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The crystal structure of (3*S*,12*R*,20*R*,24*S*)-3,12-diacetyl-20,24-epoxy-dammarane-3,12,25-triol acetone solvate, C₃₄H₅₆O₆

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.4 × 0.3 × 0.23 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	0.57 mm ⁻¹
Diffractometer, scan mode:	Bruker FRAMBO, φ and ω -scans
$\theta_{\max}$, completeness:	66°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7716, 5067, 0.053
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2090
$N(\text{param})_{\text{refined}}$:	364
Programs:	Bruker programs [1], SHELX [2, 3], Diamond [4]

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Abstract

C₃₄H₅₆O₆, orthorhombic, $P2_12_12_1$ (no. 19), $a = 8.0343(8)$ Å, $b = 17.7992(16)$ Å, $c = 24.153(3)$ Å, $V = 3453.9(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0642$, $wR_{\text{ref}}(F^2) = 0.1573$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

(3*S*,12*R*,20*R*,24*S*)-20,24-epoxy-dammarane-3,12,25-triol was prepared by oxidation of 20(*R*)-protopanaxadiol with meta-chloroperoxybenzoic acid (*m*-CPBA) as described in literature [5]. Subsequently, the title compound was obtained by acetylation reaction as described in literature [6]. Crystals of the title compound were obtained by slow evaporation from acetone at room temperature within 5 days.

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Experimental details

Hydrogen atoms were placed in idealized positions and treated as riding, with C–H distances of 0.96–0.97 Å, O–H = 0.82 Å. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{\text{eq}}$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{\text{eq}}(\text{C})$.

The application of SQUEEZE [3] led to a lower R index and the solvent molecule was presumed to be a acetone molecule on the basis of the 164 Å³ void and the fact that methanol is commonly used as solvent for growing the crystals.

Comment

Ocotillol-type saponins, sharing a tetrahydrofuran ring and a dammarane skeleton, is one class of rare ginsenosides. It has been found that they have a wide spectrum of biological activities, such as anti-amnesic [7], anti-inflammatory [8], neuro-protective [9] and cardio-protective [10] properties. Natural ocotillol-type saponins share a 20(*S*)-form, but it has been found that the 20(*R*)-stereoisomers have different pharmacological effects [11]. In order to discover new chemical entities with pharmacological activity, 20(*R*)-ocotillol-type saponins were semisynthesized through configuration transformation.

The asymmetric unit of the title compound consists of one independent dammarane and one acetone molecule. The tetrahydrofuran ring adopts an envelope conformation with C20 forming the flap. The C23–C22–C20–C21 torsion angle is $-78.71(6)^\circ$, suggesting the C20 with *R* form. The C22–C23–C24–H24 and C22–C23–C24–C25 torsion angles are

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.6916(6)	−0.0449(3)	0.6120(2)	0.0843(16)
O2	0.3436(6)	0.4316(2)	0.56616(19)	0.0715(13)
O3	0.2330(6)	0.5557(2)	0.63569(19)	0.0726(13)
O4	0.5210(7)	0.6442(4)	0.6407(3)	0.161(3)
H4	0.551731	0.601779	0.649178	0.242*
O5	0.8501(8)	−0.0303(3)	0.6872(3)	0.107(2)
O6	0.5743(8)	0.4793(4)	0.6042(3)	0.138(3)
C1	0.5770(7)	0.1582(4)	0.5867(3)	0.0647(19)
H1A	0.607393	0.191276	0.556450	0.078*
H1B	0.620755	0.179599	0.620663	0.078*
C2	0.6556(9)	0.0812(4)	0.5773(3)	0.078(2)
H2A	0.618327	0.061242	0.542066	0.094*
H2B	0.775741	0.086413	0.575787	0.094*
C3	0.6097(9)	0.0273(4)	0.6229(3)	0.070(2)
H3	0.650071	0.047166	0.658299	0.084*
C4	0.4180(9)	0.0136(4)	0.6272(3)	0.0669(19)
C5	0.3378(8)	0.0928(3)	0.6331(3)	0.0597(17)
H5	0.382101	0.111836	0.668226	0.072*
C6	0.1508(8)	0.0897(4)	0.6424(3)	0.070(2)
H6A	0.124632	0.049316	0.667983	0.084*
H6B	0.095044	0.079388	0.607639	0.084*
C7	0.0885(8)	0.1644(4)	0.6660(3)	0.074(2)
7A	−0.031768	0.162007	0.668972	0.089*
H7B	0.132546	0.170031	0.703194	0.089*
C8	0.1344(8)	0.2337(4)	0.6328(3)	0.0606(17)
C9	0.3212(7)	0.2306(3)	0.6158(3)	0.0565(17)
H9	0.382553	0.236145	0.650600	0.068*
C10	0.3846(8)	0.1537(4)	0.5907(2)	0.0567(17)
C11	0.3705(8)	0.2999(3)	0.5813(2)	0.0608(17)
H11A	0.489077	0.298008	0.573874	0.073*
H11B	0.312948	0.298020	0.546021	0.073*
C12	0.3297(8)	0.3745(3)	0.6099(3)	0.0591(17)
H12	0.407408	0.384750	0.640223	0.071*
C13	0.1485(8)	0.3787(3)	0.6297(2)	0.0572(17)
H13	0.076389	0.376172	0.597074	0.069*
C14	0.1121(8)	0.3079(4)	0.6663(3)	0.0620(18)
C15	−0.0695(8)	0.3257(4)	0.6854(3)	0.083(2)
H15A	−0.149699	0.308256	0.658273	0.100*
H15B	−0.093374	0.302144	0.720720	0.100*
C16	−0.0745(9)	0.4122(4)	0.6905(3)	0.086(2)
H16A	−0.084606	0.426640	0.729080	0.103*
H16B	−0.169754	0.431988	0.670562	0.103*
C17	0.0885(8)	0.4445(4)	0.6658(3)	0.0630(18)
H17	0.167850	0.449670	0.696436	0.076*
C18	0.0176(8)	0.2344(4)	0.5807(3)	0.076(2)
H18A	0.062234	0.268019	0.553346	0.114*
H18B	−0.091485	0.251013	0.591308	0.114*
H18C	0.010741	0.184679	0.565486	0.114*
C19	0.3148(8)	0.1387(4)	0.5313(2)	0.072(2)
H19A	0.324631	0.183503	0.509453	0.108*
H19B	0.199819	0.124412	0.533801	0.108*
H19C	0.377089	0.098919	0.514238	0.108*
C20	0.0650(9)	0.5221(4)	0.6410(3)	0.0681(19)
C21	−0.0150(9)	0.5230(4)	0.5829(3)	0.080(2)
H21A	−0.124482	0.501539	0.584814	0.121*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H21B	−0.022655	0.573909	0.569943	0.121*
H21C	0.052213	0.494259	0.557807	0.121*
C22	−0.0192(9)	0.5755(4)	0.6817(3)	0.083(2)
H22A	−0.139193	0.573569	0.677628	0.099*
H22B	0.009517	0.562593	0.719498	0.099*
C23	0.0457(10)	0.6522(4)	0.6671(4)	0.098(3)
H23A	0.054057	0.683504	0.699868	0.118*
H23B	−0.026789	0.676691	0.640534	0.118*
C24	0.2213(9)	0.6376(4)	0.6415(3)	0.075(2)
H24	0.224627	0.660137	0.604527	0.090*
C25	0.3706(12)	0.6642(5)	0.6734(4)	0.103(3)
C26	0.3683(14)	0.6323(5)	0.7319(4)	0.174(6)
H26A	0.267842	0.647602	0.750283	0.261*
H26B	0.372797	0.578484	0.730295	0.261*
H26C	0.462758	0.650831	0.752085	0.261*
C27	0.3733(12)	0.7514(4)	0.6736(4)	0.137(4)
H27A	0.279417	0.769843	0.694083	0.206*
H27B	0.474258	0.768598	0.690718	0.206*
H27C	0.368087	0.769561	0.636266	0.206*
C28	0.3867(10)	−0.0336(4)	0.6804(3)	0.084(2)
H28A	0.426264	−0.006441	0.712110	0.125*
H28B	0.269657	−0.042979	0.684289	0.125*
H28C	0.444990	−0.080541	0.677582	0.125*
C29	0.3517(9)	−0.0332(4)	0.5780(3)	0.084(2)
H29A	0.407114	−0.081021	0.577209	0.127*
H29B	0.234116	−0.040857	0.582220	0.127*
H29C	0.372785	−0.006949	0.544001	0.127*
C30	0.2219(9)	0.3091(4)	0.7192(2)	0.078(2)
H30A	0.204800	0.355548	0.738638	0.118*
H30B	0.191760	0.267844	0.742714	0.118*
H30C	0.336893	0.304781	0.708933	0.118*
C31	0.8121(11)	−0.0648(5)	0.6469(4)	0.083(2)
C32	0.8895(11)	−0.1388(4)	0.6290(4)	0.117(3)
H32A	0.838239	−0.155480	0.595282	0.176*
H32B	1.006664	−0.131950	0.623090	0.176*
H32C	0.872491	−0.175782	0.657404	0.176*
C33	0.4712(13)	0.4794(5)	0.5681(4)	0.096(3)
C34	0.4670(11)	0.5362(4)	0.5218(3)	0.113(3)
H34A	0.374551	0.525652	0.497877	0.170*
H34B	0.455073	0.585714	0.537072	0.170*
H34C	0.568688	0.533356	0.501076	0.170*

123.67(6)° and −112.38(6)°, suggesting the C24 O be in the *S* configuration. The intramolecular O4—H4···O6 hydrogen bond is present. The corresponding O4···O6 and H4···O6 distances are 3.087(9) Å and 2.442(7) Å, respectively, and O4—H4···O6 is 136.3°.

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