used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *O* (Jones & Kjeldgaard, 1993). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1191). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl 7α,12α-Dihydroxy-3α-methacryloylamino-5β-cholan-24-oate

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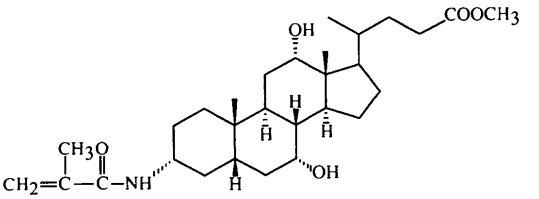
Abstract

In the title compound, $C_{29}H_{47}NO_5$, the methacryloylamino group is confirmed to be attached on position 3 of the steroid skeleton of cholic acid methyl ester as a 3α-epimer. The crystal structure of the title compound, recrystallized from acetone, contains two molecules in the asymmetric unit and is stabilized by intermolecular hydrogen bonds.

Comment

Natural compounds are used in the preparation of organic polymer materials with biomedical and pharmaceutical applications. The title compound, (I), was one of the epimers synthesized as a monomer in the preparation of such polymers (Denike & Zhu, 1994). It was readily polymerized by a free-radical polymerization in solution, despite the presence of the bulky cholic acid side group (Denike & Zhu, 1994). It also copolymerized easily with methacrylic monomers (Zhu, Moskova & Denike, 1996). The title compound can be recrystallized from a variety of solvents such as acetone, methanol or toluene. The compound crystallized from acetone was used for this X-ray diffraction study as part of an attempt to elucidate the characteristics of polymers prepared from the title compound and other related methacrylic monomers.



(I)

The title compound crystallizes in the monoclinic space group $P2_1$, with two molecules in an asymmetric unit (Figs. 1 and 2). Molecules 1 (C1–C29) and 2 (C31–C59/C59′) have the same overall configuration for the principal molecular framework, but the methacryloylamino group attached in an α configuration at position 3 of the steroid skeleton differs with respect to the orientation of the methacryloyl side chain. If the torsion angles C*m*2—C*m*3—N*mp*—C*nq* are compared, we find −111.7 (2)° for molecule 1 (m = 0, n = 2, p = 3 and q = 6), −123.4 (17)° for molecule 2*A* (m = 3, n = 5, p = 3 and q = 6) and −125.0 (14)° for molecule 2*B* (m = 3, n = 5, p = 3′ and q = 6′). In molecule 2, we find that the methacryloylamino group could not be described without the introduction of a disorder model, therefore, two sets of atoms were defined for this group for molecule 2. The occupancy in both molecules (2*A* and 2*B*) was initially refined and the occupancy factor fixed at 0.50 for each in the final cycles of refinement.

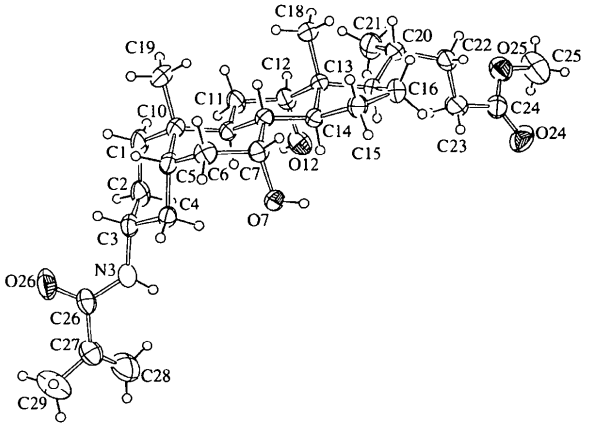


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of molecule 1 of (I). Ellipsoids correspond to the 40% probability level and H atoms are represented by spheres of arbitrary size.

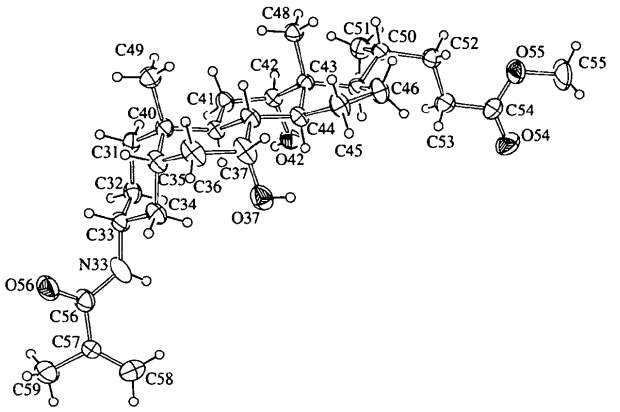


Fig. 2. *ORTEP*II (Johnson, 1976) drawing of molecule 2*A* of (I). Ellipsoids correspond to the 40% probability level. The site-occupancy factor of the methacryloylamino group is 0.50. H atoms are represented by spheres of arbitrary size.

The α configuration of the methacryloylamino group attached at position 3 of the steroid skeleton has been confirmed unambiguously by this X-ray analysis. The crystal structure is stabilized by intermolecular hydrogen bonds (Table 3). Molecules 1 and 2 are linked by intermolecular hydrogen bonds of the O—H···O type (O7···O56 and O42···O26). Molecules of the same kind (two 1 molecules or two 2 molecules) are linked by intermolecular hydrogen bonds of the N—H···O type (N3···O24, N33···O54 and N33′···O54).

Experimental

The title compound was prepared according to the method described by Denike & Zhu (1994). Single crystals were grown from an acetone solution.

Crystal data

$C_{29}H_{47}NO_5$
M_r = 489.70
Monoclinic
$P2_1$
a = 12.303 (2) Å
b = 18.668 (4) Å
c = 13.090 (2) Å
β = 112.81 (2)°
V = 2771.3 (9) Å^3
Z = 4
D_x = 1.174 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 20–22°
μ = 0.59 mm^{-1}
T = 293 (2) K
Parallelepiped
0.38 × 0.27 × 0.18 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
20 602 measured reflections
10 486 independent reflections
8526 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.018
θ_{max} = 69.83°
h = −14 → 14
k = −22 → 22
l = −15 → 14
4 standard reflections
frequency: 30 min
intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0381
$wR(F^2)$ = 0.0896
S = 0.942
10 486 reflections
700 parameters
H-atom parameters riding, C—H 0.96–0.98, N—H 0.86 and O—H 0.82 Å
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.307
$\Delta\rho_{max}$ = 0.113 e Å^{-3}
$\Delta\rho_{min}$ = −0.135 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.10 (13)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	U_{eq}
C1	0.2470 (2)	0.36362 (12)	0.8569 (2)	0.0640 (6)
C2	0.2469 (2)	0.31222 (12)	0.7672 (2)	0.0622 (6)
C3	0.3397 (2)	0.25441 (11)	0.8185 (2)	0.0518 (5)
C4	0.4602 (2)	0.28909 (10)	0.8739 (2)	0.0444 (4)
C5	0.4622 (2)	0.34248 (10)	0.9636 (2)	0.0491 (5)
C6	0.5847 (2)	0.37488 (11)	1.0252 (2)	0.0548 (5)
O7	0.64644 (11)	0.40003 (7)	0.87638 (11)	0.0515 (3)
C7	0.6192 (2)	0.43270 (10)	0.9624 (2)	0.0467 (4)
C8	0.5223 (2)	0.48961 (10)	0.9159 (2)	0.0424 (4)
C9	0.40117 (15)	0.45514 (10)	0.8497 (2)	0.0436 (4)
C10	0.3663 (2)	0.40085 (11)	0.9218 (2)	0.0511 (5)
C11	0.3042 (2)	0.51177 (11)	0.7947 (2)	0.0576 (5)
O12	0.34158 (13)	0.54447 (8)	0.63202 (12)	0.0656 (4)
C12	0.3375 (2)	0.57189 (10)	0.7331 (2)	0.0527 (5)
C13	0.4547 (2)	0.60653 (10)	0.8062 (2)	0.0434 (4)
C14	0.54848 (15)	0.54638 (9)	0.8452 (2)	0.0416 (4)
C15	0.6656 (2)	0.58684 (11)	0.8953 (2)	0.0532 (5)
C16	0.6441 (2)	0.65566 (12)	0.8251 (2)	0.0603 (5)
C17	0.5116 (2)	0.65792 (10)	0.7482 (2)	0.0468 (4)
C18	0.4404 (2)	0.64505 (11)	0.9043 (2)	0.0589 (5)
C19	0.3532 (2)	0.43753 (14)	1.0216 (2)	0.0773 (7)
C20	0.4640 (2)	0.73519 (11)	0.7229 (2)	0.0551 (5)
C21	0.3317 (2)	0.73977 (13)	0.6533 (2)	0.0795 (7)
C22	0.5313 (2)	0.77903 (11)	0.6679 (2)	0.0619 (6)
C23	0.5223 (3)	0.74984 (13)	0.5559 (2)	0.0769 (7)
O24	0.5758 (2)	0.79334 (11)	0.41124 (13)	0.0882 (6)
C24	0.5272 (2)	0.80287 (14)	0.4737 (2)	0.0638 (6)
O25	0.4672 (2)	0.86189 (10)	0.47392 (13)	0.0763 (5)
C25	0.4586 (3)	0.9156 (2)	0.3917 (2)	0.1081 (11)
N3	0.34066 (15)	0.20074 (9)	0.73855 (14)	0.0560 (4)
O26	0.2696 (2)	0.11085 (9)	0.80866 (15)	0.0898 (6)
C26	0.3072 (2)	0.13265 (12)	0.7404 (2)	0.0600 (6)
C27	0.3200 (2)	0.08343 (13)	0.6556 (2)	0.0710 (6)
C28	0.3135 (3)	0.1072 (2)	0.5569 (3)	0.1096 (11)
C29	0.3373 (3)	0.0082 (2)	0.6869 (3)	0.1202 (12)
C31	0.9659 (2)	0.21065 (10)	0.9687 (2)	0.0490 (5)
C32	0.9910 (2)	0.26228 (10)	0.8911 (2)	0.0479 (5)
C33	0.8815 (2)	0.30627 (10)	0.8303 (2)	0.0479 (4)
C34	0.7803 (2)	0.25717 (10)	0.7643 (2)	0.0519 (5)
C35	0.7532 (2)	0.20294 (10)	0.8386 (2)	0.0515 (5)
C36	0.6485 (2)	0.15506 (11)	0.7712 (2)	0.0640 (6)
O37	0.69091 (14)	0.12461 (8)	0.61144 (13)	0.0675 (4)
C37	0.6767 (2)	0.09559 (11)	0.7067 (2)	0.0600 (6)
C38	0.7848 (2)	0.05261 (9)	0.7808 (2)	0.0446 (4)
C39	0.89168 (15)	0.10186 (9)	0.84084 (15)	0.0378 (4)

C40	0.8625 (2)	0.15911 (10)	0.9129 (2)	0.0442 (4)
C41	1.0043 (2)	0.05884 (10)	0.9052 (2)	0.0444 (4)
O42	1.06721 (11)	0.01886 (7)	0.75851 (10)	0.0497 (3)
C42	1.03115 (14)	−0.00431 (9)	0.84474 (14)	0.0391 (4)
C43	0.92274 (14)	−0.05294 (9)	0.79228 (14)	0.0363 (4)
C44	0.81936 (14)	−0.00542 (9)	0.7185 (2)	0.0414 (4)
C45	0.7247 (2)	−0.05864 (10)	0.6521 (2)	0.0569 (5)
C46	0.7936 (2)	−0.12262 (11)	0.6342 (2)	0.0553 (5)
C47	0.92603 (15)	−0.10864 (9)	0.70572 (14)	0.0390 (4)
C48	0.8980 (2)	−0.09010 (10)	0.8856 (2)	0.0497 (5)
C49	0.8356 (2)	0.12365 (12)	1.0076 (2)	0.0687 (6)
C50	0.9936 (2)	−0.17907 (9)	0.74734 (15)	0.0428 (4)
C51	1.1236 (2)	−0.16722 (11)	0.8173 (2)	0.0556 (5)
C52	0.9793 (2)	−0.23170 (10)	0.6529 (2)	0.0508 (5)
C53	1.0189 (2)	−0.20255 (12)	0.5654 (2)	0.0596 (5)
O54	1.0509 (2)	−0.23709 (10)	0.40448 (13)	0.0785 (5)
C54	1.0307 (2)	−0.25461 (13)	0.4836 (2)	0.0557 (5)
O55	1.0190 (2)	−0.32143 (9)	0.50840 (15)	0.0813 (5)
C55	1.0300 (3)	−0.3755 (2)	0.4327 (3)	0.1136 (12)
N33†	0.8999 (15)	0.3570 (6)	0.7560 (12)	0.066 (4)
O56†	0.8430 (13)	0.4551 (8)	0.8199 (13)	0.081 (4)
C56†	0.8845 (19)	0.4287 (8)	0.7569 (15)	0.056 (4)
C57†	0.9077 (19)	0.4731 (6)	0.6734 (11)	0.054 (3)
C58†	0.8972 (12)	0.4497 (7)	0.5744 (8)	0.118 (5)
C59†	0.9350 (17)	0.5482 (7)	0.7089 (10)	0.092 (4)
N33′†	0.9032 (14)	0.3633 (6)	0.7613 (9)	0.048 (3)
O56′†	0.8457 (10)	0.4537 (7)	0.8451 (12)	0.057 (2)
C56′†	0.8796 (16)	0.4316 (6)	0.7732 (14)	0.043 (3)
C57′†	0.9119 (19)	0.4835 (7)	0.7022 (11)	0.056 (3)
C58′†	0.9053 (19)	0.5528 (8)	0.7178 (15)	0.119 (6)
C59′†	0.9495 (11)	0.4527 (6)	0.6145 (7)	0.093 (3)

† Site occupancy = 0.50.

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.516 (3)	C33—N33′	1.485 (9)
C2—C3	1.524 (3)	C33—C34	1.517 (3)
C3—N3	1.453 (3)	C34—C35	1.528 (3)
C3—C4	1.520 (3)	C35—C40	1.552 (3)
C4—C5	1.533 (3)	N33—C56	1.352 (13)
C5—C10	1.543 (3)	O56—C56	1.229 (13)
N3—C26	1.339 (3)	C56—C57	1.485 (12)
O26—C26	1.224 (2)	C57—C58	1.326 (13)
C26—C27	1.496 (3)	C57—C59	1.474 (13)
C27—C28	1.339 (4)	N33′—C56′	1.330 (12)
C27—C29	1.455 (4)	O56′—C56′	1.239 (13)
C31—C32	1.517 (3)	C56′—C57′	1.499 (12)
C32—C33	1.514 (3)	C57′—C58′	1.32 (2)
C33—N33	1.438 (11)	C57′—C59′	1.508 (14)
N3—C3—C4	111.0 (2)	C56—N33—C33	125.9 (10)
N3—C3—C2	112.6 (2)	O56—C56—N33	119.9 (11)
C26—N3—C3	123.6 (2)	N33—C56—C57	118.6 (11)
O26—C26—N3	122.3 (2)	C58—C57—C56	124.2 (11)
N3—C26—C27	116.6 (2)	C59—C57—C56	112.6 (10)
C28—C27—C26	121.9 (2)	C56′—N33′—C33	121.1 (7)
C29—C27—C26	115.4 (2)	O56′—C56′—N33′	124.2 (10)
N33—C33—C32	111.8 (6)	N33′—C56′—C57′	115.1 (10)
N33′—C33—C32	112.0 (6)	C58′—C57′—C56′	119.5 (12)
N33—C33—C34	108.8 (7)	C56′—C57′—C59′	117.3 (10)
N33′—C33—C34	112.8 (6)		

C1—C2—C3—N3	176.9 (2)
N3—C3—C4—C5	−176.5 (2)
C4—C3—N3—C26	125.1 (2)
C2—C3—N3—C26	−111.7 (2)
C3—N3—C26—O26	2.1 (3)
C3—N3—C26—C27	−176.7 (2)
N3—C26—C27—C28	−28.8 (4)
N3—C26—C27—C29	152.5 (3)
C31—C32—C33—N33	179.3 (6)
C31—C32—C33—N33′	174.1 (5)
N33—C33—C34—C35	−178.4 (5)
N33′—C33—C34—C35	−175.3 (5)
C32—C33—N33—C56	−123.4 (17)
C34—C33—N33—C56	115.2 (18)
C33—N33—C56—O56	−6 (3)
C33—N33—C56—C57	−179.9 (16)
N33—C56—C57—C58	28 (3)
N33—C56—C57—C59	−156.7 (19)
C32—C33—N33′—C56′	−125.0 (14)
C34—C33—N33′—C56′	110.6 (14)
C33—N33′—C56′—O56′	5 (3)
C33—N33′—C56′—C57′	176.9 (14)
N33′—C56′—C57′—C58′	−171.8 (19)
N33′—C56′—C57′—C59′	9 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O24^{i}	0.86	2.275 (2)	3.074 (2)	154.59 (6)
N33—H33···O54^{ii}	0.86	2.160 (12)	2.975 (12)	158.2 (3)
N33′—H33′···O54^{ii}	0.86	2.329 (10)	3.081 (10)	146.1 (3)
O7—H7···O56	0.82	2.176 (13)	2.973 (13)	164.1 (7)
O42—H42···O26^{iii}	0.82	2.086 (3)	2.883 (2)	164.1 (7)

Symmetry codes: (i) $1-x, y-\frac{1}{2}, 1-z$; (ii) $2-x, \frac{1}{2}+y, 1-z$; (iii) $1+x, y, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRC-2* and *NRC-2A* (Ahmed, Hall, Pippy & Huber, 1973). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1198). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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