$-y$, $-z$; (iii) $-0.5-x$, $-0.5+y$, $1-z$]. These contacts are longer but stabilize the crystal packing (Fig. 2).

Least-squares-planes calculations (*XANADU*; Roberts & Sheldrick, 1975) show that the five-membered ring has an envelope conformation with C(2) 0.60 (1) Å out of the plane defined by the other four atoms C(1), C(3), C(4) and C(5). The angle between the normals to the carboxylic group and to the planar part of the five-membered ring is 53 (1)°, slightly different from the value observed in camphoric acid [40.4 (4)°] (Barnes, Paton, Blyth & Howie, 1991).

The CH_3—S— substituted phenyl ring is planar to within experimental accuracy ($\sigma = 0.01$ Å) and is inclined 64 (1)° to the cyclopentane ring. It is on the same side as the carboxylic plane with respect to the five-membered ring.

The Newman projections along the C(1)—C(2) and C(2)—C(3) bonds show a staggered conformation and the (1*R*,3*S*) configuration (Fig. 3). The Friedel–Crafts reaction does not change the absolute configuration of native camphoric acid.

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Structures of Three Tricyclic γ-Lactams

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Abstract. Synthetic γ-lactams formed in acidic media: (I) (3aα,4α,8bα)-3,3a,4,8b-tetrahydro-4-methylindano[1,2-*b*]pyrrol-2(1*H*)-one, $C_{12}H_{13}NO$, $M_r = 187.24$, monoclinic, $P2_1/c$, $a = 14.589$ (2), $b = 8.330$ (3), $c = 8.186$ (4) Å, $\beta = 103.17$ (2)°, $V = 968.6$ (5) Å^3, $Z = 4$, $D_x = 1.284$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.764$ cm^{-1}, $F(000) = 400$, $T = 173$ K, final $R = 0.055$ for 1880 unique observations, $I \geq 3\sigma(I)$; (II) (3aα,4α,8bα)-1-benzyl-3,3a,4,8b-tetrahydro-4-methylindano[1,2-*b*]pyrrol-2(1*H*)-one, $C_{19}H_{19}NO$, $M_r = 277.37$, orthorhombic, $P2_12_12_1$, $a = 9.100$ (3), $b = 9.678$ (4), $c = 17.360$ (2) Å, $V = 1528.8$ (5) Å^3, $Z = 4$, $D_x = 1.205$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5406 Å, $\mu = 5.416$ cm^{-1}, $F(000) = 592$, $T = 295$ K, final $R = 0.049$ for 729 observations, $I \geq 3\sigma(I)$; (III) (3aα,5α,9bα)-2,3,3a,4,5,9b-hexahydro-5-methylnaphtho[1,2-*b*]pyrrol-2(1*H*)-one, $C_{13}H_{15}NO$, $M_r = 201.27$, monoclinic, $P2_1/n$, $a = 8.121$ (5), $b = 16.257$ (4), $c = 8.686$ (2) Å, $\beta = 107.65$ (2)°, $V = 1092.7$ (7) Å^3, $Z = 4$, $D_x = 1.223$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.721$ cm^{-1}, $F(000) = 432$, $T = 295$ K, final $R = 0.046$ for 1522 unique observations,

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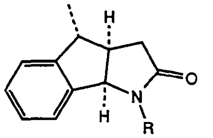
Table 1. *Crystal, intensity measurement and refinement data for* (I), (II) *and* (III)

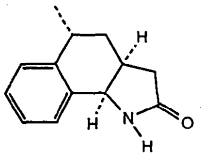
	(I)	(II)	(III)
Crystal size (mm)	0.70 × 0.70 × 0.20	0.30 × 0.15 × 0.20	0.30 × 0.40 × 0.50
Crystal shape, color	Tabloid, colorless	Acicular, colorless	Tabloid, colorless
Radiation	Mo $K\alpha$	Cu $K\alpha$	Mo $K\alpha$
Range of data	$2\theta_{\max} = 56°$	$2\theta_{\max} = 120°$	$2\theta_{\max} = 56°$
	$0 \le h \le 10$	$0 \le h \le 10$	$0 \le h \le 10$
	$0 \le k \le 11$	$0 \le k \le 10$	$0 \le k \le 21$
	$-19 \le l \le 19$	$0 \le l \le 19$	$-11 \le l \le 11$
No. of reflections collected	2505	1329	2798
No. of unique reflections	2342	1329	2626
R_{int}	0.011	–	0.012
Observed data [$I \ge 3\sigma(I)$]	1880	729	1522
Variables	166	190	196
R	0.0552	0.0492	0.0460
wR	0.0712	0.0531	0.0503
S	2.141	1.503	2.280
Absorption correction	–	0.894 minimum, 1.135 maximum	–

$I \ge 3\sigma(I)$. The lactam ring fusion in all molecules is *cis*. The methyl substituent lies on the convex face of the tricyclic systems, *syn* to the ring junction H atoms. Both five-membered rings in (I) and (II) adopt sofa conformations while in (III) the lactam ring adopts a sofa conformation and the cyclohexene ring adopts a distorted half-chair form. In the structures of (I) and (III) intermolecular hydrogen bonds are formed with N···O separations of 2.87–2.88 Å.

Introduction. We recently reported the first examples of γ-lactams prepared by formal insertions of the C atom of an aldehyde between the terminal atoms N and C4 of 3-alkenamides (Marson, Grabowska, Walsgrove, Eggleston & Baures, 1991). Crystal structure determinations of the lactams (I), (II) and (III) were undertaken to establish unambiguously the relative configurations of the lactams obtained by unprecedented condensation reactions effected in polyphosphoric acid (PPA), for lactams (I) and (III), and polyphosphoric ester (PPE) for lactam (II).



(I) $R = H$
(II) $R = CH_2C_6H_5$



(III)

Condensation of (*E*)-3-pentenamide with benzaldehyde in PPA at 308 K afforded the tricyclic lactam (I), the only stereoisomer detected. In contrast, reaction of (*E*)-3-hexenamide with benzaldehyde in PPA afforded a mixture of tricyclic lactams, epimeric at the C atom bearing a methyl group. Recrystallization of the reaction mixture from benzene afforded a pure diastereoisomer, (III), for which the configuration of the methyl group relative to the ring junction matched that of (I). Condensation of *N*-benzyl-(*E*)-3-pentenamide with benzaldehyde in PPE at 333 K afforded lactam (II), the only stereoisomer detected.

The γ-lactam ring of all three amides is apparently formed prior to an intramolecular Friedel–Crafts alkylation. Although other acid-catalyzed cyclizations afforded pyrrolidinones (Ben-Ishai, 1980; Tamura, Maeda, Akai, Ishiyama & Ishibashi, 1981), the pathways differ and the assembly of three contiguous stereogenic centers and three fused rings as for these lactams did not arise.

Experimental. Crystals of (I), (II) and (III) used for the diffraction experiments were grown by slow evaporation from ethyl acetate, diethyl ether and benzene, respectively. Crystal, intensity measurement and refinement data are presented in Table 1. Lattice parameters were determined from a least-squares analysis of the setting angles for 25 reflections in the range $30 \le 2\theta \le 35°$ (for Mo radiation) or 60–70° (for Cu radiation), as measured on an Enraf–Nonius CAD-4 diffractometer. Final diffractometer data were collected using variable-speed ω–2θ scans, where the final collection speed was determined from the intensity observed in a short prescan of each reflection and the scans were extended by 25% on each side of the predicted peak to collect estimates of background intensity. Data were corrected for background as well as Lorentz and polarization effects. For (I), three standards ($\bar{5}44$, $2\bar{6}\bar{1}$, $\bar{2}4\bar{7}$) measured at the beginning, end and every 3 h of exposure time showed no systematic deviations (maximum change ±1.1%). For (II), three standards (144, $\bar{3}\bar{3}4$, $4\bar{2}\bar{1}$) measured nine times during data collection showed a maximum increase of 3.6%. A correction (maximum 1.030, minimum 0.9198) was applied to these data. For (III), three standards ($\bar{2},\bar{3},\overline{13}$, $41\bar{8}$, $\bar{1},\bar{2},\overline{12}$) showed no systematic deviations (maximum change ±1.3%). Symmetry-equivalent data within the quadrants collected for (I) and (III) were averaged. Data for (II) were corrected for absorption using the *DIFABS* algorithm (Walker & Stuart, 1983). No attempt was made to assign an absolute configuration for the data crystal of (II); the compound apparently crystallized as a conglomerate since the bulk material displays no optical activity.

The structures were solved using *SHELXS* (Sheldrick, 1985) and refined by full-matrix least-squares techniques where the function minimized was $\sum w(|F_o| - |F_c|)^2$. Non-H atoms were refined with anisotropic displacement parameters. For (I) and (III), H-atom positions were located from difference Fourier maps and were refined; isotropic temperature factors for H atoms were assigned values of $1.3(B_{\rm iso})$ of the attached atom and held fixed for (I), but were refined for (III). For (II), the H-atom positions were assigned based on geometrical considerations and held fixed along with fixed isotropic temperature factors. For all three structures, weights were assigned to the data as $w = 4F_o^2/s^2(I)$ with $s^2(I) = \sigma^2(I_c) + [p(F_o)^2]^2$; $p = 0.05$ for (I) and (III) and 0.03 for (II). Refinements converged (maximum $\Delta/\sigma = 0.05$) to values of the standard crystallographic residuals listed in Table 1. Final difference Fourier maps were featureless with maximum features of ±0.375, ±0.158 and ±0.209 e Å^{-3} for (I), (II) and (III), respectively. Values of the neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) as incorporated in the Enraf–Nonius *SDP* (Frenz, 1987), a locally modified version of which was the source of all programs. For (I), refinement using all 2161 data not flagged as weak in a prescan gave $R = 0.061$, $wR = 0.073$; for (II), using 1131 data gave $R = 0.088$, $wR = 0.074$, and for (III), using 2171 data gave $R = 0.066$, $wR = 0.053$. There were 181 data flagged as weak in a prescan for (I), 312 for (II) and 255 such data for (III).

Discussion. Final atomic positional parameters and equivalent isotropic thermal factors for the non-H atoms are given in Table 2.* Principal bond distances and angles for all molecules are found in Table 3. Normal values for the types of bonds involved are observed in all structures. A slight asymmetry (1.3°) in exocyclic bond angles about C(2) in (I) is noted, whereas for both (II) and (III) the O(2)—C(2)—N(1) and O(2)—C(2)—C(3) angles are equivalent. For (I), the structure determination establishes that the γ-lactam-indano ring fusion is *cis* [H(4)—C(3a)—C(8b)—H(5) torsion of 18 (2)°] and that the methyl group at C(4) is *syn* to the ring-junction H atoms, residing on the convex face of the tricyclic system, as seen in Fig. 1. Both five-membered rings adopt sofa conformations with atom C(3a) out-of-plane in both rings. The dihedral angle between the planes defined by atoms C(4), C(4a), C(8a) and C(8b), and atoms C(8b), N(1), C(2) and C(3), is 64.6 (2)°.

Table 2. *Positional parameters and equivalent isotropic thermal factors* (Å^2) *for* (I), (II) *and* (III)

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
(I)				
O2	0.02051 (8)	0.2193 (2)	0.3382 (1)	2.42 (2)
N1	0.08529 (9)	0.1000 (2)	0.1403 (2)	1.70 (2)
C2	0.0704 (1)	0.1156 (2)	0.2948 (2)	1.74 (3)
C3	0.1235 (1)	−0.0160 (2)	0.4042 (2)	2.34 (3)
C3a	0.1882 (1)	−0.0885 (2)	0.3012 (2)	1.82 (3)
C4a	0.3088 (1)	0.0199 (2)	0.1736 (2)	1.70 (3)
C4	0.2901 (1)	−0.0206 (2)	0.3427 (2)	1.81 (3)
C5	0.3928 (1)	0.0659 (2)	0.1337 (2)	2.16 (3)
C6	0.3929 (1)	0.1042 (2)	−0.0312 (2)	2.37 (3)
C7	0.3107 (1)	0.0949 (2)	−0.1556 (2)	2.29 (3)
C8	0.2267 (1)	0.0493 (2)	−0.1174 (2)	1.95 (3)
C8a	0.2269 (1)	0.0122 (2)	0.0485 (2)	1.58 (3)
C8b	0.1445 (1)	−0.0360 (2)	0.1187 (2)	1.63 (3)
C9	0.3619 (1)	−0.1341 (2)	0.4488 (2)	2.84 (4)
(II)				
O2	0.4105 (5)	0.8495 (5)	0.8972 (2)	6.7 (1)
N1	0.2219 (5)	0.8970 (4)	0.9808 (2)	4.4 (1)
C2	0.3088 (7)	0.9226 (6)	0.9193 (3)	5.1 (2)
C3	0.2526 (7)	1.0526 (6)	0.8794 (4)	5.8 (2)
C3a	0.1451 (7)	1.1170 (6)	0.9375 (3)	4.8 (1)
C4	0.2166 (7)	1.2233 (6)	0.9925 (4)	5.7 (2)
C4a	0.1583 (7)	1.1848 (5)	1.0708 (3)	4.6 (1)
C5	0.1626 (8)	1.2613 (7)	1.1399 (3)	6.9 (2)
C6	0.1019 (9)	1.2014 (7)	1.2056 (3)	7.1 (2)
C7	0.0386 (8)	1.0715 (7)	1.2038 (3)	7.0 (2)
C8	0.0332 (7)	0.9971 (6)	1.1359 (3)	5.6 (2)
C8a	0.0927 (7)	1.0561 (6)	1.0694 (3)	4.1 (1)
C8b	0.1006 (7)	0.9961 (5)	0.9897 (3)	4.3 (1)
C9	0.1890 (9)	1.3723 (7)	0.9675 (4)	7.5 (2)
C10	0.2252 (7)	0.7657 (5)	1.0218 (3)	4.8 (1)
C11	0.3167 (7)	0.7636 (5)	1.0950 (3)	4.3 (1)
C12	0.2702 (8)	0.6767 (6)	1.1536 (3)	6.2 (2)
C13	0.3549 (9)	0.6648 (8)	1.2196 (3)	8.8 (2)
C14	0.4849 (9)	0.7331 (9)	1.2271 (3)	7.9 (2)
C15	0.5296 (8)	0.8211 (8)	1.1695 (3)	7.4 (2)
C16	0.4469 (7)	0.8381 (6)	1.1033 (3)	5.8 (2)
(III)				
O2	−0.0772 (2)	0.03603 (9)	0.2891 (2)	5.94 (3)
N1	0.1496 (2)	0.08343 (9)	0.4959 (2)	4.63 (3)
C2	0.0432 (2)	0.0837 (1)	0.3453 (2)	4.90 (4)
C3	0.0979 (3)	0.1526 (1)	0.2555 (2)	6.31 (6)
C3a	0.2795 (3)	0.1748 (1)	0.3606 (2)	5.18 (5)
C4	0.4155 (3)	0.1257 (1)	0.3122 (2)	5.54 (5)
C5a	0.5981 (2)	0.1201 (1)	0.5999 (2)	4.71 (4)
C5	0.5966 (3)	0.1355 (1)	0.4269 (2)	5.82 (5)
C6	0.7524 (3)	0.0986 (1)	0.7149 (3)	5.99 (6)
C7	0.7591 (3)	0.0810 (1)	0.8710 (3)	6.70 (6)
C8	0.6133 (3)	0.0852 (1)	0.9175 (2)	6.54 (6)
C9a	0.4489 (2)	0.1249 (1)	0.6466 (2)	4.25 (4)
C9	0.4593 (3)	0.1074 (1)	0.8070 (2)	5.61 (5)
C9b	0.2780 (2)	0.1502 (1)	0.5312 (2)	4.59 (4)
C10	0.6762 (3)	0.2184 (2)	0.4089 (3)	7.92 (6)

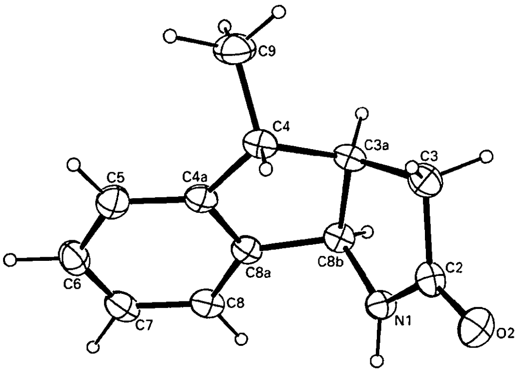


Fig. 1. *ORTEP*II (Johnson, 1976) view of (I) with non-H atoms as principal ellipses at the 50% probability level; H atoms as small spheres of arbitrary size.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond distances and angles involving H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55294 (46 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0201]

Table 3. *Principal bond distances* (Å) *and angles* (°) *for* (I), (II) *and* (III)

(I)			
O2—C2	1.233 (1)	C4a—C5	1.391 (2)
N1—C2	1.338 (1)	C4a—C8a	1.387 (2)
N1—C8b	1.460 (1)	C4—C9	1.527 (2)
C2—C3	1.512 (2)	C5—C6	1.388 (2)
C3—C3a	1.526 (2)	C6—C7	1.387 (2)
C3a—C4	1.554 (2)	C7—C8	1.384 (2)
C3a—C8b	1.548 (2)	C8—C8a	1.392 (2)
C4a—C4	1.509 (2)	C8a—C8b	1.500 (1)
C2—N1—C8b	114.69 (9)	C3a—C4—C9	113.4 (1)
O2—C2—N1	125.1 (1)	C4a—C4—C9	114.6 (1)
O2—C2—C3	126.4 (1)	C4a—C5—C6	119.2 (1)
N1—C2—C3	108.6 (1)	C5—C6—C7	120.6 (1)
C2—C3—C3a	104.93 (9)	C6—C7—C8	120.7 (1)
C3—C3a—C4	114.6 (1)	C7—C8—C8a	118.4 (1)
C3—C3a—C8b	104.54 (9)	C4a—C8a—C8	121.4 (1)
C4—C3a—C8b	105.67 (9)	C4a—C8a—C8b	111.08 (9)
C4—C4a—C5	129.0 (1)	C8—C8a—C8b	127.6 (1)
C4—C4a—C8a	111.2 (1)	N1—C8b—C3a	103.14 (9)
C5—C4a—C8a	119.7 (1)	N1—C8b—C8a	112.74 (9)
C3a—C4—C4a	104.21 (9)	C3a—C8b—C8a	104.87 (9)
(II)			
O2—C2	1.226 (7)	C5—C6	1.393 (8)
N1—C2	1.351 (7)	C6—C7	1.383 (9)
N1—C8b	1.470 (6)	C7—C8	1.382 (8)
N1—C10	1.458 (6)	C8—C8a	1.398 (7)
C2—C3	1.525 (8)	C8a—C8b	1.501 (7)
C3—C3a	1.537 (7)	C10—C11	1.520 (7)
C3a—C4	1.546 (7)	C11—C12	1.385 (7)
C3a—C8b	1.534 (7)	C11—C16	1.394 (7)
C4—C4a	1.506 (8)	C12—C13	1.386 (8)
C4—C9	1.527 (8)	C13—C14	1.36 (1)
C4a—C5	1.411 (7)	C14—C15	1.374 (9)
C4a—C8a	1.381 (7)	C15—C16	1.384 (8)
C2—N1—C8b	113.8 (5)	C6—C7—C8	120.5 (7)
C2—N1—C10	122.3 (5)	C7—C8—C8a	118.6 (6)
C8b—N1—C10	122.2 (5)	C4a—C8a—C8	121.4 (5)
O2—C2—N1	125.7 (6)	C4a—C8a—C8b	110.2 (5)
O2—C2—C3	126.0 (5)	C8—C8a—C8b	128.4 (5)
N1—C2—C3	108.3 (6)	N1—C8b—C3a	103.7 (4)
C2—C3—C3a	104.5 (5)	N1—C8b—C8a	112.7 (4)
C3—C3a—C4	114.0 (5)	C3a—C8b—C8a	105.1 (5)
C3—C3a—C8b	104.3 (4)	N1—C10—C11	115.6 (4)
C4—C3a—C8b	104.7 (4)	C10—C11—C12	117.0 (6)
C3a—C4—C4a	104.1 (5)	C10—C11—C16	123.0 (5)
C3a—C4—C9	112.6 (5)	C12—C11—C16	119.9 (6)
C4a—C4—C9	115.6 (5)	C11—C12—C13	119.1 (6)
C4—C4a—C5	128.9 (6)	C12—C13—C14	121.4 (7)
C4—C4a—C8a	111.0 (5)	C13—C14—C15	119.3 (7)
C5—C4a—C8a	120.1 (5)	C14—C15—C16	121.1 (7)
C4a—C5—C6	117.8 (6)	C11—C16—C15	119.1 (6)
C5—C6—C7	121.7 (7)		
(III)			
O2—C2	1.227 (2)	C5a—C6	1.389 (2)
N1—C2	1.331 (2)	C5a—C9a	1.391 (2)
N1—C9b	1.471 (2)	C5—C10	1.522 (2)
C2—C3	1.507 (2)	C6—C7	1.370 (2)
C3—C3a	1.524 (2)	C7—C8	1.364 (2)
C3a—C4	1.521 (2)	C8—C9	1.373 (2)
C3a—C9b	1.538 (2)	C9a—C9	1.399 (2)
C4—C5	1.514 (2)	C9a—C9b	1.503 (2)
C5a—C5	1.521 (2)		
C2—N1—C9b	114.2 (1)	C4—C5—C10	112.6 (2)
O2—C2—N1	126.2 (1)	C5a—C5—C10	111.8 (1)
O2—C2—C3	126.0 (1)	C5a—C6—C7	121.4 (2)
N1—C2—C3	107.8 (1)	C6—C7—C8	120.3 (2)
C2—C3—C3a	104.5 (1)	C7—C8—C9	119.6 (2)
C3—C3a—C4	111.3 (1)	C5a—9a—C9	118.9 (1)
C3—C3a—C9b	103.1 (1)	C5a—9a—C9b	122.1 (1)
C4—C3a—C9b	110.4 (1)	C9—C9a—C9b	119.0 (1)
C3a—C4—C5	113.9 (1)	C8—C9—C9a	121.1 (2)
C5—C5a—C6	119.2 (2)	N1—C9b—C3a	101.7 (1)
C5—C5a—C9a	122.2 (1)	N1—C9b—C9a	113.0 (1)
C6—C5a—C9a	118.7 (1)	C3a—C9b—C9a	116.0 (1)
C4—C5—C5a	110.4 (1)		

The molecular structure of (II), as seen in Fig. 2, is virtually identical to that of (I), disregarding the benzyl group. The γ-lactam ring fusion is *cis* with H(3)—C(3a)—C(8b)—H(9) torsion of 22.2 (8)°. Both five-membered rings adopt sofa forms with atom C(3a) out-of-plane from the other four atoms. The dihedral angle between these ring planes is 67.5 (2)°. The benzyl group orients perpendicularly to the lactam ring, as reflected in the C(8b)—N(1)—C(10)—C(11) torsion of −97.9 (6)°, and is turned so that one of the phenyl-ring bonds nearly eclipses the N(1)—C(10) bond; the N(1)—C(10)—C(11)—C(16) torsion is only −34.8 (8)°.

For (III), the γ-lactam-hexahydronaphtho ring junction is also *cis* [H(4)—C(3a)—C(9b)—H(5) torsion of 38 (2)°], and the methyl at C(5) is *syn* to the ring-junction H atoms, residing on the convex face of the tricyclic ring system, as seen in Fig. 3. The

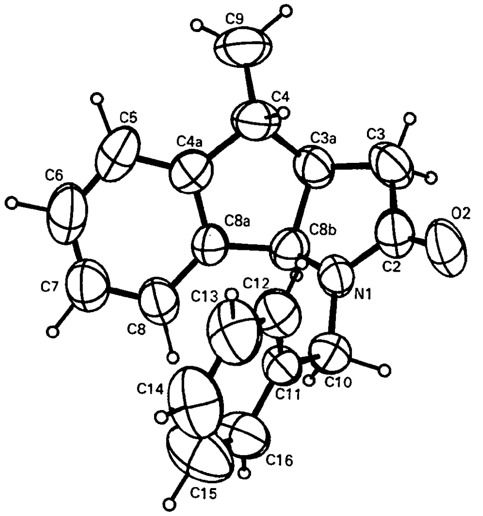

Fig. 2. View of (II) with non-H atoms as principal ellipses at the 50% probability level; H atoms as small spheres of arbitrary size.

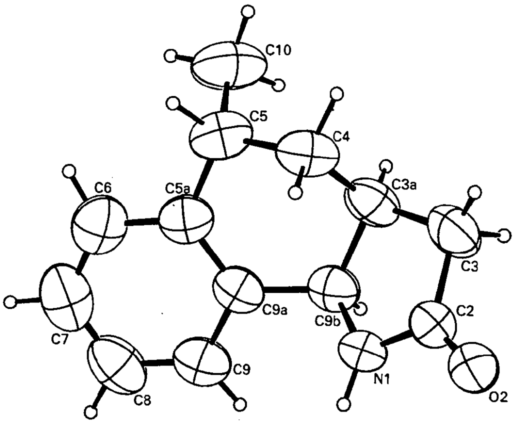

Fig. 3. View of (III) with non-H atoms as principal ellipses at the 50% probability level; H atoms as small spheres of arbitrary size.

γ-lactam ring adopts a sofa conformation in which C(3a) sits 0.462 (2) Å out-of-plane from the other four atoms. The cyclohexene ring adopts a distorted half-chair conformation with atoms C(9b), C(9a), C(5a) and C(5) virtually coplanar and atoms C(3a) and C(4) displaced by 0.136 (2) and 0.545 (2) Å, respectively, to opposite sides of the plane; the ring displacement asymmetry parameter, ΔC_2, is 4.5°. The dihedral angle between the planes defined by atoms C(9b), C(9a), C(5a) and C(5), and atoms C(9b), N(1), C(2) and C(3), is 57.6 (1)°.

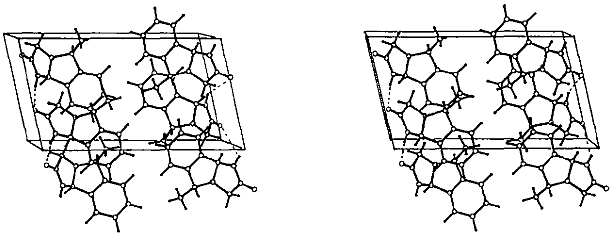

Fig. 4. Packing diagram for (I); all atoms are illustrated as spheres of arbitrary size. Hydrogen bonds are shown as dashed lines. The *c* axis is approximately vertical and the *a* axis is approximately horizontal.

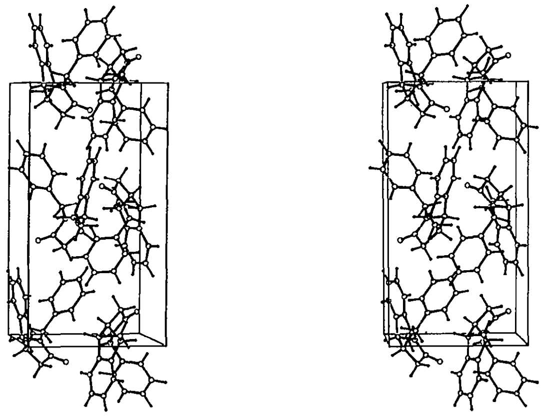

Fig. 5. Packing diagram for (II); all atoms are illustrated as spheres of arbitrary size. The *c* axis is vertical and the *a* axis is horizontal.

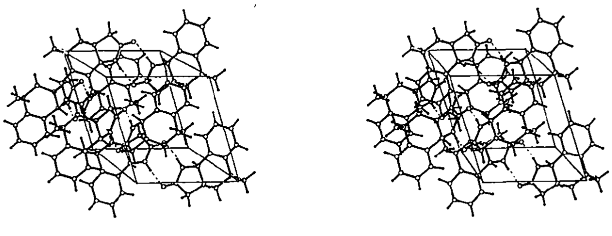

Fig. 6. Packing diagram for (III); all atoms are illustrated as spheres of arbitrary size. Hydrogen bonds are shown as dashed lines. The *a* axis is approximately vertical and the *c* axis is horizontal.

These tricyclic lactams are apparently structurally unique as a search of the Cambridge Structural Database failed to reveal any structures containing either motif with which to compare the present results. The parent system of (III) has been prepared by a thermal rearrangement (Oppolzer, 1971) but the molecular structure was not determined crystallographically.

Figs. 4, 5 and 6 display packing diagrams for (I), (II) and (III), respectively. The crystal structures of (I) and (III) are stabilized by a single hydrogen bond each. In (I), molecules are linked in chains parallel to the *c* axis; in (III) amide 'dimers' are formed through crystallographic inversion centers. Metrical details for (I) are N(1)···O(2) = 2.865 (1) and H(1)···O(2) = 1.93 (2) Å with an angle at H of 167 (3)°; for (III), N(1)···O(2) = 2.876 (2) and H(1)···O(2) = 2.01 Å with an angle at H of 173 (2)°. Numerous close contacts are observed between C and O atoms of glide-related molecules in the crystal structure of (I). The associated distances are C(3)···O(2) = 3.364 (2), C(3a)···O(2) = 3.399 (1) and C(8b)···O(2) = 3.235 (1) Å. None of the H atoms attached to close-contact C atoms in (I) are in a proper orientation to be consistent with criteria put forth by Taylor & Kennard (1982) for hydrogen-bond formation. For (II), which lacks traditional hydrogen-bonding donors, there are four intermolecular C···O distances within 3.5 Å. Of these only the C(12)···O(2) contact [3.400 (8) Å] has the H atom positioned in a geometrically reasonable orientation to constitute a C—H···O interaction. There is one close contact between C and O atoms in the crystal structure of (III), C(6)···O(2) = 3.435 (2) Å, with several others in the 3.5–3.6 Å range. None of these distances are consistent with C—H···O hydrogen-bond formation.

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