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Crystal structure of (3*S*,8*R*,10*R*,12*R*,14*R*)-12-hydroxy-4,4,8,10,14-pentamethyl-17-((*R*)-2,6,6-trimethyltetrahydro-2*H*-pyran-2-yl)hexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl acetate, C₃₂H₅₄O₄

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Abstract

C₃₂H₅₄O₄, monoclinic, *P*2₁ (no. 4), *a* = 7.8727(1) Å, *b* = 19.9173(2) Å, *c* = 19.1801(2) Å, β = 94.539(1)°, *V* = 2998.06(6) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0370, *wR*_{ref}(*F*²) = 0.0983, *T* = 293 K.

CCDC no.: 1858883

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.10 × 0.10 mm
Wavelength:	Cu Kα radiation (1.54178 Å)
μ:	0.55 mm ⁻¹
Diffraction, scan mode	Xcalibur
θ _{max} , completeness:	66.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	43,442, 10,562, 0.029
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 9800
<i>N</i> (<i>param</i>) _{refined} :	669
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3], SUPERFLIP [4]

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Source of material

A solution of 20(*R*)-panoxadiol and acetic anhydride in pyridine was stirred for 11 h at room temperature. After evaporation under reduced pressure, the residue was dissolved in ethyl acetate and washed successively by water and brine, and dried over anhydrous sodium sulfate. After filtration, the ethyl acetate was removed *in vacuo* to yield a white solid, which was purified by silica-gel column chromatography (petroleum ether:ethyl acetate = 1:2 v/v). Suitable crystals were obtained by slow evaporation in ethyl acetate.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.1680 (3)	0.06139 (12)	1.00958 (12)	0.0476 (5)
H1A	0.113929	0.104685	1.001042	0.057*
H1B	0.128413	0.031685	0.971645	0.057*
C2	0.1130 (3)	0.03301 (14)	1.07813 (13)	0.0548 (6)
H2A	0.144863	0.063853	1.116069	0.066*
H2B	−0.009825	0.027675	1.075041	0.066*
C3	0.1977 (3)	−0.03412 (14)	1.09272 (12)	0.0527 (6)
H3	0.164856	−0.063883	1.053250	0.063*
C4	0.3925 (3)	−0.03125 (14)	1.10138 (13)	0.0528 (5)
C5	0.4468 (3)	0.00180 (12)	1.03268 (12)	0.0450 (5)
H5	0.407261	−0.029844	0.995772	0.054*
C6	0.6404 (3)	0.00520 (14)	1.02797 (14)	0.0562 (6)
H6A	0.687306	0.041741	1.056864	0.067*
H6B	0.692225	−0.036298	1.045489	0.067*
C7	0.6818 (3)	0.01612 (14)	0.95263 (14)	0.0561 (6)
H7A	0.644159	−0.022801	0.925199	0.067*
H7B	0.804491	0.019410	0.951400	0.067*
C8	0.5995 (3)	0.07922 (12)	0.91843 (12)	0.0446 (5)
C9	0.4061 (2)	0.08137 (11)	0.93185 (11)	0.0405 (4)
H9	0.356250	0.042679	0.906148	0.049*
C10	0.3620 (2)	0.06991 (11)	1.00917 (11)	0.0406 (4)
C11	0.3197 (3)	0.14259 (13)	0.89570 (13)	0.0493 (5)
H11A	0.199058	0.141260	0.902691	0.059*
H11B	0.366360	0.182958	0.918098	0.059*
C12	0.3404 (3)	0.14725 (13)	0.81729 (13)	0.0523 (6)
H12	0.276463	0.110686	0.793188	0.063*
C13	0.5277 (3)	0.14166 (12)	0.80294 (12)	0.0471 (5)
H13	0.586437	0.179902	0.826215	0.057*
C14	0.6088 (3)	0.07720 (12)	0.83671 (12)	0.0477 (5)
C15	0.7886 (4)	0.08003 (18)	0.81006 (16)	0.0702 (7)
H15A	0.863448	0.107885	0.840479	0.084*
H15B	0.837235	0.035353	0.808528	0.084*
C16	0.7659 (4)	0.10999 (16)	0.73718 (16)	0.0668 (7)
H16A	0.780626	0.075611	0.702340	0.080*
H16B	0.849756	0.144960	0.732001	0.080*
C17	0.5829 (3)	0.13972 (13)	0.72739 (13)	0.0536 (6)
H17	0.511239	0.106227	0.701817	0.064*
C18	0.5734 (3)	0.20449 (13)	0.68316 (13)	0.0554 (6)
C19	0.6497 (4)	0.19132 (18)	0.61326 (16)	0.0734 (8)
H19A	0.772965	0.190965	0.620806	0.088*
H19B	0.613637	0.147368	0.596053	0.088*
C20	0.5960 (5)	0.2440 (2)	0.55813 (17)	0.0897 (11)
H20A	0.639933	0.287572	0.573234	0.108*
H20B	0.643467	0.232778	0.514453	0.108*
C21	0.4042 (5)	0.2470 (3)	0.54697 (17)	0.0940 (11)
H21A	0.361828	0.204545	0.527856	0.113*
H21B	0.371914	0.281861	0.513175	0.113*
C22	0.3219 (4)	0.2613 (2)	0.61478 (15)	0.0772 (9)
C23	0.0015 (3)	−0.10468 (13)	1.14579 (13)	0.0539 (6)
C24	−0.0582 (4)	−0.12987 (18)	1.21271 (16)	0.0732 (8)
H24A	−0.135615	−0.097974	1.230349	0.110*
H24B	0.037755	−0.135802	1.246235	0.110*
H24C	−0.115384	−0.172073	1.204715	0.110*
C25	0.4579 (4)	0.00528 (18)	1.16905 (14)	0.0695 (8)
H25A	0.405104	0.048641	1.170555	0.104*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H25B	0.579280	0.010579	1.169955	0.104*
H25C	0.430186	−0.020628	1.208800	0.104*
C26	0.4579 (4)	−0.10434 (17)	1.10499 (19)	0.0763 (8)
H26A	0.405298	−0.127976	1.141222	0.114*
H26B	0.579307	−0.104382	1.115081	0.114*
H26C	0.429813	−0.126097	1.060926	0.114*
C27	0.4149 (3)	0.12945 (13)	1.05750 (13)	0.0546 (6)
H27A	0.399706	0.170695	1.031857	0.082*
H27B	0.532460	0.124732	1.074271	0.082*
H27C	0.345493	0.129998	1.096447	0.082*
C28	0.6985 (3)	0.14067 (14)	0.94981 (14)	0.0581 (6)
H28A	0.725268	0.133750	0.998981	0.087*
H28B	0.629617	0.180239	0.942787	0.087*
H28C	0.802005	0.146155	0.927119	0.087*
C29	0.5223 (4)	0.01358 (13)	0.80307 (14)	0.0618 (6)
H29A	0.529415	0.014796	0.753357	0.093*
H29B	0.404757	0.012455	0.813127	0.093*
H29C	0.579145	−0.025808	0.821970	0.093*
C30	0.6610 (4)	0.26291 (16)	0.72245 (17)	0.0732 (8)
H30A	0.678400	0.298778	0.690339	0.110*
H30B	0.769108	0.248359	0.743899	0.110*
H30C	0.590957	0.278441	0.757886	0.110*
C31	0.3453 (6)	0.3348 (2)	0.6367 (2)	0.0997 (12)
H31A	0.308354	0.340797	0.682791	0.149*
H31B	0.278810	0.362943	0.604337	0.149*
H31C	0.463405	0.346781	0.636673	0.149*
C32	0.1343 (5)	0.2444 (3)	0.6084 (2)	0.1093 (15)
H32A	0.119854	0.197573	0.597584	0.164*
H32B	0.077394	0.270803	0.571793	0.164*
H32C	0.086520	0.253981	0.651848	0.164*
C33	0.3862 (3)	0.02020 (12)	0.43299 (13)	0.0492 (5)
H33A	0.432325	0.014825	0.387902	0.059*
H33B	0.422059	0.063706	0.451509	0.059*
C34	0.4589 (3)	−0.03468 (13)	0.48254 (14)	0.0549 (6)
H34A	0.430258	−0.078358	0.462562	0.066*
H34B	0.582097	−0.030949	0.487785	0.066*
C35	0.3899 (3)	−0.02922 (12)	0.55297 (14)	0.0510 (5)
H35	0.428608	0.013445	0.574150	0.061*
C36	0.1955 (3)	−0.03220 (13)	0.55253 (14)	0.0530 (6)
C37	0.1224 (3)	0.02019 (12)	0.49728 (12)	0.0447 (5)
H37	0.160048	0.063750	0.516571	0.054*
C38	−0.0716 (3)	0.02421 (16)	0.49193 (14)	0.0603 (7)
H38A	−0.118791	−0.013725	0.465222	0.072*
H38B	−0.111895	0.021989	0.538380	0.072*
C39	−0.1325 (3)	0.08930 (15)	0.45654 (14)	0.0575 (6)
H39A	−0.093194	0.126796	0.485796	0.069*
H39B	−0.256126	0.089981	0.452874	0.069*
C40	−0.0701 (3)	0.09896 (11)	0.38303 (12)	0.0437 (5)
C41	0.1267 (2)	0.08521 (10)	0.38582 (11)	0.0377 (4)
H41	0.177704	0.120985	0.415664	0.045*
C42	0.1901 (3)	0.01832 (11)	0.42290 (12)	0.0416 (4)
C43	0.1949 (3)	0.09742 (11)	0.31429 (12)	0.0464 (5)
H43A	0.317338	0.090669	0.318395	0.056*
H43B	0.145523	0.064394	0.281495	0.056*
C44	0.1566 (3)	0.16761 (12)	0.28471 (12)	0.0456 (5)
H44	0.220993	0.200695	0.313951	0.055*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C45	−0.0326 (3)	0.18342 (10)	0.28441 (11)	0.0397 (4)
H45	−0.093108	0.150762	0.253326	0.048*
C46	−0.0968 (3)	0.17357 (12)	0.35823 (12)	0.0434 (5)
C47	−0.2832 (3)	0.19727 (16)	0.34474 (15)	0.0638 (7)
H47A	−0.353530	0.162176	0.322440	0.077*
H47B	−0.329890	0.209931	0.388157	0.077*
C48	−0.2737 (3)	0.25804 (14)	0.29629 (14)	0.0565 (6)
H48A	−0.280046	0.299321	0.322780	0.068*
H48B	−0.367796	0.257209	0.260471	0.068*
C49	−0.1011 (3)	0.25420 (11)	0.26218 (11)	0.0440 (5)
H49	−0.024715	0.287304	0.285936	0.053*
C50	−0.1189 (3)	0.27284 (11)	0.18404 (12)	0.0463 (5)
C51	−0.2034 (4)	0.34220 (13)	0.17510 (15)	0.0596 (6)
H51A	−0.324739	0.337512	0.179356	0.072*
H51B	−0.157751	0.371388	0.212506	0.072*
C52	−0.1761 (4)	0.37490 (15)	0.10539 (16)	0.0697 (8)
H52A	−0.233127	0.348803	0.067785	0.084*
H52B	−0.225017	0.419620	0.103946	0.084*
C53	0.0128 (4)	0.37919 (13)	0.09525 (16)	0.0646 (7)
H53A	0.067141	0.408966	0.130290	0.078*
H53B	0.027831	0.398270	0.049612	0.078*
C54	0.0993 (4)	0.31055 (12)	0.10086 (13)	0.0549 (6)
C55	0.6127 (3)	−0.07488 (15)	0.63096 (13)	0.0574 (6)
C56	0.6691 (5)	−0.1358 (2)	0.6713 (2)	0.0979 (12)
H56A	0.781282	−0.128563	0.693396	0.147*
H56B	0.671024	−0.173503	0.640143	0.147*
H56C	0.591403	−0.144699	0.706292	0.147*
C57	0.1288 (4)	−0.10390 (15)	0.53810 (19)	0.0766 (9)
H57A	0.170814	−0.132992	0.575481	0.115*
H57B	0.167566	−0.119740	0.494819	0.115*
H57C	0.006524	−0.103698	0.534985	0.115*
C58	0.1476 (4)	−0.0111 (2)	0.62530 (16)	0.0775 (9)
H58A	0.205933	−0.039268	0.660058	0.116*
H58B	0.026754	−0.015685	0.627675	0.116*
H58C	0.179944	0.034818	0.633746	0.116*
C59	0.1394 (4)	−0.04407 (12)	0.37884 (15)	0.0614 (7)
H59A	0.148762	−0.034323	0.330275	0.092*
H59B	0.024004	−0.056183	0.385883	0.092*
H59C	0.213848	−0.080654	0.392859	0.092*
C60	−0.1722 (3)	0.05077 (14)	0.33225 (16)	0.0620 (7)
H60A	−0.179008	0.007381	0.353645	0.093*
H60B	−0.115999	0.046807	0.289816	0.093*
H60C	−0.285023	0.068218	0.321782	0.093*
C61	−0.0090 (4)	0.22401 (13)	0.41058 (13)	0.0560 (6)
H61A	0.109468	0.212728	0.418791	0.084*
H61B	−0.062172	0.222102	0.453878	0.084*
H61C	−0.019668	0.268548	0.391605	0.084*
C62	−0.2182 (4)	0.21914 (14)	0.14080 (14)	0.0622 (7)
H62A	−0.258495	0.237575	0.096285	0.093*
H62B	−0.313476	0.204524	0.165184	0.093*
H62C	−0.144944	0.181607	0.133796	0.093*
C63	0.0535 (5)	0.26695 (15)	0.03698 (15)	0.0712 (8)
H63A	0.090975	0.221759	0.046436	0.107*
H63B	0.108367	0.284225	−0.002214	0.107*
H63C	−0.067747	0.267357	0.026428	0.107*
C64	0.2907 (4)	0.31845 (16)	0.11139 (17)	0.0711 (8)

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H64A	0.319344	0.344771	0.152577	0.107*
H64B	0.331418	0.340600	0.071500	0.107*
H64C	0.342810	0.274987	0.116774	0.107*
O1	0.1343 (2)	−0.06352 (10)	1.15541 (9)	0.0622 (5)
O2	0.2658 (3)	0.20991 (12)	0.79560 (11)	0.0815 (7)
H2	0.286174	0.217622	0.755102	0.122*
O3	0.3913 (2)	0.21650 (10)	0.66925 (9)	0.0604 (4)
O4	−0.0686 (3)	−0.11719 (12)	1.08954 (10)	0.0750 (6)
O5	0.4589 (2)	−0.08384 (9)	0.59764 (10)	0.0630 (5)
O6	0.2184 (3)	0.16755 (11)	0.21699 (10)	0.0745 (6)
H6	0.184532	0.201280	0.195675	0.112*
O7	0.0558 (2)	0.27785 (8)	0.16543 (8)	0.0471 (3)
O8	0.6930 (3)	−0.02449 (13)	0.62787 (11)	0.0752 (6)

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.96–0.98 Å and with O–C = 1.190–1.448 Å. The absolute configuration was derived from the synthesis and the configuration of the educts.

Comment

20(*R*)-Panoxadiol which was achieved by acid degradation from leaves and stems of ginseng can inhibit the release of inflammatory cytokine interleukin-6 and increase the release of anti-inflammatory mediator interleukin-10 [5, 6]. It has been reported to possess anti-inflammatory effect [7, 8]. It is a potent approach to modify the structures of natural products and preparing a variety of derivatives for the search of new lead compound. Therefore 20(*R*)-panoxadiol and its derivatives have attracted much attention.

There are two molecules in the asymmetric unit of the title structure. One of the two molecules is shown in the figure. Bond lengths and angles are very similar to those given in the literature for protopanaxadiol [9–13].

As shown in the figure, the structure of the title compound and panoxadiol are equivalent except for the substituents at O(1). The title compound contains a classical intramolecular O–H...O hydrogen bond. All these bond lengths and angles are in the expected ranges. And it would be more interesting to compare the two crystallographically independent molecules in this structure.

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