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Lattice Inclusion Complexes of Gossypol.* Structure of the 1:1 Complex of Gossypol with Acetonitrile

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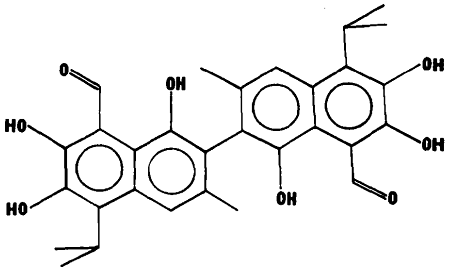
Abstract. $C_{30}H_{30}O_8.C_2H_3N$, $M_r = 559{\cdot}6$, triclinic, space group $P\bar{1}$, $a = 10{\cdot}938\,(2)$, $b = 10{\cdot}982\,(2)$, $c = 14{\cdot}162\,(3)$ Å, $\alpha = 77{\cdot}93\,(1)$, $\beta = 112{\cdot}24\,(1)$, $\gamma = 77{\cdot}25\,(1)°$, $V = 1452{\cdot}8\,(5)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54184$ Å, $\mu = 7{\cdot}19$ cm^{-1}, $F(000) = 592$, $T = 293$ K, $R = 0{\cdot}049$ for 2360 observed reflections. The 1:1 gossypol:acetonitrile complex belongs to the acetone-type lattice inclusion complexes of gossypol. Acetonitrile is the smallest guest forming this type of structure. The gossypol molecules form centrosymmetric dimers *via* a pair of O(5)—H···O(3) hydrogen bonds and these dimers are further connected into columns parallel to [0$\bar{1}$1]. The guest molecules are accommodated in channels of the host lattice and hydrogen bonded to the host O(1)—H group.

Introduction. Gossypol (see formula below), a physiologically active disesquiterpene (Vermel & Krouglyak, 1963; Vichkanova & Goryunova, 1968; National Coordinating Group on Male Antifertility Agents, 1978), is a yellow pigment localized mainly in cotton seed kernels. The crystallization work, aimed at obtaining well characterized crystal samples, disclosed that this natural product is a very flexible host, forming lattice inclusion complexes with most, polar and non-polar, organic solvents (Ibragimov, Talipov, Dadabaev, Nazarov & Aripov, 1989). These complexes exibit channel-, layer- and clathrate-type structure. It has been shown (Ibragimov, Gdaniec & Dadabaev, 1990) that in the so-called acetone-type complexes of gossypol, which crystallize in space group $P\bar{1}$ and are of the channel-forming type, the host structure can easily rearrange exposing different functional groups for hydrogen bonding. In consequence, the host is able to accommodate guests of proper size, shape and chemical nature in the channels. The lattice inclusion complexes of gossypol with branched and non-branched alcohols, with branched carboxylic acids and their halo derivatives and with aldehydes and ketones belong to this class. The crystal structures of several representatives of the class, *i.e.* the structures of the complexes with acetone (Talipov, Ibragimov, Tishchenko, Aripov, Nazarov, Strokopytov & Polyakov, 1988), cyclohexanone, tetrahydrofuran and 1-butanal (Ibragimov, Gdaniec & Dadabaev 1990) have already been published. Acetonitrile is the smallest guest forming an acetone-type complex and its crystal structure will be presented and compared to the other structures in this paper. The following abbreviations are used throughout the text: GPACT for gossypol:acetone, GPCHN for gossypol:cyclohexanone, GPTHF for gossypol:tetrahydrofuran and GPANR for gossypol:acetonitrile, GPBTA for gossypol:1-butanal.



Experimental. Yellow crystals were obtained from solution in acetonitrile/isooctane. The lattice parameters were based on 15 reflections ($16 \leq 2\theta \leq 30°$). Diffraction data were collected from a crystal of dimensions 0·1 × 0·2 × 0·3 mm in the θ–2θ mode to a maximum 2θ value of 120° on a Syntex $P2_1$ diffractometer using Cu $K\alpha$ radiation with a graphite monochromator; range of indices: h 0→10, k −11→11 and l −15→14. Two standard reflections were monitored every 100 intensity measurements, maximum variation was 4·5%. No correction for absorption or extinction was used. The final data set

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* 1,1′,6,6′,7,7′-Hexahydroxy-5,5′-diisopropyl-3,3′-dimethyl(2,2′-binaphthalene)-8,8′-dicarboxaldehyde.

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consisted of 4305 unique reflections, of which 2360 had $I > 1{\cdot}5\sigma(I)$ and were considered observed. The structure was solved routinely with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All atoms were found in the first *E* map. Full-matrix least-squares refinement of the scale factor and positional and anisotropic thermal parameters for non-H atoms was carried out using *SHELX*76 (Sheldrick, 1976). Weights were assigned as $w = 1/[\sigma^2(F) + 0{\cdot}0002F^2]$ and the quantity minimized was $\sum w(F_o - F_c)^2$. All of the H atoms attached to the O atoms and those of the methyl groups C(21) and C(26) were found in difference maps. The remaining H atoms were placed in idealized positions, assuming a C—H distance of 1·08 Å. The positional parameters of the H atoms located from ΔF maps and isotropic thermal parameters of all the H atoms were included in the refinement. The empirical isotropic extinction parameter x $[F'_c = F_c(1 - xF_c/\sin\theta)]$ converged at 108 (8) × 10^{-8}. Final residuals were $R = 0{\cdot}049$ and $wR = 0{\cdot}054$. The maximum Δ/σ value in the final cycle of refinement was 0·01, final $\Delta\rho$ max. and min. were $\pm 0{\cdot}21$ e Å^{-3}. Atomic scattering factors were those incorporated in *SHELX*76 (Sheldrick, 1976). An *ORTEP* (Johnson, 1976) representation of the complex with atomic labelling is shown in Fig. 1, and the packing is shown in Fig. 2. The atomic parameters are given in Table 1.* An XT IBM PC computer was used to carry out all crystallographic computations.

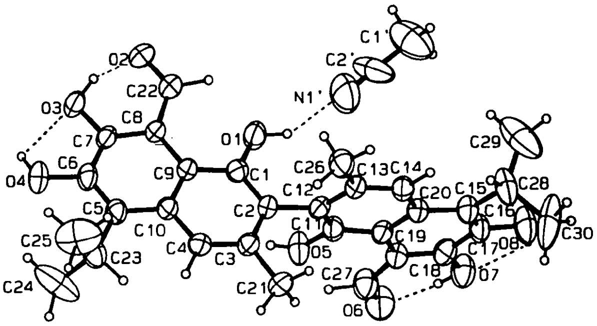

Fig. 1. The atom-numbering scheme of the gossypol and acetonitrile molecules.

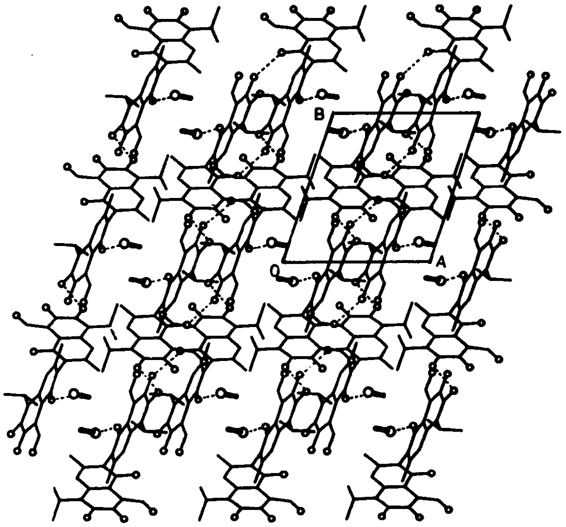

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) view of the crystal packing.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
C(1)	0·2846 (4)	0·8546 (3)	0·5006 (3)	0·045 (2)
C(2)	0·2848 (3)	0·7283 (3)	0·5056 (3)	0·042 (2)
C(3)	0·3157 (3)	0·6740 (3)	0·4323 (3)	0·045 (2)
C(4)	0·3528 (3)	0·7451 (3)	0·3621 (3)	0·047 (2)
C(5)	0·4067 (4)	0·9430 (3)	0·2877 (3)	0·049 (2)
C(6)	0·4127 (4)	1·0650 (3)	0·2902 (3)	0·048 (2)
C(7)	0·3689 (3)	1·1261 (3)	0·3552 (3)	0·044 (2)
C(8)	0·3179 (3)	1·0657 (3)	0·4218 (3)	0·043 (2)
C(9)	0·3183 (3)	0·9328 (3)	0·4269 (3)	0·041 (2)
C(10)	0·3587 (3)	0·8739 (3)	0·3582 (3)	0·044 (2)
C(21)	0·3139 (5)	0·5368 (4)	0·4331 (4)	0·059 (2)
C(22)	0·2628 (4)	1·1445 (4)	0·4749 (3)	0·068 (2)
C(23)	0·4514 (4)	0·8808 (4)	0·2139 (3)	0·063 (2)
C(24)	0·5979 (6)	0·8709 (9)	0·2303 (7)	0·137 (5)
C(25)	0·3439 (8)	0·9440 (7)	0·0980 (4)	0·128 (5)
O(1)	0·2583 (3)	0·9086 (3)	0·5728 (2)	0·069 (2)
O(2)	0·2674 (3)	1·2581 (2)	0·4703 (2)	0·076 (2)
O(3)	0·3755 (3)	1·2492 (2)	0·3477 (2)	0·059 (2)
O(4)	0·4568 (3)	1·1367 (3)	0·2250 (2)	0·064 (2)
C(11)	0·3596 (4)	0·5807 (3)	0·6904 (3)	0·047 (2)
C(12)	0·2539 (4)	0·6495 (3)	0·5882 (3)	0·044 (2)
C(13)	0·1175 (4)	0·6432 (3)	0·5633 (3)	0·047 (2)
C(14)	0·0956 (4)	0·5693 (3)	0·6430 (3)	0·051 (2)
C(15)	0·1739 (4)	0·4250 (4)	0·8301 (3)	0·055 (2)
C(16)	0·2797 (4)	0·3644 (4)	0·9314 (3)	0·057 (2)
C(17)	0·4181 (4)	0·3621 (4)	0·9568 (3)	0·054 (2)
C(18)	0·4539 (4)	0·4256 (3)	0·8806 (3)	0·049 (2)
C(19)	0·3420 (4)	0·5005 (3)	0·7740 (3)	0·045 (2)
C(20)	0·2037 (4)	0·4971 (3)	0·7484 (3)	0·047 (2)
C(26)	−0·0005 (5)	0·7181 (5)	0·4524 (4)	0·065 (3)
C(27)	0·6011 (4)	0·4074 (4)	0·9140 (4)	0·067 (2)
C(28)	0·0274 (4)	0·4196 (5)	0·8053 (4)	0·080 (3)
C(29)	−0·0435 (7)	0·5077 (9)	0·8504 (6)	0·151 (5)
C(30)	0·0270 (7)	0·2756 (8)	0·8396 (6)	0·143 (5)
O(5)	0·4923 (3)	0·5882 (3)	0·7166 (2)	0·067 (2)
O(6)	0·6935 (3)	0·3436 (3)	1·0078 (2)	0·080 (2)
O(7)	0·5117 (3)	0·2921 (3)	1·0598 (2)	0·071 (2)
O(8)	0·2549 (3)	0·3023 (3)	1·0140 (2)	0·081 (2)
C(1′)	0·0110 (6)	0·8912 (7)	0·8325 (4)	0·139 (4)
C(2′)	0·0728 (6)	0·8798 (5)	0·7596 (4)	0·110 (4)
N(1′)	0·1203 (6)	0·8690 (5)	0·7040 (4)	0·135 (4)

Discussion. Bond distances and angles of GPANR are given in Table 2. The bond distances and angles in the gossypol and acetonitrile molecules are similar to those found for these molecules in other structure determinations. The gossypol molecule can have aldehyde, lactol and quinoid form in solution (Kamaev, Baram, Ismailov, Leontev & Sadykov, 1979; Reyes, Wyrick, Borriero & Benas, 1986) but it

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52431 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)—C(2)	1·373 (5)	C(11)—C(12)	1·376 (4)
C(1)—C(9)	1·420 (6)	C(11)—C(19)	1·424 (6)
C(1)—O(1)	1·372 (6)	C(11)—O(5)	1·381 (5)
C(2)—C(3)	1·409 (6)	C(12)—C(13)	1·418 (6)
C(3)—C(4)	1·372 (6)	C(13)—C(14)	1·378 (6)
C(3)—C(21)	1·509 (6)	C(13)—C(26)	1·504 (5)
C(4)—C(10)	1·420 (5)	C(14)—C(20)	1·417 (4)
C(5)—C(6)	1·362 (6)	C(15)—C(16)	1·358 (4)
C(5)—C(10)	1·450 (6)	C(15)—C(20)	1·446 (6)
C(5)—C(23)	1·528 (7)	C(15)—C(28)	1·523 (7)
C(6)—C(7)	1·406 (6)	C(16)—C(17)	1·410 (6)
C(6)—O(4)	1·381 (5)	C(16)—O(8)	1·377 (6)
C(7)—C(8)	1·392 (6)	C(17)—C(18)	1·388 (7)
C(7)—O(3)	1·354 (5)	C(17)—O(7)	1·350 (4)
C(8)—C(9)	1·446 (5)	C(18)—C(19)	1·446 (4)
C(8)—C(22)	1·436 (6)	C(18)—C(27)	1·449 (6)
C(9)—C(10)	1·422 (6)	C(19)—C(20)	1·427 (6)
C(22)—O(2)	1·249 (5)	C(27)—O(6)	1·249 (4)
C(23)—C(24)	1·505 (9)	C(28)—C(29)	1·500 (11)
C(23)—C(25)	1·504 (6)	C(28)—C(30)	1·556 (10)
C(2)—C(12)	1·496 (6)	C(1′)—C(2′)	1·443 (10)
C(2′)—N(1′)	1·107 (10)		

C(9)—C(1)—O(1)	116·6 (3)	C(19)—C(11)—O(5)	116·2 (3)
C(2)—C(1)—O(1)	120·1 (3)	C(12)—C(11)—O(5)	119·7 (3)
C(2)—C(1)—C(9)	123·2 (3)	C(12)—C(11)—C(19)	124·1 (4)
C(1)—C(2)—C(12)	120·5 (3)	C(2)—C(12)—C(11)	119·6 (4)
C(1)—C(2)—C(3)	118·9 (3)	C(11)—C(12)—C(13)	119·0 (3)
C(3)—C(2)—C(12)	120·6 (3)	C(2)—C(12)—C(13)	121·4 (3)
C(2)—C(3)—C(21)	120·8 (4)	C(12)—C(13)—C(26)	120·2 (4)
C(2)—C(3)—C(4)	119·4 (3)	C(12)—C(13)—C(14)	118·6 (3)
C(4)—C(3)—C(21)	119·8 (4)	C(14)—C(13)—C(26)	121·2 (4)
C(3)—C(4)—C(10)	122·7 (3)	C(13)—C(14)—C(20)	123·1 (4)
C(10)—C(5)—C(23)	121·3 (3)	C(20)—C(15)—C(28)	121·4 (3)
C(6)—C(5)—C(23)	120·7 (4)	C(16)—C(15)—C(28)	120·3 (4)
C(6)—C(5)—C(10)	117·9 (3)	C(16)—C(15)—C(20)	118·2 (4)
C(5)—C(6)—O(4)	121·1 (3)	C(15)—C(16)—O(8)	120·6 (4)
C(5)—C(6)—C(7)	122·0 (4)	C(15)—C(16)—C(17)	122·1 (4)
C(7)—C(6)—O(4)	116·9 (3)	C(17)—C(16)—O(8)	117·3 (4)
C(6)—C(7)—O(3)	116·3 (3)	C(16)—C(17)—O(7)	114·5 (4)
C(6)—C(7)—C(8)	122·4 (3)	C(16)—C(17)—C(18)	122·1 (4)
C(8)—C(7)—O(3)	121·3 (3)	C(18)—C(17)—O(7)	123·4 (4)
C(7)—C(8)—C(22)	116·6 (3)	C(17)—C(18)—C(27)	117·0 (4)
C(7)—C(8)—C(9)	117·5 (3)	C(17)—C(18)—C(19)	117·4 (4)
C(9)—C(8)—C(22)	125·9 (4)	C(19)—C(18)—C(27)	125·6 (4)
C(1)—C(9)—C(8)	123·1 (3)	C(11)—C(19)—C(18)	124·1 (4)
C(8)—C(9)—C(10)	119·4 (3)	C(18)—C(19)—C(20)	119·7 (3)
C(1)—C(9)—C(10)	117·5 (3)	C(11)—C(19)—C(20)	116·2 (3)
C(5)—C(10)—C(9)	120·7 (3)	C(15)—C(20)—C(19)	120·2 (3)
C(4)—C(10)—C(9)	118·1 (3)	C(14)—C(20)—C(19)	119·0 (3)
C(4)—C(10)—C(5)	121·3 (3)	C(14)—C(20)—C(15)	120·8 (4)
C(8)—C(22)—O(2)	123·8 (4)	C(18)—C(27)—O(6)	123·0 (4)
C(5)—C(23)—C(25)	111·5 (4)	C(15)—C(28)—C(30)	112·2 (5)
C(5)—C(23)—C(24)	114·3 (5)	C(15)—C(28)—C(29)	111·4 (5)
C(24)—C(23)—C(25)	113·3 (5)	C(29)—C(28)—C(30)	113·1 (5)
		C(1′)—C(2′)—N(1′)	178·9 (7)

exists only in the aldehyde form in the crystalline state. The gossypol sample used for our studies was racemic; however, owing to the restricted rotation around the C(2)—C(12) bond, optically active gossypol can also be isolated (King & de Silva, 1968; Matlin, Belenguer, Tyson & Brookes, 1987). The steric hindrance between the substituents *ortho* to the C(2)—C(12) bond give rise to the nearly perpendicular orientation of the naphthyl moieties. The dihedral angle between the naphthyl least-squares planes is 86·4 (1)° in the acteonitrile complex, in the range 83–87° found in other acetone-type complexes. The two aromatic rings are not planar, the χ^2 values being 1547 and 819 for C(1)–C(10) and C(11)–C(20) rings, respectively.

In the gossypol molecule there are six H atoms able to participate in the hydrogen bonding. Those at O(3) and O(7) take part in strong intramolecular hydrogen bonds which form six-membered rings. The hydroxyl groups O(4)—H(4O) and O(8)—H(8O) are involved, in most cases, in three-centre hydrogen bonds, of which the intramolecular hydrogen bonds O(4)—H(4O)···O(3) and O(8)—H(8O)···O(7), closing five-membered rings, are one component. The hydroxyl groups O(1)—H(1O) and O(5)—H(5O) participate in intermolecular hydrogen bonds. The geometry of the hydrogen bonds in GPANR is given in Table 3.

Table 3. *Geometry of hydrogen bonds*

D—H···*A*	*D*···*A* (Å)	*D*—H (Å)	H···*A* (Å)	*D*—H···*A* (°)
(*a*) Intramolecular hydrogen bonds				
O(3)—H(3O)···O(2)	2·449 (5)	1·04 (5)	1·52 (5)	146 (3)
O(7)—H(7O)···O(6)	2·500 (5)	0·91 (5)	1·67 (5)	148 (4)
O(4)—H(4O)···O(3)	2·630 (5)	1·06 (7)	2·15 (7)	105 (4)
O(8)—H(8O)···O(7)	2·603 (5)	0·92 (4)	2·08 (5)	115 (3)
(*b*) Intermolecular hydrogen bonds				
O(5)—H(5O)···O(3^{i})	2·751 (5)	0·86 (5)	2·11 (5)	131 (4)
O(4)—H(4O)···O(5^{i})	3·400 (5)	1·06 (7)	2·36 (7)	165 (5)
O(8)—H(8O)···O(4^{ii})	2·841 (3)	0·92 (4)	1·99 (4)	153 (5)
O(1)—H(1O)···N(1′)	2·828 (8)	0·91 (5)	2·08 (6)	139 (4)

Symmetry code: (i) $1-x, 2-y, 1-z$; (ii) $x, -1+y, 1+z$.

The host molecules form centrosymmetric dimers *via* O(5)—H(5O)···O(3) hydrogen bonds. Such dimers are often present in the gossypol inclusion complexes and are also observed within the acetone class with the exception of cases where the acyclic guest molecule has a carbon chain longer than three atoms, in which case centrosymmetric dimers of a new type are formed (Ibragimov, Gdaniec & Dadabaev, 1990). The geometry of the O(5)—H(5O)···O(3) hydrogen bond changes only slightly for the structures studied up to now, the O(5)···O(3) distance being in the range 2·75–2·80 Å (Table 4). The naphthyl units C(1)–C(10) of the dimer are parallel, at a distance of 3·30 Å, and overlap significantly. The stacking interaction, in addition to the O(5)—H(5O)···O(3) hydrogen bond, is probably responsible for the stability of the gossypol dimer.

The O(6)–O(8) region of the gossypol dimer interacts with the O(2)–O(4) at x, $y-1$, $z+1$, and columns of host molecules are formed. There are two short O···O contacts between those regions; however, only one of them, O(8)···O(4), is a hydrogen bond (Table 4). The donor–acceptor distance of the O(8)—H(8O)···O(4) hydrogen bond changes significantly with the guest size. In the complex with the smallest guest, GPANR, this distance is the shortest [2·841 (3) Å], while in GPCHN, with the largest guest molecule, this bond is broken [O(8)···O(4) 3·747 (7) Å]. Relative to the dimer positions in GPANR, the gossypol dimers in GPCHN are shifted *ca* 1·7 Å along the line C(8), O(6), enlarging the space 'over' the naphthyl moiety for a larger guest and increasing the O(8)···O(4) intermolecular distance. The other short contact between the O(6)–

Table 4. *Comparison of some* O···O *and* O···H *intermolecular contacts in acetone-type complexes of gossypol*

	GPANR	GPACT	GPTHF	GPCHN	GPBTA
O(8)—H(8O)···O(4)	2·841 (3)	2·949 (6)	3·296 (4)	3·747 (3)	3·059 (5)
H(8O)···O(4)	1·99 (4)	1·95 (6)	2·45 (4)	2·75	2·44 (7)
O(4)—H(4O)···O(7)	2·931 (5)	2·868 (7)	2·785 (4)	2·978 (8)	2·853 (5)
H(4O)···O(7)	2·89 (7)	2·83 (7)	2·77 (5)	3·00	2·34 (7)
O(5)—H(5O)···O(3)	2·751 (5)	2·777 (6)	2·762 (4)	2·795 (7)	2·948 (5)
H(5O)···O(3)	2·11 (5)	2·16 (6)	2·19 (4)	1·96	2·91 (6)

O(8) and O(2)—O(4) regions of the dimers is between O(4) and O(7) (Table 4). This contact is not a hydrogen bond due to the unfavourable orientation of the H(4O) proton involved in the intramolecular O(4)—H(4O)···O(3) hydrogen bond.

There are only van der Waals interactions between the gossypol columns constituting the host lattice. Channels parallel to [001] can be recognized. The channel is hydrophilic with the hydroxyl group O(1)—H and aldehyde oxygen O(2) located on its surface. The guests having hydrogen-bond-acceptor groups as well as those having both acceptor and donor groups could be accommodated in these channels. In GPANR, GPACT, GPTHF, GPCHN and GPBTA the guests have only hydrogen-bond-acceptor groups and a hydrogen bond between O(1)—H and the guest is formed. The guest molecule occupies the space 'over' the C(11)—C(20) naphthyl ring. In GPANR the 'best' line through acetonitrile C(1′), C(2′) and N(1′) atoms forms an angle of 52·3 (2)° with the normal to the C(11)—C(20) ring. As mentioned before, the amount of space over the ring depends on guest size and can be increased by a mutual shift of the gossypol dimers, decreasing the O(8)—H(8O)···O(4) hydrogen-bond strength.

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References

Ibragimov, B. T., Gdaniec, M. & Dadabaev, B. N. (1990). *J. Inclusion Phenom.* In the press.

Ibragimov, B. T., Talipov, S. A., Dadabaev, B. N., Nazarov, G. B. & Aripov, T. F. (1988). *Khim. Prir. Soedin.* pp. 675–679.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kamaev, F. G., Baram, N. I., Ismailov, A. I., Leontev, V. B. & Sadykov, S. A. (1979). *Izv. Akad. Nauk SSSR Ser. Khim.* pp. 1003–1010.

King, T. J. & de Silva, L. B. (1968). *Tetrahedron Lett.* pp. 261–263.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Matlin, S. A., Belenguer, A., Tyson, R. G. & Brookes, A. N. (1987). *J. High Resolut. Chromatogr. Chromatogr. Commun.* **10**, 86–91.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

National Coordinating Group on Male Antifertility Agents (1978). *Chin. Med. J.* **4**, 417–428.

Reyes, J., Wyrick, S. D., Borierro, L. & Benas, D. J. (1986). *Biochem. Biophys. Acta*, **863**, 101–109.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Talipov, S. A., Ibragimov, B. T., Tishchenko, G. N., Aripov, T. F., Nazarov, G. B., Strokopytov, B. V. & Polyakov, K. M. (1988). *Kristallografiya*, **33**, 384–389.

Vermel, E. M. & Krouglyak, S. A. (1963). *Vopr. Onkol.* **12**, 39–43.

Vichkanova, S. A. & Goryunova, L. V. (1968). *Antibiotiki (Moscow)*, **9**, 828–832.

Acta Cryst. (1990). C**46**, 813–816

Solid-State Reactivity of Benzoxazinones. X-ray Structure of 2-Acetamido-5-chlorobenzoic Acid

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Abstract. $C_9H_8ClNO_3$, $M_r = 216·6$, triclinic, $P\bar{1}$, $a = 7·414\ (2)$, $b = 9·059\ (1)$, $c = 15·839\ (3)$ Å, $\alpha = 73·600\ (14)$, $\beta = 77·421\ (19)$, $\gamma = 67·786\ (18)°$, $V = 936·7$ Å^3, $Z = 4$, $D_x = 1·53$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·7109$ Å, $\mu = 1·9$ cm^{-1}, $F(000) = 792$, $T = 293$ K. The structure was refined from 3198 reflections with

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