Structure of a Mixed Crystal of Stereoisomers of 4-Hydroxy-6-methoxy-5,9,13,13-tetramethyl-2-oxatetracyclo[6.5.0.0^{1,8}.0^{8,12}]tridecan-7-one*

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Abstract. $C_{17}H_{24}O_4$, $M_r = 292.3$, monoclinic, $P2_1$, $a = 7.883$ (2), $b = 14.347$ (3), $c = 27.673$ (6) Å, $\beta = 96.73$ (2)°, $V = 3108$ (1) Å^3, $Z = 8$, $D_x = 1.25$ Mg m^{-3}, $\lambda(CuK\alpha) = 1.5418$ Å, $\mu = 0.674$ mm^{-1}, $F(000) = 1264$, $T = 293$ K, $R = 0.050$ for 3885 observed reflections. This X-ray diffraction study establishes the molecular structure of the mixed crystal of diastereomers of the title compound. The stereochemistry at C(1), C(4), C(8) and C(12) for diastereomers (I) and (III) and (II) and (IV) is (R,S,R,S) and (S,R,S,R), respectively. The six-membered rings adopt skew-boat [molecules (I) and (III)] and half-chair [molecules (II) and (IV)] conformations. The five-membered rings adopt β-envelope [molecule (I)], half-chair [molecule (III)], and intermediate between half-chair and α-envelope [molecules (II) and (IV)] conformations. The crystal structure is stabilized by a three-dimensional network of hydrogen bonds and four intermolecular C—H···O interactions.

Introduction. In the course of our work on the ultraviolet irradiation of *O*-methylmethoxyperezone (Barrera, Barrios & Walls, 1980), the title compound was prepared. Its structure could not easily be deduced by ^{13}C NMR spectroscopy (Barrios, Salazar, Díaz, Walls & Joseph-Nathan, 1986) and X-ray analysis was required to establish this unequivocally.

The title compound was crystallized from an acetone–hexane solution and yielded primarily pale-yellow crystals together with a small proportion of colourless crystals. In a previous paper (Soriano-García, Walls, Yuste, Sánchez-Obregón, Ortíz, Díaz, Toscano & Barrios, 1989) we determined the X-ray structure of the pale-yellow crystals. We now report the crystal structure of the colourless form.

Experimental. Crystal used for data collection 0·12 × 0·24 × 0·40 mm, m.p. 410–411 K. Nicolet *R*3 four-

circle diffractometer and Ni-filtered Cu radiation. Lattice parameters from 25 machine-centred reflections with $10.9 < 2\theta < 24.6°$. 4115 reflections with $3 < 2\theta < 110°$ for two octants, 3885 independent with $I < 2.5\sigma(I)$, index range $h \pm 8$, k 0→15, l 0→29, ω-scan mode, variable scan speed, two standard reflections (00$\bar{2}$, 11$\bar{5}$) monitored every 50 measurements, Lp correction, absorption ignored and $R_{int} = 0.017$. Structure solved by direct methods using *SHELXTL5* (Sheldrick, 1985). Least-squares refinement of all non-H atoms with anisotropic thermal parameters; C(10), C(15) show some disorder (see thermal parameters in Table 1); H atoms of CH, CH$_2$ and CH$_3$ groups riding on bonded C with fixed isotropic temperature factors, coordinates of H atom bonded to O were refined, $U = 0.060$ Å^2. Function minimized $\sum w(\Delta F)^2$, $w = [\sigma^2(F_o) + 0.004(F_o)^2]^{-1}$, where σ is the standard deviation of observed amplitudes based on counting statistics. In the last cycle $(\Delta/\sigma)_{max} = 0.29$; residual electron density between -0.26 and 0·53 e Å^{-3}; final $R = 0.050$, $wR = 0.080$ and $S = 1.17$. Isotropic extinction parameter $X = 0.0019$. Scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL5* system of programs.

Discussion. A perspective view of the four independent molecules and the atomic numbering are shown in Fig. 1. Final atomic coordinates are given in Table 1‡ and bond lengths in Table 2.

The monoclinic unit cell contains two sets of four crystallographically independent molecules, an unusual situation. The carbon skeleton of the molecules comprises a system of fused cyclohexenone

‡ Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates, bond angles and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52875 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$U_{eq} = (U_1 U_2 U_3)^{1/3}.$$

Table 1 *(cont.)*

	x	y	z	$U_{eq}(\text{Å}^2)$
C(1)	0.5422 (5)	0.7875 (3)	0.3494 (1)	0.039 (1)
O(2)	0.6060 (4)	0.7288 (2)	0.3132 (1)	0.050 (1)
C(3)	0.7251 (6)	0.6855 (3)	0.3491 (1)	0.053 (1)
C(4)	0.6553 (5)	0.7365 (3)	0.3906 (1)	0.040 (1)
C(5)	0.7785 (5)	0.7953 (3)	0.4222 (1)	0.043 (1)
C(6)	0.7727 (5)	0.8876 (3)	0.4223 (1)	0.047 (1)
C(7)	0.6531 (5)	0.9422 (3)	0.3878 (2)	0.051 (2)
C(8)	0.5728 (5)	0.8953 (3)	0.3427 (1)	0.042 (1)
C(9)	0.6547 (7)	0.9218 (3)	0.2961 (1)	0.061 (2)
C(10)	0.5123 (9)	0.9167 (8)	0.2589 (2)	0.132 (4)
C(11)	0.3492 (7)	0.9430 (5)	0.2782 (2)	0.085 (2)
C(12)	0.3776 (6)	0.9097 (4)	0.3306 (2)	0.055 (2)
C(13)	0.3453 (5)	0.8049 (3)	0.3428 (1)	0.049 (1)
C(14)	0.9051 (5)	0.7404 (4)	0.4568 (2)	0.059 (2)
C(15)	0.7627 (12)	1.0025 (7)	0.2998 (3)	0.204 (6)
C(16)	0.2646 (5)	0.7969 (4)	0.3898 (2)	0.066 (2)
C(17)	0.2482 (6)	0.7463 (4)	0.3041 (2)	0.064 (2)
C(18)	0.9799 (7)	1.0079 (4)	0.4423 (2)	0.068 (2)
O(1)	0.5715 (4)	0.6730 (2)	0.4183 (1)	0.049 (1)
O(3)	0.6221 (5)	1.0232 (2)	0.3966 (1)	0.077 (1)
O(4)	0.8708 (4)	0.9376 (2)	0.4585 (1)	0.059 (1)
C(21)	0.2103 (5)	0.3796 (3)	0.3783 (1)	0.042 (1)
O(22)	0.2847 (4)	0.4304 (2)	0.3417 (1)	0.051 (1)
C(23)	0.3843 (6)	0.4881 (4)	0.3769 (1)	0.053 (2)
C(24)	0.3094 (5)	0.4395 (3)	0.4185 (1)	0.041 (1)
C(25)	0.4366 (5)	0.3892 (3)	0.4540 (1)	0.042 (1)
C(26)	0.4309 (5)	0.2957 (3)	0.4584 (1)	0.038 (1)
C(27)	0.3071 (5)	0.2346 (3)	0.4283 (1)	0.043 (1)
C(28)	0.2387 (5)	0.2712 (3)	0.3792 (1)	0.044 (1)
C(29)	0.3314 (6)	0.2264 (3)	0.3394 (2)	0.053 (2)
C(30)	0.2021 (7)	0.2375 (4)	0.2932 (2)	0.075 (2)
C(31)	0.0328 (7)	0.2173 (4)	0.3109 (2)	0.077 (2)
C(32)	0.0448 (6)	0.2588 (4)	0.3635 (2)	0.057 (2)
C(33)	0.0131 (5)	0.3640 (4)	0.3693 (2)	0.053 (1)
C(34)	0.5598 (6)	0.4475 (3)	0.4863 (2)	0.055 (2)
C(35)	0.5134 (6)	0.2589 (4)	0.3345 (2)	0.068 (2)
C(36)	-0.0728 (6)	0.4194 (4)	0.3263 (2)	0.068 (2)
C(37)	-0.0791 (6)	0.3826 (5)	0.4141 (2)	0.073 (2)
C(38)	0.6511 (6)	0.1864 (3)	0.4799 (2)	0.054 (2)
O(21)	0.2144 (4)	0.5033 (2)	0.4444 (1)	0.057 (1)
O(23)	0.2728 (4)	0.1580 (2)	0.4418 (1)	0.058 (1)
O(24)	0.5331 (4)	0.2529 (2)	0.4955 (1)	0.048 (1)
C(41)	0.8301 (5)	0.0606 (3)	0.1180 (1)	0.040 (1)
O(42)	0.7556 (4)	0.0123 (2)	0.1561 (1)	0.048 (1)
C(43)	0.6539 (6)	-0.0469 (3)	0.1222 (1)	0.055 (2)
C(44)	0.7219 (5)	0.0011 (3)	0.0787 (1)	0.039 (1)
C(45)	0.5920 (4)	0.0522 (3)	0.0451 (1)	0.036 (1)
C(46)	0.5895 (5)	0.1457 (3)	0.0428 (1)	0.037 (1)
C(47)	0.7064 (5)	0.2075 (3)	0.0728 (1)	0.037 (1)
C(48)	0.8067 (4)	0.1682 (3)	0.1169 (1)	0.035 (1)
C(49)	0.7666 (5)	0.2115 (3)	0.1653 (1)	0.047 (1)
C(50)	0.8935 (6)	0.2922 (4)	0.1728 (2)	0.058 (2)
C(51)	1.0601 (6)	0.2512 (3)	0.1590 (2)	0.056 (2)
C(52)	1.0052 (5)	0.1815 (3)	0.1185 (1)	0.043 (1)
C(53)	1.0289 (5)	0.0724 (3)	0.1249 (1)	0.043 (1)
C(54)	0.4695 (5)	-0.0079 (3)	0.0127 (1)	0.045 (1)
C(55)	0.5802 (6)	0.2375 (4)	0.1671 (2)	0.061 (2)
C(56)	1.1090 (6)	0.0398 (4)	0.1750 (2)	0.060 (2)
C(57)	1.1300 (5)	0.0322 (4)	0.0859 (2)	0.058 (2)
C(58)	0.3584 (5)	0.2517 (4)	0.0232 (2)	0.052 (1)
O(41)	0.8084 (4)	-0.0645 (2)	0.0526 (1)	0.054 (1)
O(43)	0.7224 (4)	0.2890 (2)	0.0614 (1)	0.051 (1)
O(44)	0.4797 (3)	0.1893 (2)	0.0059 (1)	0.044 (1)
C(61)	0.4344 (5)	0.6556 (3)	0.1493 (1)	0.037 (1)
O(62)	0.3659 (3)	0.7137 (2)	0.1858 (1)	0.044 (1)
C(63)	0.2562 (5)	0.7634 (3)	0.1492 (1)	0.047 (1)
C(64)	0.3276 (5)	0.7110 (3)	0.1088 (1)	0.039 (1)
C(65)	0.2045 (5)	0.6549 (3)	0.0747 (1)	0.039 (1)
C(66)	0.2103 (5)	0.5605 (3)	0.0747 (1)	0.040 (1)
C(67)	0.3308 (5)	0.5048 (3)	0.1076 (1)	0.039 (1)
C(68)	0.4099 (5)	0.5490 (3)	0.1540 (1)	0.039 (1)
C(69)	0.3286 (5)	0.5085 (3)	0.1980 (1)	0.046 (1)
C(70)	0.4674 (6)	0.5282 (3)	0.2420 (1)	0.054 (1)
C(71)	0.6335 (5)	0.5049 (3)	0.2208 (1)	0.055 (1)
C(72)	0.6065 (5)	0.5374 (3)	0.1674 (1)	0.045 (1)
C(73)	0.6321 (5)	0.6432 (3)	0.1563 (1)	0.038 (1)
C(74)	0.0784 (5)	0.7090 (4)	0.0409 (2)	0.053 (1)
C(75)	0.1489 (5)	0.5407 (4)	0.2049 (2)	0.063 (2)
C(76)	0.7181 (5)	0.6528 (4)	0.1101 (1)	0.051 (1)
C(77)	0.7272 (5)	0.7033 (3)	0.1973 (1)	0.052 (1)
C(78)	-0.0053 (5)	0.4432 (3)	0.0550 (2)	0.054 (1)
O(61)	0.4174 (4)	0.7735 (2)	0.0809 (1)	0.046 (1)
O(63)	0.3573 (4)	0.4238 (2)	0.0986 (1)	0.060 (1)
O(64)	0.1073 (4)	0.5122 (2)	0.0385 (1)	0.050 (1)

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

C(1)—O(2)	1.443 (5)	C(1)—C(4)	1.546 (5)
C(1)—C(8)	1.579 (6)	C(1)—C(13)	1.562 (5)
O(2)—C(3)	1.428 (5)	C(3)—C(4)	1.517 (6)
C(4)—C(5)	1.491 (5)	C(4)—O(1)	1.405 (5)
C(5)—C(6)	1.325 (6)	C(4)—C(14)	1.519 (6)
C(6)—C(7)	1.485 (6)	C(6)—O(4)	1.391 (5)
C(7)—C(8)	1.493 (6)	C(7)—O(3)	1.219 (6)
C(8)—C(9)	1.555 (6)	C(8)—C(12)	1.550 (6)
C(9)—C(10)	1.434 (7)	C(9)—C(15)	1.433 (11)
C(10)—C(11)	1.498 (9)	C(11)—C(12)	1.517 (7)
C(12)—C(13)	1.569 (7)	C(13)—C(16)	1.516 (6)
C(13)—C(17)	1.500 (6)	C(18)—O(4)	1.431 (6)
C(21)—O(22)	1.430 (5)	C(21)—C(24)	1.544 (5)
C(21)—C(28)	1.571 (6)	C(21)—C(33)	1.561 (5)
O(22)—C(23)	1.441 (5)	C(23)—C(24)	1.522 (6)
C(24)—C(25)	1.504 (5)	C(24)—O(21)	1.428 (5)
C(25)—C(26)	1.348 (6)	C(25)—C(34)	1.495 (6)
C(26)—C(27)	1.491 (5)	C(26)—O(24)	1.374 (4)
C(27)—C(28)	1.497 (5)	C(27)—O(23)	1.202 (5)
C(28)—C(29)	1.532 (6)	C(28)—C(32)	1.550 (6)
C(29)—C(30)	1.547 (6)	C(29)—C(35)	1.530 (7)
C(30)—C(31)	1.503 (8)	C(31)—C(32)	1.565 (7)
C(32)—C(33)	1.542 (7)	C(33)—C(36)	1.521 (7)
C(33)—C(37)	1.532 (7)	C(38)—O(24)	1.435 (6)
C(41)—O(42)	1.443 (5)	C(41)—C(44)	1.556 (5)
C(41)—C(48)	1.554 (6)	C(41)—C(53)	1.566 (5)
O(42)—C(43)	1.437 (5)	C(43)—C(44)	1.538 (6)
C(44)—C(45)	1.492 (5)	C(44)—O(41)	1.408 (5)
C(45)—C(46)	1.342 (6)	C(45)—C(54)	1.509 (5)
C(46) C(47)	1.465 (5)	C(46)—O(44)	1.406 (4)
C(47)—C(48)	1.484 (5)	C(47)—O(43)	1.222 (5)
C(48)—C(49)	1.543 (5)	C(48)—C(52)	1.571 (5)
C(49)—C(50)	1.527 (6)	C(49)—C(55)	1.523 (6)
C(50)—C(51)	1.527 (7)	C(51)—C(52)	1.525 (6)
C(52)—C(53)	1.584 (6)	C(53)—C(56)	1.529 (6)
C(53)—C(57)	1.527 (6)	C(58)—O(44)	1.432 (5)
C(61)—O(62)	1.460 (5)	C(61)—C(64)	1.542 (5)
C(61)—C(68)	1.549 (6)	C(61)—C(73)	1.558 (5)
O(62)—C(63)	1.440 (5)	C(63)—C(64)	1.510 (6)
C(64)—C(65)	1.504 (5)	C(64)—O(61)	1.425 (5)
C(65)—C(66)	1.354 (6)	C(65)—C(74)	1.500 (6)
C(66)—C(67)	1.472 (5)	C(66)—O(64)	1.398 (4)
C(67)—C(68)	1.499 (5)	C(67)—O(63)	1.213 (5)
C(68)—C(69)	1.554 (6)	C(68)—C(72)	1.560 (5)
C(69)—C(70)	1.565 (5)	C(69)—C(75)	1.523 (6)
C(70)—C(71)	1.534 (6)	C(71)—C(72)	1.541 (6)
C(72)—C(73)	1.566 (6)	C(73)—C(76)	1.524 (5)
C(73)—C(77)	1.545 (5)	C(78)—O(64)	1.440 (6)

(*A*), cyclobutane (*C*) and cyclopentane (*D*) rings. Further, the *A* ring is fused to an oxetane (*B*) ring. The *A/B*, *A/C* and *C/D* ring junctions are *cis*, except for the *A/B* and *A/C* junctions in molecules (II) and (III) which are *trans*.

The absolute molecular structures are illustrated in Fig. 1. The two pairs of diastereomers [(I) and (III)] (1*R*,4*S*,8*R*,12*S*) and [(II) and (IV)] (1*S*,4*R*,8*S*,12*R*) have been assigned from the C(9)*R*

$C_{17}H_{24}O_4$

stereochemistry of (+)-cedrol deduced from chemical studies (Stork & Clarke, 1961) and from X-ray studies on cedryl chromate (Amirthalingam, Grant & Senol, 1972).
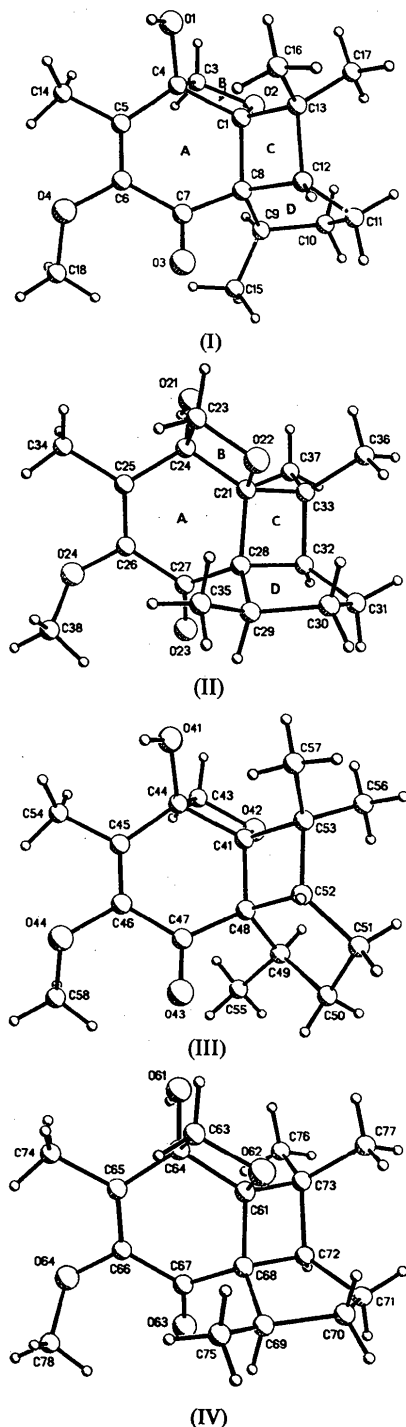

(I)

(II)

(III)

(IV)

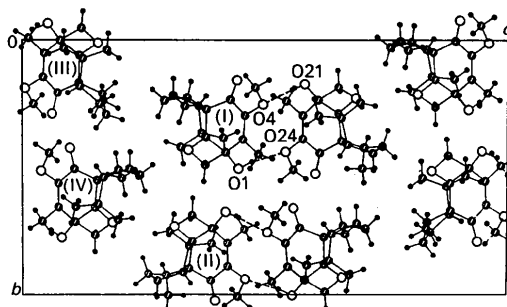Fig. 1. View of the four independent molecules showing the atom labelling.

Fig. 2. The packing arrangement as viewed along a. Hydrogen bonds are shown by broken lines.

The parameters of the ring-puckering analysis (Cremer & Pople, 1975) [for the sequences C(5)—C(6)—C(7)—C(8)—C(1)—C(4), C(25)—C(26)—C(27)—C(28)—C(21)—C(24), C(45)—C(46)—C(47)—C(48)—C(41)—C(44) and C(65)—C(66)—C(67)—C(68)—C(61)—C(64)] are $\varphi = 342$ (1), $\theta = 111$ (1)°, $Q = 0.283$ (3) Å; $\varphi = 148$ (1), $\theta = 73$ (1)°, $Q = 0.333$ (4) Å; $\varphi = 323$ (1), $\theta = 103$ (1)°, $Q = 0.200$ (3) Å; and $\varphi = 153$ (1), $\theta = 68$ (1)°, $Q = 0.274$ (4) Å, indicating skew-boat, half-chair, skew-boat and half-chair conformations, respectively.

In (I) and (III) the five-membered (D) rings have Δ and φ_m values (Altona, Geise & Romers, 1968) of 37·6 (6), −34·1 (6)° and 3·4 (5), −42·5 (5)°, indicating β-envelope and half-chair conformations, respectively, whereas in (II) and (IV) the Δ and φ_m values are −17·2 (5), −40·7 (5)° and −14·1 (4), −41·5 (4)°, indicating an intermediate between half-chair and α-envelope conformations.

Examination of the crystal packing (Fig. 2) reveals that the structure is stabilized by four intermolecular hydrogen bonds, forming two pairs of molecules [(I) and (II); (III) and (IV)]: O(1)—H···O(24)(1 − x, 1/2 + y, 1 − z) 2·854 (4), O(21)—H···O(4)(1 − x, −1/2 + y, 1 − z) 2·997 (4), O(41)—H···O(64)(1 − x, −1/2 + y, −z) 2·898 (4) and O(61)—H···O(44)(1 − x, 1/2 + y, −z) 2·888 (4) Å; and four intermolecular C—H···O interactions <3·4 Å: C(18)···O(23)(1 + x, 1 + y, z) 3·158 (6), C(78)···O(43)(−1 + x, y, z) 3·102 (5) and C(38)···O(3), C(43)···O(61)(x, −1 + y, z) 3·275 (6), 3·305 (5) Å, respectively.

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Structures du *N*-Diméthylaminoéthyl Méthoxy-4 Pyridinesulfonamide-3 et du *N*-[(Éthyl-1 pyrrolidinyl-2) méthyl] Pyridinesulfonamide-3

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Abstract. (I) *N*-(Dimethylaminoethyl)-4-methoxypyridine-3-sulfonamide, $C_{10}H_{17}N_3O_3S$, $M_r = 259\cdot33$, m.p. = 351 K, monoclinic, $P2_1/n$, $a = 7\cdot158$ (1), $b = 25\cdot714$ (7), $c = 7\cdot479$ (1) Å, $\beta = 107\cdot85$ (1)°, $V = 1310\cdot2$ (1) Å^3, $Z = 4$, $D_x = 1\cdot314$ g cm^{-3}, λ(Cu $K\bar{\alpha}$) $= 1\cdot5418$ Å, $\mu = 21\cdot71$ cm^{-1}, $F(000) = 552$, $T = 290$ K, final $R = 0\cdot060$ for 1489 reflections measured with $F_o > 0$. (II) *N*-[(1-Ethyl-2-pyrrolidinyl)methyl]pyridine-3-sulfonamide, $C_{12}H_{19}N_3O_2S$, $M_r = 269\cdot37$, m.p. = 343 K, monoclinic, $P2_1/n$, $a = 9\cdot557$ (1), $b = 14\cdot767$ (1), $c = 10\cdot471$ (1) Å, $\beta = 105\cdot08$ (1)°, $V = 1426\cdot9$ (1) Å^3, $Z = 4$, $D_x = 1\cdot253$ g cm^{-3}, λ(Cu $K\bar{\alpha}$) $= 1\cdot5418$ Å, $\mu = 19\cdot60$ cm^{-1}, $F(000) = 576$, $T = 290$ K, final $R = 0\cdot068$ for 1641 reflections measured with $F_o > 0$. Conformational differences between the two molecules are discussed. The cohesion of the crystals is the result of van der Waals contacts and NH···N hydrogen bonds with N···N = 2·971 (6) (I) and 3·015 (6) Å (II).

Introduction. Dans un article précédent, a été publiée la structure d'un analogue hétérocyclique sulfonamidé des orthopramides classiques (Sbit, Dupont, Dideberg, Liégeois & Delarge, 1988) [Fig. 1*c*, molécule (III)]. Dans ces derniers, la nature de la chaîne latérale azotée peut varier dans certaines limites sans altérer profondément la disposition spatiale et l'activité pharmacologique (Cesario, Pascard, El Moukhtari & Jung, 1981; Bass & Robie, 1984;

Collin, Norberg, Evrard, Durant, Tollenaere & Moereels, 1988; De Paulis, Kumar, Johansson, Rämsby, Hall, Sällemark, Ängeby-Möller & Ögren, 1986). Il semble établi que l'orientation de cette chaîne aminée est largement conditionnée par la présence d'une liaison hydrogène entre l'oxygène du groupement méthoxylé et l'azote de la fonction amide qui détermine la formation d'une nouvelle structure pseudocyclique (Furaya, Iwanami, Takenata & Sasada, 1982; Van de Waterbeemd & Testa, 1981, 1983). L'objet de ce travail est l'étude de deux autres composés hétérocycliques, l'un (I) (Fig. 1*a*) voisin du premier dérivé examiné (III), l'autre (II) (Fig. 1*b*) fortement différent puis qu'il ne possède plus de groupement méthoxylé et que l'azote basique est inclu dans une *N*-éthyl-pyrrolidine. Les travaux réalisés jusqu'à présent sur les dérivés sulfonamidés sont rares et, mis à part un travail très récent (Collin, Vercauteren, Evrard & Durant, 1989) ne s'intéressent pas à la formation éventuelle du pseudocycle évoqué plus haut (Boudet-Dalbin, Durand, Adam, Moreau & Foussart-Blanpin, 1986; Boudet-Dalbin, Grassy, Adam & Moreau, 1986).

Partie expérimentale. (I) Cristallisé dans le méthanol. Cristal incolore: 0,7 × 0,1 × 0,2 mm. Paramètres de la maille déterminés à partir de 38 réflexions (18,6 $\leq \theta \leq 33,2°$). Diffractomètre Siemens, 1856 réflexions mesurées, $3 \leq \theta \leq 55°$, Cu $K\bar{\alpha}$, monochro-