geometry of these 'strong' hydrogen bonds are: $N(1)^+$—H···O(6), N(1)—H = 0·93 (3), N(1)···O(6) = 2·666 (3), H···O(6) = 1·74 (3) Å, N(1)—H···O(6) = 173 (2)°; O(2)—H···O(5), O(2)—H = 0·94 (3), O(2)···O(5) = 2·530 (3), H···O(5) = 1·60 (3) Å, O(2)—H···O(5) = 174 (3)°. Thus, such an association leads to the formation of centrosymmetric groups containing two cations of opposite chirality and one dianion. The second N^+—H bond is involved in a 'normal' N(1)—H···O(2) $(1 - x, 1 - y, 1 - z)$ hydrogen bond [N(1)—H = 0·89 (2), N(1)···O(2) = 2·798 (2), H···O(2) = 1·92 (2) Å, N(1)—H···O(2) = 167 (2)°] which connects two adjacent groups related by an inversion centre to form larger groups of six ions. The latter groups are held together by van der Waals forces only. Thus, although the packing is controlled by hydrogen bonding, it totally ignores the hydrogen-acceptor capability of the acetyl O(4) atom.

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Lattice Inclusion Compounds of Gossypol. Structure of the 2:3 Gossypol–Benzaldehyde Coordinatoclathrate

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Abstract. $C_{30}H_{30}O_8 \cdot 1 \cdot 5C_7H_6O$, $M_r = 677 \cdot 66$, triclinic, $P\bar{1}$, $a = 10 \cdot 959$ (2), $b = 14 \cdot 116$ (2), $c = 11 \cdot 418$ (2) Å, $\alpha = 73 \cdot 62$ (1), $\beta = 92 \cdot 27$ (1), $\gamma = 91 \cdot 71$ (1)°, $V = 1693 \cdot 0$ (5) Å^3, $Z = 2$, $D_x = 1 \cdot 33$ g cm^{-3}, λ(Cu $K\alpha$) = $1 \cdot 54184$ Å, μ(Cu $K\alpha$) = $7 \cdot 35$ cm^{-1}, $F(000) = 716$, $T = 293$ K, $R = 0 \cdot 054$ for 2499 observed reflections. The structure of the first gossypol lattice inclusion compound with host:guest molecular ratio of 2:3 is presented. The host molecules form centrosymmetric dimers, typical for gossypol, via a pair of O(5)—H···O(3) hydrogen bonds. The guest molecules are enclosed in two different types of centrosymmetric cages. In cage $T1$ the guest molecule is statistically disordered and can adopt four different orientations in which it is hydrogen bonded to the O(8)—H hydroxyl of gossypol. Cage $T2$ accommodates two benzaldehyde molecules related by a symmetry center. These guest molecules are hydrogen bonded to the gossypol O(1)—H hydroxyl groups.

Introduction. Gossypol [1,1',6,6',7,7'-hexahydroxy-5,5'-diisopropyl-3,3'-dimethyl(2,2'-binaphthalene)-8,8'-dicarboxaldehyde], a physiologically active disesquiterpene localized mainly in cotton seed kernels, shows remarkable inclusion ability towards a number of chemically different guest substrates.

Topological as well as coordinative interactions play an important role in the formation of its lattice inclusion compounds. The host structure is easily rearranged to accommodate guests of a proper shape, size and chemical nature. Gossypol crystallizes in triclinic, monoclinic and orthorhombic systems (Ibragimov, Talipov, Dadabaev, Nazarov & Aripov, 1988). There are several types of triclinic host lattices with host:guest stoichiometry of 1:1 and 2:1 for which crystal structures are known (Changfu, Cunheng, Guanghong & Shantian, 1982; Gdaniec, Ibragimov & Talipov, 1990; Ibragimov, Gdaniec & Dadabaev, 1990; Gdaniec, Ibragimov & Dadabaev, 1990; Ibragimov, Talipov, Aripov & Sadykov, 1990). The present gossypol–benzaldehyde inclusion compound is the only known example with 2:3 host:guest molecular ratio.

Experimental. Yellow crystals were obtained from a gossypol solution in benzaldehyde/benzene. The lattice parameters are based on 15 centered reflections with 2θ values between 16 and 28°. Diffraction data were collected from a crystal of dimensions $0.2 \times 0.3 \times 0.3$ 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 $0\rightarrow12$, $k - 14\rightarrow15$ and $l - 11\rightarrow12$. Two standard reflections were monitored every 100 intensity measurements. The standard intensities varied by up to 4.2%. No correction for absorption or extinction was used. The final data set consisted of 5016 unique reflections, of which 2499 had $I \geq 1.5\sigma(I)$ and were considered observed. The structure was solved with the program $MULTAN80$ (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All atoms of gossypol and benzaldehyde molecule B were found in the best E map. The second guest molecule, A, which is required by symmetry to be disordered, was found from a ΔF map. The center of symmetry is located in the plane of the phenyl ring. Constraints were imposed on interatomic distances of this benzaldehyde molecule by adding extra observational equations to the least-squares matrix with weights proportional to $1/\sigma^2$ ($\sigma = 0.005$ Å). Full-matrix least-squares refinement of positional and anisotropic thermal parameters of gossypol and benzaldehyde B non-hydrogen atoms and of positional and isotropic temperature factors of benzaldehyde A non-hydrogen atoms was carried out using $SHELX76$ (Sheldrick, 1976). A difference Fourier synthesis showed high peaks (0.89 e Å^{-3}) around the disordered guest molecule, indicating that benzaldehyde A can adopt another orientation (A') in which it is also hydrogen bonded to the host. Constraints were imposed on the interatomic distances of the benzaldehyde molecule in this orientation and the guest molecules in orientation A and A' were

given an occupancy factor of 0.25 each. The hydrogen atoms attached to the oxygen atoms and those of methyl groups C(21) and C(26) were located in a ΔF map. The remaining hydrogen atoms of the gossypol and benzaldehyde B molecule were placed in idealized positions assuming C—H distances of 1.08 Å. Positional parameters of the hydroxyl hydrogen atoms and isotropic temperature factors of all gossypol hydrogen atoms were allowed to vary during the refinement. In the final refinement cycles the benzaldehyde molecules in orientations A and A' were refined as rigid groups with individual temperature factors for non-hydrogen atoms. The refinement converged to give final residuals $R = 0.054$ and $wR = 0.057$. Weights were assigned as $w = 1/[\sigma^2(F) + 0.0002F^2]$ and the quantity minimized was $\Sigma w(F_o - F_c)^2$. An empirical isotropic extinction parameter x was used to correct F_c according to $F_c' = F_c(1 - xF_c^2/\sin\theta)$; x converged at 103 (7) $\times$ 10^{-8}. The maximum Δ/σ value in the final cycle of refinement was 0.3; extreme values of the residual electron density in the final ΔF map were -0.21 and 0.38 e Å^{-3} (near the disordered guest molecule). Atomic scattering factors used were those incorporated in $SHELX76$ (Sheldrick, 1976). An $ORTEP$ (Johnson, 1976) representation of the complex with atomic labeling is shown in Fig. 1. The atomic parameters are given in Table 1.* An XT IBM PC computer was used to carry out all crystallographic calculations.

Discussion. Bond distances and angles of the gossypol molecule given in Table 2 are in good agreement with other structure determinations (Changfu et al., 1982; Gdaniec, Ibragimov & Dadabaev, 1990; Gdaniec, Ibragimov & Talipov, 1990; Ibragimov, Gdaniec & Dadabaev, 1990; Ibragimov, Talipov et al., 1990). The gossypol molecule is in the aldehyde

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* Lists of structure factors, H-atom parameters, anisotropic thermal parameters and results of least-squares planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53213 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
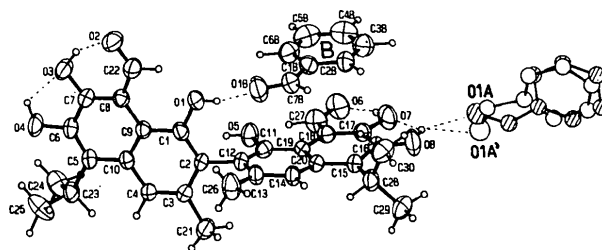

Fig. 1. Atom numbering scheme of gossypol and benzaldehyde molecule B.

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

$$U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
C(1)	0·9963 (4)	0·1235 (4)	0·2196 (4)	0·047 (2)
C(2)	1·0039 (4)	0·2247 (3)	0·1646 (4)	0·043 (2)
C(3)	0·9260 (4)	0·2874 (3)	0·2026 (4)	0·047 (2)
C(4)	0·8501 (4)	0·2482 (3)	0·2980 (4)	0·045 (2)
C(5)	0·7647 (4)	0·1069 (4)	0·4609 (4)	0·047 (2)
C(6)	0·7636 (4)	0·0077 (4)	0·5155 (4)	0·049 (2)
C(7)	0·8320 (4)	−0·0575 (3)	0·4718 (4)	0·047 (2)
C(8)	0·9034 (4)	−0·0248 (3)	0·3704 (4)	0·044 (2)
C(9)	0·9146 (4)	0·0799 (3)	0·3154 (4)	0·042 (2)
C(10)	0·8426 (4)	0·1450 (3)	0·3578 (4)	0·042 (2)
C(11)	1·2109 (4)	0·2887 (3)	0·0998 (4)	0·045 (2)
C(12)	1·0946 (4)	0·2655 (3)	0·0673 (4)	0·044 (2)
C(13)	1·0627 (4)	0·2833 (3)	−0·0579 (4)	0·049 (2)
C(14)	1·1474 (4)	0·3254 (3)	−0·1431 (4)	0·049 (2)
C(15)	1·3537 (4)	0·3920 (3)	−0·2046 (4)	0·047 (2)
C(16)	1·4717 (4)	0·4018 (3)	−0·1690 (4)	0·056 (2)
C(17)	1·5087 (4)	0·3826 (3)	−0·0446 (5)	0·053 (2)
C(18)	1·4267 (4)	0·3509 (3)	0·0495 (4)	0·046 (2)
C(19)	1·3022 (4)	0·3309 (3)	0·0151 (4)	0·041 (2)
C(20)	1·2660 (4)	0·3515 (3)	−0·1111 (4)	0·042 (2)
C(21)	0·9252 (5)	0·3971 (3)	0·1388 (6)	0·070 (3)
C(22)	0·9518 (5)	−0·0996 (4)	0·3227 (5)	0·067 (2)
C(23)	0·6846 (4)	0·1751 (4)	0·5065 (4)	0·057 (2)
C(24)	0·7197 (5)	0·1754 (5)	0·6374 (5)	0·081 (3)
C(25)	0·5476 (5)	0·1509 (6)	0·4940 (7)	0·092 (3)
C(26)	0·9370 (5)	0·2527 (6)	−0·0956 (5)	0·076 (3)
C(27)	1·4738 (5)	0·3413 (4)	0·1714 (5)	0·070 (3)
C(28)	1·3171 (4)	0·4199 (3)	−0·3394 (4)	0·057 (2)
C(29)	1·3577 (5)	0·5250 (4)	−0·4090 (5)	0·072 (3)
C(30)	1·3607 (6)	0·3434 (4)	−0·4005 (5)	0·077 (3)
O(1)	1·0716 (3)	0·0598 (3)	0·1878 (3)	0·068 (2)
O(2)	0·9406 (3)	−0·1905 (2)	0·3742 (3)	0·082 (2)
O(3)	0·8173 (3)	−0·1545 (2)	0·5320 (3)	0·063 (2)
O(4)	0·6928 (3)	−0·0330 (3)	0·6149 (3)	0·064 (2)
O(5)	1·2448 (3)	0·2675 (2)	0·2231 (3)	0·063 (2)
O(6)	1·5845 (3)	0·3532 (3)	0·1955 (3)	0·082 (2)
O(7)	1·6285 (3)	0·3994 (3)	−0·0240 (4)	0·073 (2)
O(8)	1·5627 (3)	0·4319 (3)	−0·2520 (3)	0·083 (2)
O(1B)	1·1940 (3)	0·0746 (3)	−0·0244 (3)	0·087 (3)
C(1B)	1·4084 (5)	0·1115 (3)	−0·0169 (5)	0·053 (3)
C(2B)	1·5061 (5)	0·1472 (3)	−0·0901 (4)	0·058 (2)
C(3B)	1·6191 (5)	0·1582 (4)	−0·0384 (6)	0·073 (3)
C(4B)	1·6347 (6)	0·1319 (4)	0·0869 (6)	0·083 (3)
C(5B)	1·5353 (7)	0·0959 (4)	0·1588 (5)	0·082 (3)
C(6B)	1·4218 (6)	0·0847 (4)	0·1082 (5)	0·067 (3)
C(7B)	1·2889 (5)	0·1010 (4)	−0·0773 (5)	0·068 (3)
C(1A)	1·0730 (10)	0·5254 (8)	0·4210 (10)	0·095 (9)*
C(2A)	1·0130 (10)	0·4350 (8)	0·4650 (10)	0·071 (8)*
C(3A)	0·9300 (10)	0·4228 (8)	0·5570 (10)	0·054 (7)*
C(4A)	0·9130 (10)	0·4996 (8)	0·6100 (10)	0·060 (6)*
C(5A)	0·9730 (10)	0·5888 (8)	0·5660 (10)	0·08 (4)*
C(6A)	1·0600 (10)	0·5995 (8)	0·4770 (10)	0·09 (1)*
C(7A)	1·1650 (10)	0·5409 (8)	0·3260 (10)	0·12 (1)*
O(1A)	1·1920 (10)	0·4792 (8)	0·2760 (10)	0·17 (1)*
C(1A')	1·0410 (10)	0·4860 (8)	0·4420 (10)	0·072 (7)*
C(2A')	1·0260 (10)	0·5826 (8)	0·4510 (10)	0·08 (1)*
C(3A')	0·9610 (10)	0·5925 (8)	0·5500 (10)	0·10 (4)*
C(4A')	0·8830 (10)	0·5161 (8)	0·6110 (10)	0·13 (2)*
C(5A')	0·9020 (10)	0·4196 (8)	0·6040 (10)	0·094 (8)*
C(6A')	0·9660 (10)	0·4095 (8)	0·5040 (10)	0·051 (7)*
C(7A')	1·1170 (10)	0·4657 (8)	0·3460 (10)	0·064 (6)*
O(1A')	1·1830 (10)	0·5288 (8)	0·2790 (10)	0·118 (7)*

* Isotropic temperature factor.

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

Gossypol

C(1)—C(2)	1·390 (6)	C(11)—C(12)	1·370 (6)
C(1)—C(9)	1·429 (6)	C(11)—C(19)	1·417 (6)
C(1)—O(1)	1·369 (6)	C(11)—O(5)	1·392 (6)
C(2)—C(3)	1·411 (7)	C(12)—C(13)	1·411 (6)
C(2)—C(12)	1·499 (6)	C(13)—C(14)	1·369 (6)
C(3)—C(4)	1·374 (6)	C(13)—C(26)	1·514 (7)
C(3)—C(21)	1·514 (6)	C(14)—C(20)	1·403 (6)
C(4)—C(10)	1·425 (6)	C(15)—C(16)	1·355 (6)
C(5)—C(6)	1·363 (7)	C(15)—C(20)	1·447 (6)
C(5)—C(10)	1·449 (6)	C(15)—C(28)	1·518 (6)
C(5)—C(23)	1·529 (8)	C(16)—C(17)	1·414 (7)
C(6)—C(7)	1·410 (7)	C(16)—O(8)	1·377 (6)
C(6)—O(4)	1·377 (5)	C(17)—C(18)	1·394 (7)
C(7)—C(8)	1·384 (6)	C(17)—O(7)	1·350 (6)
C(7)—O(3)	1·356 (5)	C(18)—C(19)	1·447 (6)
C(8)—C(9)	1·437 (6)	C(18)—C(27)	1·436 (7)
C(8)—C(22)	1·440 (8)	C(19)—C(20)	1·430 (6)
C(9)—C(10)	1·423 (7)	C(27)—O(6)	1·250 (6)
C(22)—O(2)	1·256 (6)	C(28)—C(29)	1·534 (6)
C(23)—C(24)	1·530 (8)	C(28)—C(30)	1·537 (8)
C(23)—C(25)	1·540 (7)		
C(9)—C(1)—O(1)	115·9 (4)	C(19)—C(11)—O(5)	116·8 (4)
C(2)—C(1)—O(1)	121·2 (4)	C(12)—C(11)—O(5)	119·1 (4)
C(2)—C(1)—C(9)	122·8 (5)	C(12)—C(11)—C(19)	124·0 (4)
C(1)—C(2)—C(12)	119·9 (4)	C(2)—C(12)—C(11)	119·5 (4)
C(1)—C(2)—C(3)	119·0 (4)	C(11)—C(12)—C(13)	118·7 (4)
C(3)—C(2)—C(12)	121·1 (4)	C(2)—C(12)—C(13)	121·8 (4)
C(2)—C(3)—C(21)	120·5 (4)	C(12)—C(13)—C(26)	119·6 (4)
C(2)—C(3)—C(4)	119·5 (4)	C(12)—C(13)—C(14)	119·3 (4)
C(4)—C(3)—C(21)	120·0 (4)	C(14)—C(13)—C(26)	121·1 (4)
C(3)—C(4)—C(10)	122·5 (4)	C(13)—C(14)—C(20)	122·6 (4)
C(10)—C(5)—C(23)	121·4 (4)	C(20)—C(15)—C(28)	121·7 (4)
C(6)—C(5)—C(23)	120·7 (4)	C(16)—C(15)—C(28)	120·2 (4)
C(6)—C(5)—C(10)	117·9 (5)	C(16)—C(15)—C(20)	118·2 (4)
C(5)—C(6)—O(4)	120·8 (5)	C(15)—C(16)—O(8)	121·9 (4)
C(5)—C(6)—C(7)	121·9 (4)	C(15)—C(16)—C(17)	122·1 (4)
C(7)—C(6)—O(4)	117·3 (5)	C(17)—C(16)—O(8)	116·0 (4)
C(6)—C(7)—O(3)	115·2 (4)	C(16)—C(17)—O(7)	115·1 (5)
C(6)—C(7)—C(8)	122·1 (4)	C(16)—C(17)—C(18)	122·3 (4)
C(8)—C(7)—O(3)	122·6 (4)	C(18)—C(17)—O(7)	122·6 (4)
C(7)—C(8)—C(22)	116·6 (4)	C(17)—C(18)—C(27)	116·9 (4)
C(7)—C(8)—C(9)	117·9 (4)	C(17)—C(18)—C(19)	116·9 (4)
C(9)—C(8)—C(22)	125·3 (4)	C(19)—C(18)—C(27)	126·2 (4)
C(1)—C(9)—C(8)	123·3 (4)	C(11)—C(19)—C(18)	124·0 (4)
C(8)—C(9)—C(10)	119·5 (4)	C(18)—C(19)—C(20)	119·9 (4)
C(1)—C(9)—C(10)	117·3 (4)	C(11)—C(19)—C(20)	116·0 (4)
C(5)—C(10)—C(9)	120·4 (4)	C(15)—C(20)—C(19)	120·2 (4)
C(4)—C(10)—C(9)	118·7 (4)	C(14)—C(20)—C(19)	119·3 (4)
C(4)—C(10)—C(5)	120·9 (4)	C(14)—C(20)—C(15)	120·4 (4)
C(8)—C(22)—O(2)	123·3 (5)	C(18)—C(27)—O(6)	122·8 (5)
C(5)—C(23)—C(25)	112·1 (5)	C(15)—C(28)—C(30)	111·3 (4)
C(5)—C(23)—C(24)	112·2 (4)	C(15)—C(28)—C(29)	113·4 (4)
C(24)—C(23)—C(25)	110·9 (4)	C(29)—C(28)—C(30)	111·5 (4)

Benzaldehyde

	Mol. B	Mol. A	Mol. A'
O(1)—C(7)	1·219 (6)	1·217 (18)	1·234 (14)
C(1)—C(2)	1·378 (7)	1·388 (15)	1·411 (17)
C(1)—C(6)	1·374 (8)	1·384 (18)	1·380 (14)
C(1)—C(7)	1·479 (8)	1·477 (16)	1·497 (17)
C(2)—C(3)	1·374 (8)	1·387 (16)	1·401 (17)
C(3)—C(4)	1·378 (9)	1·403 (18)	1·389 (15)
C(5)—C(4)	1·386 (9)	1·375 (15)	1·407 (17)
C(5)—C(6)	1·374 (10)	1·395 (16)	1·406 (17)
C(6)—C(1)—C(7)	120·9 (5)	117·7 (10)	116·3 (10)
C(2)—C(1)—C(7)	117·9 (5)	121·5 (10)	121·9 (10)
C(2)—C(1)—C(6)	121·2 (5)	120·2 (10)	120·5 (10)
C(1)—C(2)—C(3)	120·1 (5)	119·6 (11)	117·2 (10)
C(2)—C(3)—C(4)	119·7 (5)	119·8 (10)	119·7 (11)
C(4)—C(5)—C(6)	121·6 (6)	120·1 (10)	119·1 (10)
C(3)—C(4)—C(5)	119·2 (6)	119·8 (11)	117·5 (10)
C(1)—C(6)—C(5)	118·1 (5)	119·8 (11)	119·3 (11)
O(1)—C(7)—C(1)	124·9 (5)	124·5 (11)	123·3 (11)

form (Kamaev, Baram, Ismailov, Leontev & Sadykov, 1979; Reyes, Wyrick, Borriero & Benas, 1986). The two naphthyl moieties are nearly perpendicular, the dihedral angle between their least-squares planes being 86·1 (3)°. In consequence, the oxygen atoms of the hydroxyl groups O(1)—H and O(5)—H are 3·555 (5) Å apart. The naphthyl rings are not planar, the χ^2 values being 924 and 1372 for the C(1)–C(10) and C(11)–C(20) rings, respectively. The isopropyl

substituents at C(5) and C(15) have their methine hydrogens directed towards C(4) and C(14), respectively.

In the gossypol molecule there are six potential hydrogen-bond donors. H(3O) and H(7O) take part

in strong intramolecular hydrogen bonds which close six-membered conjugated ring systems. The hydroxyl groups O(4)—H(4O) and O(8)—H(8O) are involved in three-center hydrogen bonds with O(4)—H(4O)-···O(3) and O(8)—H(8O)···O(7) as intramolecular components closing five-membered rings. The hydroxyl O(4)—H(4O) is additionally involved in a weak interaction with O(5). Due to the disorder of the guest molecule, only every second hydroxyl O(8)—H(8O) is involved in an intermolecular hydrogen bond with the benzaldehyde A or A' oxygen atom. The carbonyl group of the benzaldehyde B molecule is involved in a hydrogen bond with the O(1)—H(1O) hydroxyl of gossypol. This hydroxyl group acts as a 'sensor group' in a prevailing number of gossypol inclusion compounds donating a hydrogen bond to an acceptor group of the guest molecule. The benzaldehyde B molecule and the C(11)–C(20) moiety of gossypol are nearly parallel [dihedral angle 2·5 (2)°] and show significant overlap (plane-to-plane distance 3·40 Å). The hydroxyl group O(5)—H(5O) acts as a hydrogen-bond donor to O(3) of the molecule related through the inversion center forming typical centrosymmetric gossypol dimers. The dimer is further stabilized by a weak O(4)—H(4O)···O(5) interaction. The two C(1)–C(10) moieties of the dimer overlap significantly and are at a distance of 3·51 Å. The dimers are the only hydrogen-bond associates of the host molecules in the structure. The geometry of the hydrogen bonds is given in Table 3.

A stereoview of the crystal packing of this lattice inclusion compound is shown in Fig. 2. The guest molecules are enclosed in two types of centrosymmetric cages. The parallelepipedal cage $T2$ located around the inversion center at $\frac{1}{2},0,0$ accommodates two guest molecules. These molecules are parallel to the C(1)–C(10) and perpendicular to the C(11)–C(20) moieties of the host which form lipophilic walls of the cage. The remaining walls of the cage $T2$ have on their surface hydroxyl groups O(1)—H(1O) which are hydrogen bonded to the aldehyde group of the

Table 3. *Geometry* (Å *and* °) *of hydrogen bonds*

D—H···A	D···A	D—H	H···A	D—H···A
Intramolecular				
O(3)—H(3O)···O(2)	2·466 (5)	1·03 (6)	1·57 (6)	143 (5)
O(7)—H(7O)···O(6)	2·469 (6)	0·87 (7)	1·67 (7)	151 (7)
O(4)—H(4O)···O(3)	2·619 (5)	0·89 (7)	2·12 (8)	115 (6)
O(8)—H(8O)···O(7)	2·589 (6)	0·98 (6)	1·95 (6)	121 (4)
Intermolecular				
O(5)—H(5O)···O(3^i)	2·898 (5)	0·88 (5)	2·20 (5)	136 (4)
O(4)—H(4O)···O(5^i)	3·381 (5)	0·89 (7)	2·52 (6)	163 (6)
O(1)—H(1O)···O(1B)	2·769 (5)	0·83 (6)	1·97 (6)	161 (8)
O(8)—H(8O)···O(1A^{ii})	2·922 (3)	0·98 (6)	2·27 (6)	122 (4)
O(8)—H(8O)···O(1A'^{ii})	2·837 (11)	0·98 (6)	2·17 (6)	122 (4)

Symmetry code: (i) $2 - x, -y, 1 - z$; (ii) $3 - x, 1 - y, -z$.

guest molecules B. Another centrosymmetric cage, $T1$, located around the center of symmetry at $0,\frac{1}{2},\frac{1}{2}$ is capable of accommodating only one guest molecule. Therefore, the benzaldehyde molecule, which has no center of symmetry, has to be disordered in this cage. As the ΔF map has shown (see *Experimental*), the benzaldehyde molecule in cage $T2$ adopts two orientations, A and A', not related through the center of symmetry, in which it is hydrogen bonded to O(8)—H(8O). In consequence, four different coplanar orientations of this guest are observed in cage $T1$.

According to the nomenclature proposed by Weber & Josel (1983) the gossypol–benzaldehyde lattice inclusion compound can be classified as coordinatoclathrate because topological as well as hydrogen-bond interactions play a role in its formation. There have been no other guest molecules identified so far which would give a lattice inclusion compound isostructural with gossypol–benzaldehyde, however some benzene derivatives with polar substituents would fit well in the cages of the host lattice presented here.
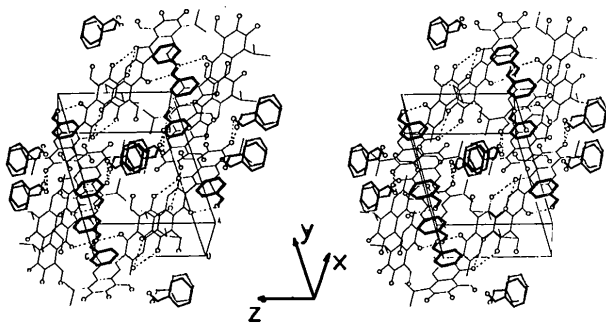
Fig. 2. Stereoview of the crystal packing (*PLUTO*; Motherwell & Clegg, 1978). The oxygen atoms are marked with circles, hydrogen atoms have been omitted for clarity.

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Structure and Conformation of the 1:1 Molecular Complex Sulfaproxyline–Caffeine

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Abstract. *p*-Isopropoxy-*N*-sulfanilylbenzamide–3,7-dihydro-1,3,7-trimethyl-1*H*-purine-2,6-dione (1/1), $C_{16}H_{18}N_2O_4S.C_8H_{10}N_4O_2$, $M_r = 528.58$, monoclinic, $P2_1/c$, $Z = 8$, $a = 12.197$ (1), $b = 34.109$ (5), $c = 12.878$ (1) Å, $\beta = 108.68$ (1)°, $V = 5075.4$ (1.0) Å^3, $D_x = 1.38$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 15.32$ cm^{-1}, $F(000) = 2224$, $T = 293$ K, final $R = 0.046$ for 5997 observed reflections with $F \geq 3\sigma(F)$. The asymmetric unit contains two sulfaproxyline and two caffeine molecules. In the crystal the flat caffeine molecule is packed parallel to another caffeine molecule. Hydrogen bonds, where present, are weak ($D\cdots A$ lengths > 3.0 Å), showing that the packing is dominated by the non-bonded interactions.

Introduction. Sulfonamides form molecular complexes with different small molecules. Trimethoprim–sulfamethoxazole (Nakai, Takasuka & Shiro, 1984), sulfathiazole–xanthine (Higuchi & Lach, 1954), sulfathiazole–theophylline (Shefter & Sackman, 1971) *etc.* are such molecular complexes. The molecular basis for complex formation in such a simple system helps in the understanding of the interaction of small molecules with a bioreceptor and of the resulting antibacterial activity.

Here crystallographic studies on such a sulfonamide complex (1:1 sulfaproxyline–caffeine) have been carried out to determine the nature of the interaction between the compounds, hydrogen bonding and change in structural features, if any, due to

this complex formation. Sulfaproxyline is widely used in synergestic combination with sulfamerazine, as the metabolites are readily soluble in acid urine preventing the formation of crystalluria even at acidic pH. Caffeine, a xanthine derivative, stimulates the central nervous system, cardiac muscle, cortex and respiratory system.

Experimental. Needle-shaped light-yellowish crystals were obtained by slow evaporation of an acetone solution of a 1:1 mixture of sulfaproxyline and caffeine. Weissenberg photographs showed the crystals to be monoclinic and systematic absence conditions determined the space group as $P2_1/c$. The lattice parameters a, b, c and β were also found using oscillation as well as Weissenberg photographs.

A crystal of dimensions $0.26 \times 0.08 \times 0.05$ mm was used for intensity data collection on an Enraf–Nonius CAD-4F diffractometer using Cu $K\alpha$ radiation. The cell parameters were determined by least-squares refinement of 48 reflections with $19 < \theta < 42°$. ω–2θ scans. The data were measured for values of 2θ up to 120° with $-13 \leq h \leq 13$, $0 \leq k \leq 38$ and $-14 \leq l \leq 14$. The reflection $\overline{3}57$ was measured at regular intervals as an intensity check with an average count of 449.5 and σ (calculated from distribution) = 7.2 (1.6%). The data were corrected for Lorentz and polarization factors but not for absorption. 11 447 values were recorded and merged using *SHELX76* (Sheldrick, 1976) to give 6454 unique reflections (merging $R = 0.02$). The structure was solved by direct methods using *MULTAN78* (Main,

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