Related literature. New regio- and stereoselective preparation of trichlorinated γ-butyrolactones by copper-catalyzed cyclization of allyl trichloroacetates (Nagashima, Wakamatsu, Itoh, Tomo & Tsuji, 1983).

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Structure of 6-Fluoro-1β,2β-methylene-3,20-dioxo-9β,10α-pregna-4,6-dien-17α-yl Acetate

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Abstract. $C_{24}H_{29}FO_4$, $M_r = 400.5$, orthorhombic, $P2_12_12_1$, $a = 12.207\,(1)$, $b = 14.715\,(1)$, $c = 11.572\,(1)$ Å, $V = 2078.7\,(5)$ Å^3, $Z = 4$, $D_x = 1.28$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.5418$ Å, $\mu = 7.62$ cm^{-1}, $F(000) = 856$, $T = 291$ K, final $R = 0.041$ for 2230 observed reflections. The *A* and *B* rings assume flat boat and 9β,10α-half-chair conformations, with asymmetry parameters $\Delta C_s^3 = 1.5$, $\Delta C_s^{1,2} = 11.2$ and $\Delta C_2^{6,7} = 3.1$, $\Delta C_s^7 = 15.0$, respectively.

Experimental. A crystal (0.08 × 0.40 × 0.48 mm) was grown from acetone solution. The X-ray data were collected on a CAD-4 diffractometer using Cu $K\alpha$ (Ni filter) radiation for reflections with 2θ from 4 to 150°, range h,k,l, $0 \le 15$, $0 \le 19$, $0 \le 16$; $\omega/2\theta$ scan with minimum scan speed of 4° min^{-1} in θ and scan width of $(0.75 + 0.14\tan\theta)$°. Cell dimensions calculated by least-squares refinement of 25 reflections, $50 < 2\theta < 60$°. Intensities of two standard reflections (274, 524) were measured every 4 h and varied by less than 2% throughout the data collection. Intensities for 2478 reflections measured, 2422 unique reflections and 2230 with $F_o > 2\sigma(F_o)$ used in the refinement. The structure was determined using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined on *F* by full-matrix least-squares method. H atoms were located by difference Fourier synthesis and were refined isotropically. H atoms on C21 were unreasonable and were replaced with theoretical hydrogens, held constant. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.041$, $wR = 0.055$, $S = 1.555$, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\max} = 0.02$. Weighting scheme based on estimates of experimental errors from counting statistics. The final difference map showed maximum and minimum peaks of 0.19 and -0.20 e Å^{-3}. No corrections for absorption or extinction were made.

Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1. The bond lengths and angles are presented in Table 2.* An

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* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54647 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for non*-H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
C(1)	8334 (2)	165 (1)	2161 (2)	52 (1)
C(2)	9393 (2)	583 (2)	2597 (2)	58 (1)
C(3)	10113 (2)	37 (2)	3332 (2)	56 (1)
C(4)	9878 (2)	− 938 (2)	3403 (2)	56 (1)
C(5)	8931 (2)	− 1309 (1)	3034 (2)	47 (1)
C(6)	8760 (2)	− 2285 (2)	3103 (2)	56 (1)
C(7)	7978 (2)	− 2740 (1)	2575 (2)	57 (1)
C(8)	7113 (2)	− 2290 (1)	1871 (2)	47 (1)
C(9)	7397 (1)	− 1286 (1)	1591 (2)	42 (1)
C(10)	7956 (1)	− 760 (1)	2602 (2)	42 (1)
C(11)	6418 (2)	− 793 (1)	1011 (2)	46 (1)
C(12)	5268 (2)	− 968 (1)	1513 (2)	45 (1)
C(13)	5055 (2)	− 1991 (1)	1643 (2)	44 (1)
C(14)	5968 (2)	− 2394 (1)	2404 (2)	44 (1)
C(15)	5563 (2)	− 3352 (1)	2696 (3)	63 (1)
C(16)	4313 (2)	− 3224 (2)	2838 (3)	62 (1)
C(17)	4024 (2)	− 2271 (1)	2368 (2)	48 (1)
C(18)	4974 (2)	− 2441 (2)	443 (2)	60 (1)
C(19)	7216 (2)	− 592 (2)	3664 (2)	50 (1)
C(20)	2961 (2)	− 2246 (2)	1655 (2)	56 (1)
C(21)	2462 (2)	− 1329 (2)	1410 (3)	70 (1)
C(22)	3071 (2)	− 1625 (2)	3998 (2)	62 (1)
C(23)	3065 (2)	− 832 (2)	4785 (2)	79 (1)
C(1*B*)	9258 (2)	270 (2)	1348 (2)	66 (1)
F(6)	9528 (1)	− 2734 (1)	3735 (2)	81 (1)
O(3)	10892 (1)	373 (1)	3840 (2)	77 (1)
O(17*A*)	3953 (1)	− 1605 (1)	3293 (1)	51 (1)
O(20)	2594 (2)	− 2925 (1)	1227 (2)	75 (1)
O(22)	2381 (2)	− 2206 (2)	3937 (2)	85 (1)

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

C(1)—C(2)	1.518 (3)	C(10)—C(19)	1.546 (3)
C(1)—C(10)	1.525 (3)	C(11)—C(12)	1.540 (3)
C(1)—C(1*B*)	1.476 (3)	C(12)—C(13)	1.535 (3)
C(2)—C(3)	1.464 (3)	C(13)—C(14)	1.540 (3)
C(2)—C(1*B*)	1.526 (4)	C(13)—C(17)	1.568 (3)
C(3)—C(4)	1.465 (3)	C(13)—C(18)	1.541 (3)
C(3)—O(3)	1.223 (3)	C(14)—C(15)	1.531 (3)
C(4)—C(5)	1.348 (3)	C(15)—C(16)	1.546 (4)
C(5)—C(6)	1.454 (3)	C(16)—C(17)	1.545 (3)
C(5)—C(10)	1.523 (3)	C(17)—C(20)	1.538 (3)
C(6)—C(7)	1.316 (3)	C(17)—O(17*A*)	1.454 (3)
C(6) F(6)	1.360 (3)	C(20)—C(21)	1.508 (4)
C(7)—C(8)	1.489 (3)	C(20)—O(20)	1.201 (3)
C(8)—C(9)	1.552 (3)	C(22)—C(23)	1.481 (4)
C(8)—C(14)	1.536 (3)	C(22)—O(17*A*)	1.351 (3)
C(9)—C(10)	1.560 (3)	C(22)—O(22)	1.202 (3)
C(9)—C(11)	1.551 (3)		

C(2)—C(1)—C(10)	120.6 (2)	C(9)—C(10)—C(19)	114.9 (1)
C(2)—C(1)—C(1*B*)	61.3 (1)	C(9)—C(11)—C(12)	117.5 (1)
C(10)—C(1)—C(1*B*)	122.5 (2)	C(11)—C(12)—C(13)	110.8 (1)
C(1)—C(2)—C(3)	118.8 (2)	C(12)—C(13)—C(14)	108.1 (1)
C(1)—C(2)—C(1*B*)	58.0 (1)	C(12)—C(13)—C(17)	116.5 (1)
C(3)—C(2)—C(1*B*)	116.7 (2)	C(12)—C(13)—C(18)	110.1 (2)
C(2)—C(3)—C(4)	117.0 (2)	C(14)—C(13)—C(17)	100.0 (1)
C(2)—C(3)—O(3)	121.6 (2)	C(14)—C(13)—C(18)	113.3 (2)
C(4)—C(3)—O(3)	121.5 (2)	C(17)—C(13)—C(18)	108.5 (2)
C(3)—C(4)—C(5)	123.1 (2)	C(8)—C(14)—C(13)	113.0 (1)
C(4)—C(5)—C(6)	120.4 (2)	C(8)—C(14)—C(15)	118.3 (2)
C(4)—C(5)—C(10)	124.0 (2)	C(13)—C(14)—C(15)	104.3 (2)
C(6)—C(5)—C(10)	115.4 (2)	C(14)—C(15)—C(16)	103.3 (2)
C(5)—C(6)—C(7)	125.5 (2)	C(15)—C(16)—C(17)	107.4 (2)
C(5)—C(6)—F(6)	114.2 (2)	C(13)—C(17)—C(16)	104.1 (2)
C(7)—C(6)—F(6)	120.2 (2)	C(13)—C(17)—C(20)	112.5 (2)
C(6)—C(7)—C(8)	122.8 (2)	C(13)—C(17)—O(17*A*)	105.3 (1)
C(7)—C(8)—C(9)	112.3 (2)	C(16)—C(17)—C(20)	113.8 (2)
C(7)—C(8)—C(14)	112.4 (2)	C(16)—C(17)—O(17*A*)	111.5 (2)
C(9)—C(8)—C(14)	112.5 (1)	C(20)—C(17)—O(17*A*)	109.2 (1)
C(8)—C(9)—C(10)	114.4 (1)	C(17)—C(20)—C(21)	117.5 (2)
C(8)—C(9)—C(11)	111.3 (1)	C(17)—C(20)—O(20)	121.1 (2)
C(10)—C(9)—C(11)	115.4 (1)	C(21)—C(20)—O(20)	121.1 (2)
C(1)—C(10)—C(5)	110.2 (1)	C(23)—C(22)—O(17*A*)	110.9 (2)
C(1)—C(10)—C(9)	108.9 (1)	C(23)—C(22)—O(22)	126.4 (2)
C(1)—C(10)—C(19)	107.5 (1)	O(17*A*)—C(22)—O(22)	122.6 (2)
C(5)—C(10)—C(9)	108.9 (1)	C(1)—C(1*B*)—C(2)	60.7 (1)
C(5)—C(10)—C(19)	106.3 (1)	C(17)—O(17*A*)—C(22)	118.5 (1)

*ORTEP*II view of the molecule with atomic numbering (Johnson, 1976) is shown in Fig. 1. A stereoview of the crystal packing along the *b* axis is presented in Fig. 2.

Related literature. The title compound was studied to examine the effect of the unnatural configuration at C9 and C10, combined with substitution, on the conformation of the *B* ring and progesterone side chain. In a study of 85 steroids having the progesterone side chain, the average value of the C16—C17—C20—O20 torsion angle was −21 (9)°, in a sample of 81/85 with angles from −40 to 0°. Although the sample of 17α-ester compounds was small (eight structures) there was evidence of restriction in the conformation [average torsion angle = −21 (2)°] (Duax, Griffin & Rohrer, 1981). The value of this angle in the present study is −20.9 (3)°.

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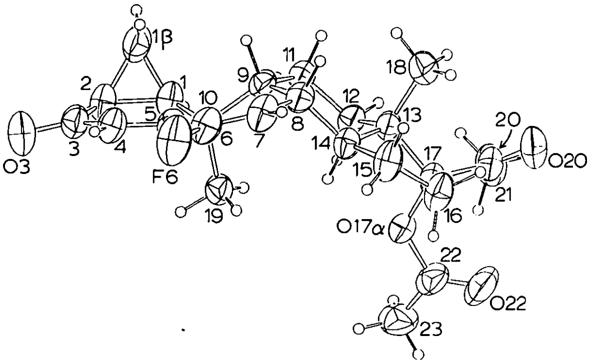

Fig. 1. *ORTEP*II view of the molecule with 50% probability thermal ellipsoids, showing atomic numbering.

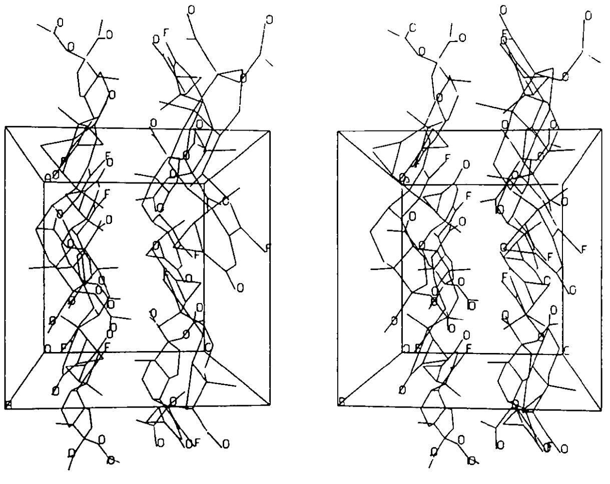

Fig. 2. Stereoview of the molecular packing in the unit cell viewed down the *b* axis.

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N-(4,6-Diméthylpyridin-2-yl)(thién-2-yl)carboxamide

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(*Reçu le* 23 *juillet* 1991, *accepté le* 23 *août* 1991)

Abstract. *N*-(4,6-Dimethyl-2-pyridyl)-2-thiophenecarboxamide, $C_{12}H_{12}N_2OS$, $M_r = 232.3$, triclinic, $P\bar{1}$, $a = 10.723$ (2), $b = 10.656$ (1), $c = 15.210$ (3) Å, $\alpha = 88.51$ (1), $\beta = 120.46$ (1), $\gamma = 123.41$ (1)°, $V = 1171$ (1) Å^3, $Z = 4$, $D_x = 1.318$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 2.24$ mm^{-1}, $F(000) = 488$, $T = 294$ (1) K, $R = 0.038$ for 3269 independent observed reflections. Molecules (1) and (2) in the asymmetric unit have similar geometries and are almost planar. However, the thienyl ring of (2) occupies two positions approximately rotated by 180° around the C(28)—C(29) bond, the larger occupancy factor being equal to 0.915 (2). In each molecule, there is an intramolecular hydrogen bond which forms a pseudo-six-membered ring: C(3)—H(3)···O(14) in (1) and C(23)—H(23)···O(34) in (2). Each molecule is also involved in three intermolecular hydrogen bonds: N(7)—H(7)···N(21$^{\rm i}$) [(i) $1 + x, y, z$], C(12)—H(12)···O(34) and N(27)—H(27)···N(1$^{\rm ii}$) [(ii) $-1 + x, y, z$]. The title compound exhibits a significant anti-inflammatory activity. An X-ray study was performed in order to compare its molecular geometry with those of other *N*-(4,6-dimethyl-2-pyridyl)arylcarboxamides.

Partie expérimentale. Dimensions approximatives du cristal: 0,17 × 0,20 × 0,35 mm. Dimensions de la maille déterminées avec 25 réflexions telles que $14{,}17 \le \theta \le 34{,}39°$. Diffractomètre Enraf–Nonius CAD-4. $0{,}023 \le (\sin\theta)/\lambda \le 0{,}573$ Å^{-1}, $0 \le h \le 12$, $-12 \le k$

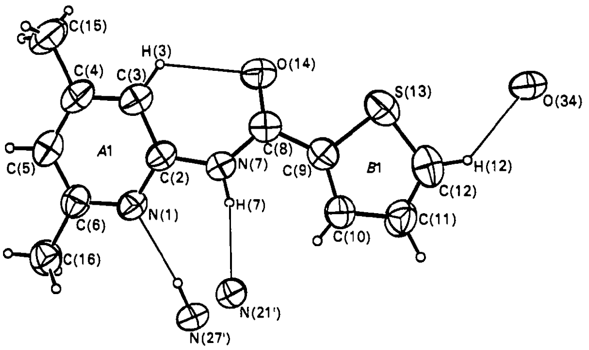

Fig. 1. Dessin de la molécule (1) et des liaisons hydrogène qu'elle réalise, numéros attribués aux atomes et lettres utilisées pour désigner les cycles.

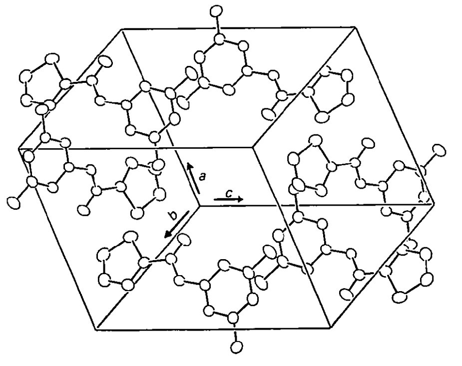

Fig. 2. Vue de la structure en perspective. Seule la position *a* du cycle thiényl de la molécule (2) a été représentée.

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