

ASD and DSW acknowledge support from the North Atlantic Treaty Organization (grant 880346 D2). We thank Dr M. Kaftory for useful discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1189). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 2437–2439

## 9 $\alpha$ -Fluoro-16 $\alpha$ -methyl-3,17-dioxoandrosta-1,4-dien-11 $\beta$ -yl 2-Butynoate

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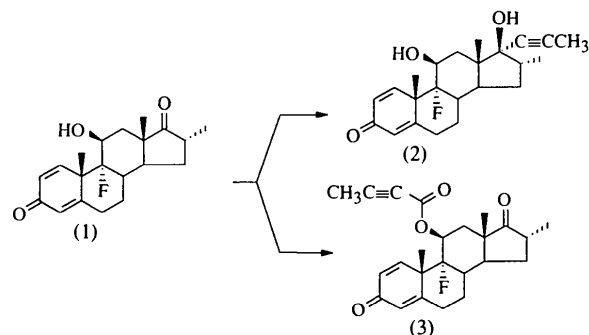
### Abstract

The structure of the title compound, C<sub>24</sub>H<sub>27</sub>FO<sub>4</sub>, which was an unexpected reaction product, was determined in order to establish its connectivity.

### Comment

The title compound was supposedly produced in a reaction in which the lithium salt of propyne was added to 9 $\alpha$ -fluoro-11 $\beta$ -hydroxy-16 $\alpha$ -methylandrosta-1,4-diene-3,17-dione, (1), in order to obtain the expected adduct 9 $\alpha$ -fluoro-11 $\beta$ ,17 $\beta$ -dihydroxy-16 $\alpha$ -methyl-17 $\alpha$ -propynylandrosta-1,4-diene-3-one, (2). Instead, com-

pound (3) was obtained unexpectedly. Efforts to identify the source of the one-carbon unit that comprises the carbonyl of the ester in (3) (*e.g.* adventitious DMF, *etc.*) proved uninformative. The mode of synthesis of (3) remains unexplained.



The combination of the rather flat steroid ring structure and the nearly cylindrical butynoate substituent might have been expected to be unfavorable for crystal packing, especially since the cylinder axis must make a fairly small angle with the plane normal. The displacement ellipsoids and the melting point, however, are both normal. The space group is common and there is one molecule in the asymmetric unit. The packing diagrams (Figs. 2 and 3) show that the butynoate substituents are aligned in columns by the translation along *a*. Columns fit together such that the butynoate stacks fit into cavities between steroid groups. The *a* axis is the morphological axis of the very long needles and there are fairly short (Desiraju, 1991) C—H...O=C distances in molecules related by this translation [C6...O1<sup>i</sup> 3.335 (4), C19...O1<sup>i</sup> 3.450 (4) and C24...O4<sup>i</sup> 3.627 (4) Å; symmetry code: (i) *x* − 1, *y*, *z*].

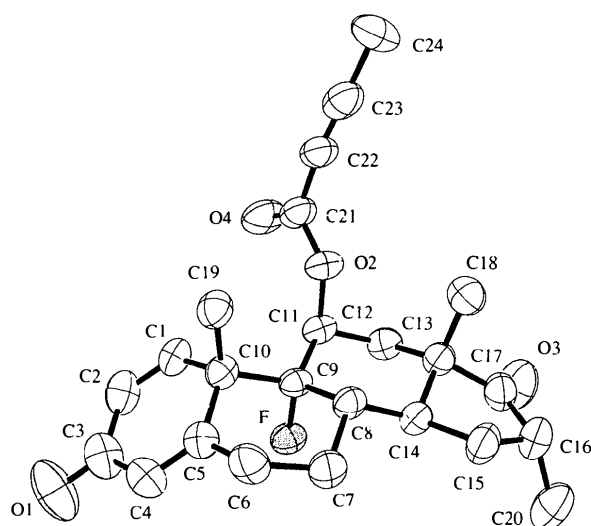


Fig. 1. Perspective drawing of the title molecule showing the atom-numbering scheme. The shapes of the ellipsoids correspond to 50% probability contours of atomic displacement. The H atoms have been omitted for clarity.

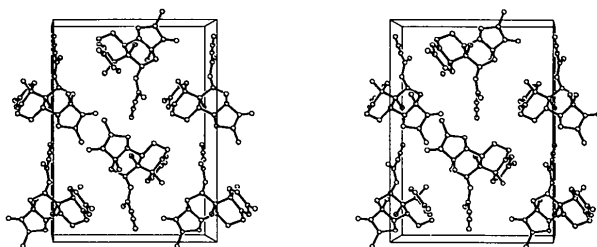


Fig. 2. Stereoscopic drawing of the unit cell. The *b* axis points from left to right, the *c* axis points upwards and the *a* axis points out of the plane of the paper.

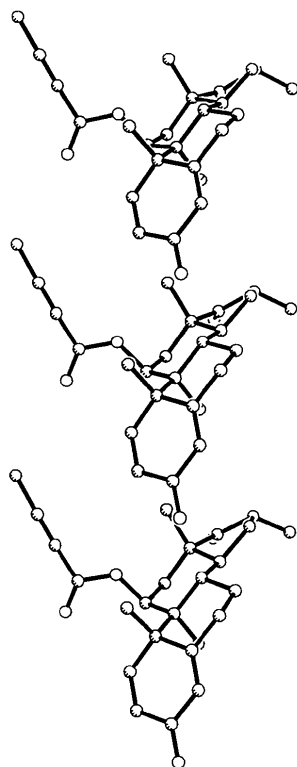


Fig. 3. Drawing showing a group of three molecules related by translation along the *a* axis.

## Experimental

The title compound was synthesized by J. Song. Crystals were grown by slow evaporation from methanol solution.

### Crystal data

C<sub>24</sub>H<sub>27</sub>FO<sub>4</sub>  
*M<sub>r</sub>* = 398.47  
 Orthorhombic  
*P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 7.6922 (5) Å  
*b* = 14.1688 (8) Å  
*c* = 19.3663 (11) Å

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 22 reflections  
 $\theta$  = 12.2–12.9°  
 $\mu$  = 0.084 mm<sup>-1</sup>  
*T* = 295 (2) K

*V* = 2110.7 (2) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.254 Mg m<sup>-3</sup>

Needle (major faces are {011}, {012}, {110}, {101} and {010})  
 0.46 × 0.40 × 0.32 mm  
 Colorless

### Data collection

Enraf–Nonius CAD-4-VAX diffractometer

$\omega/2\theta$  scans

Absorption correction: none

5140 measured reflections

2752 independent reflections

1698 observed reflections

[*I* > 2σ(*I*)]

*R*<sub>int</sub> = 0.0359

$\theta_{\max}$  = 27.50°

*h* = -9 → 9

*k* = 0 → 18

*l* = -25 → 25

3 standard reflections

frequency: 60 min

intensity decay: <0.5%

### Refinement

Refinement on *F*<sup>2</sup>

*R*(*F*) = 0.0387

*wR*(*F*<sup>2</sup>) = 0.1065

*S* = 1.000

2751 reflections

262 parameters

H atoms refined as riding

$w = 1/[\sigma^2(F_o^2) + (0.0630P)^2 + 0.0117P]$

where  $P = (F_o^2 + 2F_c^2)/3$

( $\Delta/\sigma$ )<sub>max</sub> = 0.005

$\Delta\rho_{\max} = 0.131 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.156 \text{ e \AA}^{-3}$

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Absolute configuration:

assigned on the basis of

the synthetic pathway

[Flack (1983) parameter

= -2.0 (12)]

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
F	0.0479 (2)	0.49747 (10)	0.88512 (7)	0.0495 (4)
O1	0.4800 (3)	0.2633 (2)	0.8494 (2)	0.1043 (9)
O2	-0.2427 (2)	0.51621 (12)	0.74121 (8)	0.0509 (5)
O3	-0.3621 (4)	0.77699 (14)	0.91548 (12)	0.0882 (8)
O4	-0.0710 (3)	0.5557 (2)	0.65196 (10)	0.0775 (7)
C1	0.1462 (3)	0.3913 (2)	0.76742 (14)	0.0533 (7)
C2	0.3014 (4)	0.3569 (2)	0.7791 (2)	0.0602 (8)
C3	0.3362 (4)	0.2957 (2)	0.8378 (2)	0.0659 (8)
C4	0.1885 (4)	0.2736 (2)	0.8818 (2)	0.0586 (7)
C5	0.0290 (4)	0.3058 (2)	0.87063 (13)	0.0473 (6)
C6	-0.1213 (4)	0.2816 (2)	0.91570 (14)	0.0572 (7)
C7	-0.2070 (4)	0.3705 (2)	0.94403 (13)	0.0545 (7)
C8	-0.2456 (3)	0.4450 (2)	0.88888 (12)	0.0424 (6)
C9	-0.0877 (3)	0.4628 (2)	0.84219 (11)	0.0397 (5)
C10	-0.0123 (3)	0.3702 (2)	0.81012 (12)	0.0443 (6)
C11	-0.1150 (3)	0.5455 (2)	0.79208 (11)	0.0443 (6)
C12	-0.1836 (4)	0.6357 (2)	0.82637 (13)	0.0484 (6)
C13	-0.3429 (4)	0.6166 (2)	0.87086 (12)	0.0467 (6)
C14	-0.2997 (4)	0.5380 (2)	0.92242 (11)	0.0449 (6)
C15	-0.4502 (4)	0.5406 (2)	0.97407 (14)	0.0595 (7)
C16	-0.4795 (4)	0.6473 (2)	0.98427 (14)	0.0631 (8)
C17	-0.3921 (4)	0.6935 (2)	0.92182 (15)	0.0591 (7)
C18	-0.5110 (4)	0.5996 (2)	0.82879 (15)	0.0612 (7)
C19	-0.1416 (4)	0.3177 (2)	0.76234 (13)	0.0543 (7)
C20	-0.4060 (5)	0.6862 (3)	1.05119 (15)	0.0862 (11)
C21	-0.2098 (4)	0.5323 (2)	0.67401 (12)	0.0524 (7)
C22	-0.3651 (4)	0.5174 (2)	0.63436 (13)	0.0589 (8)
C23	-0.4975 (5)	0.5116 (2)	0.60384 (13)	0.0628 (8)
C24	-0.6617 (4)	0.5063 (3)	0.5663 (2)	0.0843 (10)

Table 2. Selected geometric parameters (Å, °)

F—C9	1.421 (3)	C8—C9	1.535 (3)
O1—C3	1.219 (4)	C9—C11	1.536 (3)
O2—C21	1.345 (3)	C9—C10	1.563 (3)
O2—C11	1.452 (3)	C10—C19	1.549 (4)
O3—C17	1.212 (3)	C11—C12	1.534 (3)
O4—C21	1.197 (4)	C12—C13	1.522 (4)
C1—C2	1.310 (4)	C13—C17	1.517 (4)
C1—C10	1.503 (3)	C13—C14	1.533 (3)
C2—C3	1.454 (5)	C13—C18	1.547 (4)
C3—C4	1.454 (4)	C14—C15	1.530 (3)
C4—C5	1.326 (4)	C15—C16	1.542 (4)
C5—C6	1.489 (4)	C16—C20	1.517 (4)
C5—C10	1.519 (3)	C16—C17	1.531 (4)
C6—C7	1.524 (4)	C21—C22	1.436 (4)
C7—C8	1.531 (3)	C22—C23	1.180 (4)
C8—C14	1.526 (3)	C23—C24	1.459 (4)
C21—O2—C11	118.8 (2)	C19—C10—C9	113.7 (2)
C2—C1—C10	124.7 (3)	O2—C11—C12	107.5 (2)
C1—C2—C3	121.7 (3)	O2—C11—C9	107.6 (2)
O1—C3—C2	122.3 (3)	C12—C11—C9	114.2 (2)
O1—C3—C4	121.4 (3)	C13—C12—C11	112.0 (2)
C2—C3—C4	116.3 (3)	C17—C13—C12	116.2 (2)
C5—C4—C3	123.6 (3)	C17—C13—C14	98.8 (2)
C4—C5—C6	122.9 (2)	C12—C13—C14	108.9 (2)
C4—C5—C10	121.7 (3)	C17—C13—C18	104.2 (2)
C6—C5—C10	115.4 (2)	C12—C13—C18	113.7 (2)
C5—C6—C7	110.9 (2)	C14—C13—C18	114.2 (2)
C6—C7—C8	113.8 (2)	C8—C14—C15	120.3 (2)
C14—C8—C7	110.6 (2)	C8—C14—C13	114.2 (2)
C14—C8—C9	109.0 (2)	C15—C14—C13	104.1 (2)
C7—C8—C9	111.7 (2)	C14—C15—C16	102.6 (2)
F—C9—C8	107.0 (2)	C20—C16—C17	110.9 (3)
F—C9—C11	101.9 (2)	C20—C16—C15	114.3 (3)
C8—C9—C11	112.9 (2)	C17—C16—C15	104.7 (2)
F—C9—C10	104.5 (2)	O3—C17—C13	126.0 (3)
C8—C9—C10	113.0 (2)	O3—C17—C16	125.5 (3)
C11—C9—C10	116.1 (2)	C13—C17—C16	108.5 (2)
C1—C10—C5	112.0 (2)	O4—C21—O2	124.0 (3)
C1—C10—C19	106.7 (2)	O4—C21—C22	126.3 (2)
C5—C10—C19	107.9 (2)	O2—C21—C22	109.6 (3)
C1—C10—C9	110.6 (2)	C23—C22—C21	174.8 (3)
C5—C10—C9	106.0 (2)	C22—C23—C24	179.0 (3)

Data collection: CAD-4-VAX diffractometer software (Enraf-Nonius, 1988). Cell refinement: CAD-4-VAX diffractometer software. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

JS acknowledges support from Apollo Genetics Inc., Cambridge, MA, USA.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1188). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 2439–2441

## 2-[2-(2-Ethyl-2,3-dihydrobenzofuranyl)]-2-imidazoline Hydrobromide

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(Received 27 February 1995; accepted 18 May 1995)

## Abstract

The isomorphous structures of 2-[2-(2-ethyl-2,3-dihydro-2-benzofuranyl)]-2-imidazolium [(+)-efaroxan cation] chloride,  $C_{13}H_{17}N_2O^+ \cdot Cl^-$ , and bromide,  $C_{13}H_{17}N_2O^+ \cdot Br^-$ , have been determined. The absolute configuration of the active molecule (efaroxan) could be resolved only in the hydrobromide salt, the structure of which is reported. (+)-Efaroxan has the *R* configuration.

## Comment

Efaroxan { $(\pm)$ -2-[2-(2-ethyl-2,3-dihydro-2-benzofuranyl)]-2-imidazoline (CAS Registry Number 99197–32–0)} is a potent and highly selective  $\alpha$ -2-adrenoreceptor antagonist. Efaroxan has one asymmetric C atom on the dihydrobenzofuranyl ring [C(2)] and therefore exhibits two enantiomers. It has long been recognized that many receptor systems are highly isomerically selective and compounds possessing a chiral centre should be resolved so that the configuration of the active isomer may be established. The *R* configuration of the molecule of the title compound, (I), is shown in Fig. 1 and the crystal packing of the structure is represented in Fig. 2.

