero reflections. Two discrete molecules in the asymmetric unit. Two covalent I–C$_{ar}$ bonds [2·09 (2)–2·14 (2) Å] and one secondary I···S bond [2·927 (5), 2·902 (4) Å] form T-shaped coordination geometry around each of the two I atoms. A very distorted tetragonal coordination around each I atom is complemented by a weak I···S interaction. The crystal cohesion may be attributed to van der Waals forces.

Introduction. The structure of the title compound (BPDI hereafter) has been determined within the framework of a systematic study of diaryliodinanes, of which the structure of (2,2'-biphenylylene)-(1-pyrrolidinecarbodithioato)iodine(III) (BPPI) has already been reported (Bozopoulos & Rentzeperis, 1986).

Experimental. Yellow crystals, 0·30 × 0·30 × 0·40 mm, m.p. 381–383 K. D_m measured by flotation in carbon tetrachloride/chloroform. Computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. Cell parameters and standard deviations by least-squares analysis of measured θ angles of 100