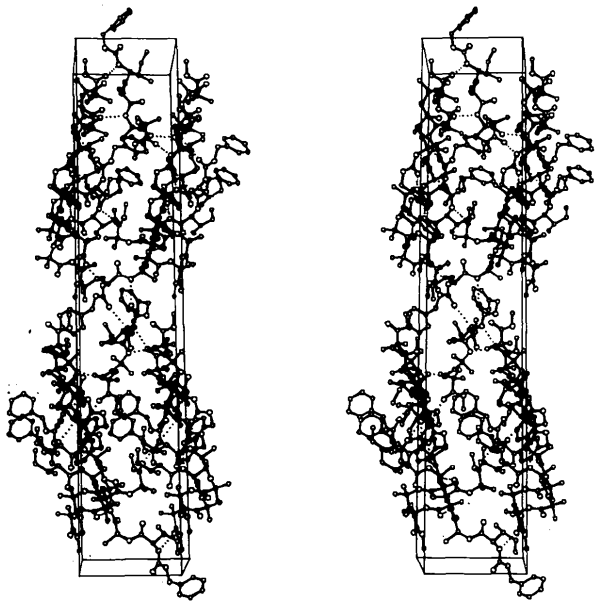

Fig. 2. Stereoview of the crystal structure. Dotted lines indicate intermolecular hydrogen bonds.

A stereoview of the structure is shown in Fig. 2. One molecule is hydrogen bonded to five adjacent molecules. All N and O atoms of the amide groups except for O(4) participate in hydrogen bonding, molecules being held together by three kinds of intermolecular hydrogen bonds: N(1)···O(6)($-\frac{1}{2}+x$, $\frac{3}{2}-y$, $\frac{1}{4}-z$) 2·895 (7), N(2)···O(2)($1-y$, $1-x$, $\frac{1}{2}-z$) 2·970 (7) and N(3)···O(5)($-\frac{1}{2}+x$, $\frac{5}{2}-y$, $\frac{1}{4}-z$) 2·887 (5) Å. In addition to these hydrogen bonds, van der Waals contacts between the hydrophobic isoleucine and *N*-terminal protecting groups stabilize the crystal structure. The isoleucyl side group adopts a conformation with χ_1[N(1)—C(9)—C(10)—C(12)] = −49·0 (6)° and χ_2[C(9)—C(10)—C(12)—C(13)] = −58·8 (9)°, while the *N*-terminal protecting group has torsion angles 125·2 (5) for O(1)—C(7)—C(1)—C(2), −76·7 (5) for C(8)—O(1)—C(7)—C(1), 161·9 (4) for C(7)—O(1)—C(8)—N(1) and −171·6 (4)° for O(1)—C(8)—N(1)—C(9). Short intermolecular distances are 3·660 (7) Å between C(3) and C(11)($1-y$, $1-x$, $\frac{1}{2}-z$) and 3·524 (8) Å between C(2) and C(20)($-\frac{3}{2}+y$, $\frac{1}{2}-x$, $\frac{1}{4}+z$). O(4) has a close intermolecular contact of 3·417 (8) Å to C(2)($1-y$, $1-x$, $\frac{1}{2}-z$).

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Structure of a New Cyclotetrapeptide Trapoxin A

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Abstract. $C_{34}H_{42}N_4O_6$, M_r = 602·73, triclinic, $P1$, a = 14·707 (2), b = 15·559 (2), c = 13·026 (1) Å, α = 112·23 (1), β = 97·25 (1), γ = 60·67 (1)°, V = 2399·1 (6) Å^3, Z = 3, D_x = 1·251 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 0·71 mm^{-1}, $F(000)$ = 966, T = 295 K, R = 0·042 for 7802 observed reflections [$F_o > 3\sigma(F_o)$]. The structure of trapoxin A was determined as *cyclo*[-(*S*)-phenylalanyl-(*S*)-phenylalanyl-(*R*)-pipecolinyl-(2*S*,9*S*)-2-amino-8-oxo-9,10-epoxydecanoyl-]. There are three crystallographically independent molecules in the cell. These molecules are linked to each other by NH···O hydrogen bonds to form an infinite chain extending along the *a* axis.

Introduction. Trapoxin A (I) is a new cyclotetrapeptide. It was isolated from the culture broth of

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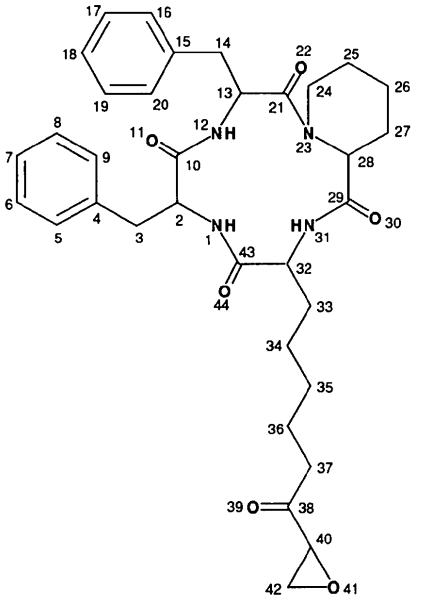
Helicoma ambiens RF-1023 (Itazaki, Nagashima, Sugita, Yoshida, Kawamura, Yasuda, Matsumoto, Ishii, Uotani, Nakai, Terui, Yoshimatsu, Ikenishi & Nakagawa, 1990). This compound exhibits a detransformation activity against *v*-sis oncogene-transformed NIH3T3 cells as an antitumor agent.



(I)

Experimental. Colorless needles obtained from an aqueous 2-propanol solution. Crystal of dimensions 0·1 × 0·2 × 0·4 mm. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell dimensions determined from 2θ angles for 24 reflections in the range $29 < 2\theta < 46°$. Intensities measured up to $\theta = 70°$ in h −17/0, k −18/16 and l −15/15, ω–2θ scans, ω-scan width $(1{\cdot}4 + 0{\cdot}2\tan\theta)°$. Three standard reflections monitored every 100 measurements showed about 3% decay, intensities corrected. 8879 unique reflections measured, 7802 intensities observed [those with $F_o \le 3\sigma(F_o)$ and three very strong reflections rejected], no absorption corrections. Fragments in structures obtained by *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987). Three crystallographically independent molecules (*A*, *B* and *C*) revealed after Fourier repetition. Molecules *A* and *B* both contained a disordered portion with two conformations. Occupancies of the disordered portions were estimated to be 0·5 and 0·5 for molecule *A* and 0·8 and 0·2 for molecule *B* from values of thermal parameters in least-squares refinements. H atoms in the disordered portions except those having the low occupancy in molecule *B* were calculated at their ideal positions. These positions were included in structure-factor calculations but not refined in least-squares refinements. Other H atoms were located on a difference density map. Anisotropic thermal parameters for non-H atoms, and isotropic for H atoms and the C and O atoms having the low occupancy in molecule *B*, refined by block-diagonal least squares. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00104|F_o|^2]$, $w = 0$ for 182 reflections with $w^{1/2}|\Delta F| \ge 3$. The final $R = 0{\cdot}042$, $wR = 0{\cdot}048$, $S = 1{\cdot}11442$. The highest and lowest peaks in the final difference map were 0·3 and −0·2 e Å^{-3}. Max. Δ/σ in the final cycle 0·3. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ $(i = 1,...,4)$ (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Calculations performed on a VAX station 3100 computer at Shionogi Research Laboratories.

Discussion. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Selected bond lengths and angles are listed in Table 2. The crystal structure of trapoxin A contains three independent molecules in the unit cell. These molecules are termed molecules *A*, *B* and *C*. A perspective view of molecule *A* is presented in Fig. 1.

* Lists of H-atom coordinates, anisotropic temperature factors of the non-H atoms, bond lengths, bond angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53762 (65 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

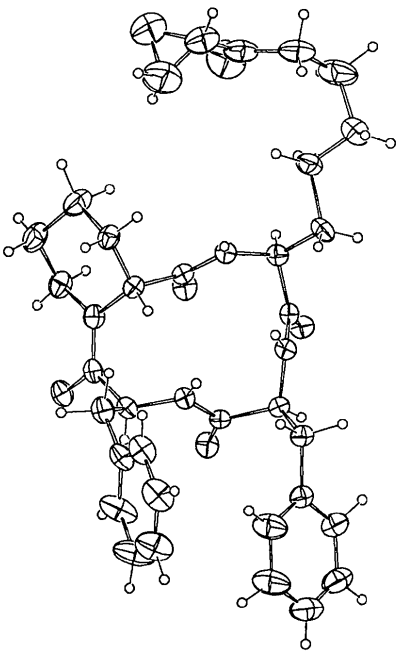


Fig. 1. *ORTEP* drawing of molecule *A* (Johnson, 1971; Hall & Stewart, 1989). The atoms with primes in the disordered portions are omitted for clarity. Non-H atoms are represented by thermal ellipsoids with 30% probability.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Molecule *A*				
N1	0·3336	0·3432	0·6098	3·3 (1)
C2	0·4018 (2)	0·2394 (2)	0·6179 (2)	3·3 (1)
C3	0·3407 (2)	0·2182 (2)	0·6845 (3)	4·0 (1)
C4	0·4022 (2)	0·1165 (2)	0·7070 (3)	4·0 (1)
C5	0·3775 (4)	0·0365 (4)	0·6618 (5)	7·2 (2)
C6	0·4297 (5)	−0·0544 (4)	0·6865 (6)	8·8 (3)
C7	0·5057 (4)	−0·0638 (4)	0·7617 (5)	7·3 (2)
C8	0·5322 (4)	0·0140 (4)	0·8060 (4)	7·1 (2)
C9	0·4808 (3)	0·1040 (3)	0·7794 (4)	5·7 (2)
C10	0·4434 (2)	0·1554 (2)	0·5014 (2)	3·2 (1)
O11	0·5368 (2)	0·0957 (2)	0·4778 (2)	4·6 (1)
N12	0·3710 (2)	0·1547 (2)	0·4272 (2)	3·4 (1)
C13	0·3957 (2)	0·0771 (2)	0·3140 (2)	3·6 (1)
C14	0·3066 (3)	0·0507 (3)	0·2758 (3)	4·8 (1)
C15	0·2908 (3)	−0·0055 (3)	0·3388 (3)	4·7 (1)
C16	0·3716 (3)	−0·0959 (3)	0·3494 (5)	7·6 (2)
C17	0·3591 (4)	−0·1491 (4)	0·4045 (6)	8·9 (3)
C18	0·2628 (4)	−0·1139 (4)	0·4503 (5)	7·9 (3)
C19	0·1804 (3)	−0·0243 (4)	0·4417 (4)	6·6 (2)
C20	0·1936 (3)	0·0317 (3)	0·3869 (4)	5·5 (2)
C21	0·4291 (2)	0·1004 (2)	0·2238 (2)	3·7 (1)
O22	0·5074 (2)	0·0282 (2)	0·1639 (2)	4·9 (1)
N23	0·3694 (2)	0·1919 (2)	0·2051 (2)	4·1 (1)
C24	0·4081 (3)	0·2073 (3)	0·1172 (3)	5·1 (2)
C25	0·3206 (4)	0·2604 (4)	0·0527 (3)	6·6 (2)
C26	0·2347 (3)	0·3639 (4)	0·1295 (4)	6·5 (2)
C27	0·1940 (2)	0·3427 (3)	0·2138 (3)	4·5 (1)
C28	0·2818 (2)	0·2851 (2)	0·2791 (2)	3·6 (1)
C29	0·3197 (2)	0·3569 (2)	0·3672 (2)	3·4 (1)
O30	0·4119 (2)	0·3351 (2)	0·3671 (2)	4·8 (1)
N31	0·2513 (2)	0·4394 (2)	0·4504 (2)	3·4 (1)
C32	0·2919 (2)	0·4887 (2)	0·5506 (2)	3·5 (1)
C33	0·2042 (3)	0·5802 (2)	0·6383 (3)	4·8 (1)
C34	0·1563 (7)	0·6739 (6)	0·5843 (7)	5·7 (3)
C35	0·0932 (7)	0·7781 (7)	0·6673 (8)	6·7 (4)
C36	0·0653 (5)	0·8558 (4)	0·6059 (6)	9·4 (3)
C37	−0·0140 (4)	0·8506 (4)	0·5217 (5)	7·1 (2)
C38	0·0243 (3)	0·7902 (4)	0·4055 (5)	6·8 (2)
O39	0·1113 (3)	0·7586 (4)	0·3708 (4)	10·7 (2)
C40	−0·0539 (5)	0·7697 (5)	0·3303 (5)	8·2 (3)
O41	−0·0307 (5)	0·7301 (4)	0·2150 (4)	11·7 (3)
C42	−0·0351 (8)	0·6660 (6)	0·2669 (8)	12·4 (5)
C43	0·3694 (2)	0·4030 (2)	0·5959 (2)	3·2 (1)
O44	0·4577 (1)	0·3914 (2)	0·6180 (2)	4·0 (1)
C34′	0·1183 (5)	0·6736 (5)	0·6197 (6)	4·4 (3)
C35′	0·1625 (6)	0·7349 (6)	0·5930 (8)	5·3 (3)
Molecule *B*				
N1	0·5732 (2)	0·5051 (2)	0·6120 (2)	3·0 (1)
C2	0·6194 (2)	0·5071 (2)	0·7181 (2)	3·2 (1)
C3	0·5374 (2)	0·5514 (3)	0·8118 (3)	4·2 (1)
C4	0·4717 (2)	0·6698 (3)	0·8505 (3)	4·6 (1)
C5	0·4978 (4)	0·7348 (4)	0·9393 (4)	7·3 (2)
C6	0·4419 (5)	0·8441 (4)	0·9712 (5)	9·5 (3)
C7	0·3601 (5)	0·8887 (4)	0·9198 (6)	9·5 (3)
C8	0·3347 (4)	0·8283 (4)	0·8285 (6)	9·5 (3)
C9	0·3900 (4)	0·7179 (3)	0·7922 (4)	7·0 (2)
C10	0·7086 (2)	0·3974 (2)	0·7068 (2)	3·0 (1)
O11	0·7958 (2)	0·3842 (2)	0·7351 (2)	3·9 (1)
N12	0·6863 (2)	0·3169 (2)	0·6654 (2)	2·9 (1)
C13	0·7634 (2)	0·2080 (2)	0·6511 (2)	3·0 (1)
C14	0·7121 (2)	0·1477 (2)	0·6677 (2)	3·4 (1)
C15	0·6836 (2)	0·1779 (2)	0·7887 (2)	3·7 (1)
C16	0·5997 (2)	0·2721 (3)	0·8484 (3)	4·4 (1)
C17	0·5761 (3)	0·2967 (3)	0·9588 (3)	5·9 (2)
C18	0·6364 (4)	0·2254 (4)	1·0099 (3)	6·5 (2)
C19	0·7214 (4)	0·1334 (4)	0·9532 (4)	6·7 (2)
C20	0·7450 (3)	0·1079 (3)	0·8418 (3)	5·2 (2)
C21	0·8374 (2)	0·1410 (2)	0·5437 (2)	3·1 (1)
O22	0·9302 (1)	0·0979 (2)	0·5567 (2)	4·6 (1)
N23	0·7999 (2)	0·1198 (2)	0·4411 (2)	3·1 (1)
C24	0·8797 (2)	0·0569 (3)	0·3459 (3)	4·2 (1)
C25	0·8416 (3)	0·0000 (3)	0·2429 (3)	4·9 (1)
C26	0·7382 (3)	0·0757 (3)	0·2147 (2)	4·7 (1)
C27	0·6588 (2)	0·1302 (2)	0·3127 (2)	3·7 (1)
C28	0·6943 (2)	0·1889 (2)	0·4180 (2)	3·0 (1)
C29	0·6906 (2)	0·2891 (2)	0·4161 (2)	2·9 (1)
O30	0·7662 (1)	0·3044 (2)	0·4389 (2)	3·8 (1)
N31	0·6002 (2)	0·3613 (2)	0·3945 (2)	3·3 (1)
C32	0·5891 (2)	0·4649 (2)	0·4136 (2)	3·3 (1)
C33	0·4823 (2)	0·5387 (2)	0·3878 (3)	4·2 (1)
C34	0·4639 (3)	0·5062 (4)	0·2662 (4)	6·6 (2)
C35	0·3585 (5)	0·5828 (5)	0·2411 (5)	9·0 (3)
C36	0·3425 (7)	0·5657 (7)	0·1231 (7)	9·7 (4)
C37	0·2381 (7)	0·6458 (6)	0·1021 (6)	10·0 (4)
C38	0·2307 (9)	0·6155 (8)	−0·0228 (7)	11·0 (5)
O39	0·2427 (10)	0·6685 (8)	−0·0663 (7)	16·9 (7)
C40	0·1941 (9)	0·5382 (7)	−0·0902 (6)	10·6 (5)
O41	0·2202 (7)	0·4557 (6)	−0·0555 (6)	12·8 (4)
C42	0·1204 (8)	0·5301 (10)	−0·0481 (8)	12·2 (6)
C43	0·6169 (2)	0·5105 (2)	0·5318 (2)	3·1 (1)
O44	0·6749 (2)	0·5501 (2)	0·5504 (2)	4·3 (1)
C36′	0·398 (2)	0·622 (2)	0·160 (2)	6·8 (5)
C37′	0·308 (2)	0·698 (2)	0·117 (2)	8·1 (6)
C38′	0·279 (2)	0·642 (2)	0·024 (3)	8·4 (6)
O39′	0·341 (2)	0·562 (2)	−0·069 (2)	12·2 (7)
C40′	0·180 (2)	0·634 (2)	0·021 (2)	7·6 (5)
O41′	0·091 (3)	0·687 (3)	−0·016 (3)	15·5 (10)
C42′	0·135 (4)	0·622 (4)	−0·084 (4)	12·1 (11)
Molecule *C*				
N1	0·9775 (2)	0·3633 (2)	0·4884 (2)	3·1 (1)
C2	0·9652 (2)	0·3979 (2)	0·3951 (2)	3·1 (1)
C3	0·8858 (2)	0·3730 (2)	0·3212 (2)	3·6 (1)
C4	0·8685 (2)	0·3931 (2)	0·2134 (2)	3·7 (1)
C5	0·9086 (3)	0·4442 (3)	0·1838 (3)	4·5 (1)
C6	0·8889 (3)	0·4603 (3)	0·0847 (3)	5·4 (2)
C7	0·8292 (3)	0·4228 (4)	0·0111 (3)	6·4 (2)
C8	0·7891 (4)	0·3709 (4)	0·0382 (3)	7·0 (2)
C9	0·8088 (3)	0·3555 (3)	0·1382 (3)	5·5 (2)
C10	0·9400 (2)	0·5148 (2)	0·4449 (2)	3·0 (1)
O11	1·0044 (2)	0·5402 (2)	0·4349 (2)	4·1 (1)
N12	0·8473 (2)	0·5844 (2)	0·5035 (2)	3·1 (1)
C13	0·8096 (2)	0·6990 (2)	0·5500 (2)	3·5 (1)
C14	0·6913 (2)	0·7589 (2)	0·5354 (3)	4·2 (1)
C15	0·6709 (2)	0·7544 (2)	0·4185 (3)	4·3 (1)
C16	0·6707 (3)	0·8320 (3)	0·3886 (4)	5·6 (2)
C17	0·6572 (3)	0·8285 (5)	0·2820 (5)	7·5 (3)
C18	0·6439 (3)	0·7493 (5)	0·2044 (4)	8·3 (3)
C19	0·6422 (4)	0·6702 (5)	0·2310 (4)	8·2 (2)
C20	0·6562 (3)	0·6740 (3)	0·3405 (3)	5·7 (2)
C21	0·8325 (2)	0·7532 (2)	0·6693 (3)	4·1 (1)
O22	0·8464 (2)	0·8269 (2)	0·6801 (2)	5·8 (1)
N23	0·8265 (2)	0·7311 (2)	0·7587 (2)	4·2 (1)
C24	0·8626 (4)	0·7841 (3)	0·8627 (3)	5·9 (2)
C25	0·8074 (4)	0·8022 (4)	0·9645 (4)	7·4 (2)
C26	0·8173 (4)	0·6999 (4)	0·9617 (3)	6·3 (2)
C27	0·7714 (3)	0·6552 (3)	0·8577 (3)	4·4 (1)
C28	0·8248 (2)	0·6359 (2)	0·7513 (2)	3·4 (1)
C29	0·9360 (2)	0·5410 (2)	0·7227 (2)	3·3 (1)
O30	1·0085 (2)	0·5485 (2)	0·6979 (2)	4·3 (1)
N31	0·9490 (2)	0·4479 (2)	0·7188 (2)	3·1 (1)
C32	1·0478 (2)	0·3530 (2)	0·6658 (2)	3·3 (1)
C33	1·0541 (2)	0·2546 (2)	0·6728 (2)	3·5 (1)
C34	1·0700 (3)	0·2540 (3)	0·7913 (3)	4·4 (1)
C35	1·0618 (3)	0·1654 (3)	0·8050 (3)	5·0 (2)
C36	1·0827 (3)	0·1634 (3)	0·9211 (3)	5·3 (2)
C37	1·0536 (3)	0·0921 (3)	0·9446 (3)	5·6 (2)
C38	1·0664 (3)	0·0964 (4)	1·0623 (4)	6·3 (2)
O39	1·0950 (3)	0·1516 (4)	1·1332 (3)	9·1 (2)
C40	1·0374 (5)	0·0312 (5)	1·0965 (6)	9·4 (3)
O41	0·9840 (5)	−0·0180 (5)	1·0223 (5)	13·4 (4)
C42	1·0816 (9)	−0·0797 (8)	1·0322 (14)	18·1 (8)
C43	1·0626 (2)	0·3427 (2)	0·5471 (2)	3·1 (1)
O44	1·1480 (1)	0·3175 (2)	0·5082 (2)	3·9 (1)

Structures of the molecules were determined as *cyclo*[-(*S*)-phenylalanyl-(*S*)-phenylalanyl-(*R*)-pipecolinyl-(2*S*,9*S*)-2-amino-8-oxo-9,10-epoxydecanoyl-], where the absolute configuration was based on an *R* configuration in the pipecolic acid determined by amino-acid chirality analysis. C34–C35 in molecule *A* and C36–C42 in molecule *B* are disordered with two conformations. The occupancies were estimated to be 0·5 and 0·5 for each conformation in molecule *A* and 0·8 and 0·2 for C36–C42 and C36′–C42′,

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

	Molecule *A*	Molecule *B*	Molecule *C*
N1—C2	1·463 (3)	1·457 (4)	1·464 (4)
N1—C43	1·344 (3)	1·342 (4)	1·343 (4)
C2—C3	1·540 (5)	1·530 (6)	1·522 (4)
C2—C10	1·528 (4)	1·523 (4)	1·541 (4)
C10—O11	1·218 (4)	1·226 (4)	1·228 (4)
C10—N12	1·346 (4)	1·347 (4)	1·343 (4)
N12—C13	1·457 (4)	1·457 (4)	1·467 (4)
C13—C14	1·530 (6)	1·544 (4)	1·547 (5)
C13—C21	1·548 (4)	1·548 (4)	1·547 (5)
C21—O22	1·222 (4)	1·213 (4)	1·215 (5)
C21—N23	1·357 (4)	1·363 (4)	1·357 (5)
N23—C24	1·483 (6)	1·482 (6)	1·483 (7)
N23—C28	1·455 (4)	1·466 (4)	1·460 (4)
C28—C29	1·534 (4)	1·543 (4)	1·538 (4)
C29—O30	1·226 (4)	1·226 (4)	1·222 (4)
C29—N31	1·339 (4)	1·338 (4)	1·346 (4)
N31—C32	1·464 (4)	1·461 (4)	1·466 (4)
C37—C38	1·453 (9)	1·519 (17)	1·501 (8)
C38—O39	1·202 (9)	1·246 (20)	1·193 (9)
C38—C40	1·491 (10)	1·491 (19)	1·491 (10)
C40—O41	1·405 (11)	1·383 (17)	1·405 (11)
C40—C42	1·405 (14)	1·352 (20)	1·431 (20)
O41—C42	1·428 (14)	1·338 (19)	1·302 (20)
C43—O44	1·232 (4)	1·231 (4)	1·231 (4)
C2—N1—C43	123·5 (2)	121·5 (2)	122·7 (3)
N1—C2—C3	108·6 (2)	111·9 (3)	108·7 (3)
N1—C2—C10	109·5 (2)	110·1 (3)	107·0 (3)
C3—C2—C10	114·0 (3)	112·3 (3)	116·8 (3)
C2—C3—C4	115·5 (3)	112·5 (4)	117·5 (3)
C2—C10—O11	121·2 (3)	121·1 (3)	120·7 (3)
C2—C10—N12	115·8 (3)	116·7 (3)	116·2 (3)
O11—C10—N12	123·0 (3)	122·2 (3)	123·0 (3)
C10—N12—C13	123·3 (3)	122·8 (3)	122·5 (3)
N12—C13—C14	110·4 (3)	111·4 (3)	109·9 (3)
N12—C13—C21	118·0 (3)	117·3 (3)	120·9 (3)
C14—C13—C21	110·2 (3)	110·4 (2)	109·5 (3)
C13—C14—C15	114·2 (4)	113·1 (3)	111·8 (3)
C13—C21—O22	116·2 (3)	116·0 (3)	113·9 (3)
C13—C21—N23	121·9 (3)	121·5 (3)	124·7 (3)
O22—C21—N23	121·6 (3)	121·9 (3)	120·9 (4)
C21—N23—C24	118·0 (3)	115·5 (3)	115·7 (4)
C21—N23—C28	124·6 (3)	123·0 (3)	124·1 (3)
C24—N23—C28	116·2 (3)	115·8 (3)	116·3 (3)
N23—C24—C25	111·5 (4)	111·7 (4)	112·0 (5)
N23—C28—C27	111·5 (3)	111·7 (3)	110·6 (3)
N23—C28—C29	110·5 (3)	108·9 (3)	110·0 (3)
C27—C28—C29	113·5 (3)	113·7 (3)	113·5 (3)
C28—C29—O30	121·3 (3)	121·2 (3)	121·0 (3)
C28—C29—N31	118·2 (3)	117·6 (3)	117·6 (3)
O30—C29—N31	120·3 (3)	121·2 (3)	121·3 (3)
C29—N31—C32	118·4 (3)	118·1 (3)	117·2 (3)
N31—C32—C33	111·9 (3)	112·1 (3)	111·5 (3)
N31—C32—C43	108·7 (3)	109·3 (3)	109·5 (3)
C33—C32—C43	111·6 (3)	113·3 (3)	113·3 (3)
C32—C33—C34	101·4 (4)	113·6 (4)	111·9 (3)
C37—C38—O39	124·0 (6)	117·2 (12)	124·8 (6)
C37—C38—C40	114·4 (6)	122·7 (11)	118·8 (5)
O39—C38—C40	121·6 (6)	119·4 (13)	116·4 (6)

Table 2 (*cont.*)

	Molecule *A*	Molecule *B*	Molecule *C*
C38—C40—O41	118·0 (7)	116·0 (11)	117·9 (7)
C38—C40—C42	121·3 (8)	121·4 (13)	118·7 (9)
O41—C40—C42	61·1 (7)	58·6 (10)	54·7 (8)
C40—O41—C42	59·4 (6)	59·6 (10)	63·7 (9)
C40—C42—O41	59·5 (6)	61·9 (10)	61·7 (9)
N1—C43—C32	115·3 (2)	115·6 (3)	116·3 (3)
N1—C43—O44	123·2 (3)	123·2 (3)	122·3 (3)
C32—C43—O44	121·5 (3)	121·2 (3)	121·4 (3)

respectively, in molecule *B* from the values of their thermal parameters. The accuracy of bond lengths in these disordered sites is relatively poor. Conformations of the 12-membered rings in the molecules *A*, *B* and *C* are almost the same.

In each main-chain ring all the three NH groups are on one side of the ring and the four CO groups are on the opposite side. The molecules are linked to each other along the *a* axis through intermolecular NH···O hydrogen bonds between the rings. O30*A*···N31*B* = 2·951 (4) [O···H = 2·25 (5)], O44*A*···N1*B* = 3·017 (4) [2·34 (3)], O44*A*···N12*B* = 3·038 (4) [2·16 (3)], O11*B*···N31*C* = 2·928 (4) [2·05 (5)], O44*B*···N12*C* = 3·008 (4) [2·27 (5)], O11*C*···N31*A* (1 + *x*, *y*, *z*) = 3·188 (4) [2·53 (5)], O44*C*···N1*A* = 3·020 (3) [2·17 (5)], O44*C*···N12*A* = 3·000 (4) Å [2·11 (5) Å].

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Lattice Inclusion Compounds of Gossypol. Structure of the 1:2 Gossypol/Salicylaldehyde Coordinatoclathrate

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Abstract. $C_{30}H_{30}O_8.2C_7H_6O_2$, $M_r = 762{\cdot}81$, monoclinic, $P2_1/n$, $a = 11{\cdot}130\ (2)$, $b = 29{\cdot}542\ (5)$, $c = 11{\cdot}744\ (2)$ Å, $\beta = 98{\cdot}45\ (1)°$, $V = 3820\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}33$ g cm^{-3}, μ(Cu $K\alpha$) = 8·09 cm^{-1}, $\lambda = 1{\cdot}51478$ Å, $F(000) = 1608$, $T = 293$ K, $R = 0{\cdot}059$ for 3541 observed reflections. The gossypol molecules

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