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The crystal structure of 4a-formyl-1,2,3,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,12b,13,14b-icosahydro-1-2-6a,6b,9,9,12a-heptamethylpicen-10-yl acetate, C₃₂H₅₀O₃

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Abstract

C₃₂H₅₀O₃, orthorhombic, *P*₂₁2₁2₁ (no. 19), *a* = 8.0417(5) Å, *b* = 11.7996(6) Å, *c* = 30.1631(16) Å, *V* = 2862.1(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0712, *wR*_{ref}(*F*²) = 0.2046, *T* = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was isolated from a methanolic extract of the bark material of *Strychnos henningsii* (Nelson Mandela University Herbarium voucher PEU6859). The methanolic

Table 1: Data collection and handling.

Crystal:	Colourless platelet
Size:	0.64 × 0.44 × 0.19 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.07 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	28.4°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	45550, 7133, 0.078
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 5234
<i>N</i> (<i>param</i>) _{refined} :	324
Programs:	Bruker [1], SHELX [2–4], WinGX/ORTEP [5], Platon [6], Mercury [7]

extract was then subjected to vacuum liquid chromatography where a fraction collected through a solvent system of dichloromethane: methanol (98:2) resulting in a colourless elute. The elute was slowly evaporated in a fume hood overnight to yield colourless platelet crystals.

Experimental details

Absolute configuration is unknown due to poor anomalous-dispersion effects in diffraction measurements. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with *U*_{iso}(H) set to 1.2 *U*_{eq}(C). The H atoms of the methyl

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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	1.2222(5)	0.3680(3)	0.16693(16)	0.0863(12)
O2	0.2930(5)	0.5583(3)	0.43386(11)	0.0738(10)
O3	0.1195(7)	0.6993(5)	0.42123(17)	0.1174(18)
C1	0.8383(5)	0.4931(3)	0.13276(14)	0.0444(9)
H1	0.806154	0.421958	0.118384	0.053*
C2	0.7590(5)	0.5890(4)	0.10476(15)	0.0559(11)
H2	0.784521	0.661104	0.119390	0.067*
C3	0.5708(7)	0.5776(6)	0.1030(2)	0.0830(17)
H3A	0.527413	0.573717	0.132606	0.124*
H3B	0.541784	0.509740	0.087214	0.124*
H3C	0.524340	0.642045	0.088037	0.124*
C4	0.8294(7)	0.5948(5)	0.05758(16)	0.0692(14)
H4	0.797148	0.525159	0.042128	0.083*
C5	0.7599(11)	0.6946(7)	0.0315(2)	0.108(2)
H5A	0.813020	0.698244	0.003063	0.162*
H5B	0.780622	0.763556	0.047494	0.162*
H5C	0.642320	0.684915	0.027615	0.162*
C6	1.0182(7)	0.5997(5)	0.05862(18)	0.0737(15)
H6A	1.059981	0.598553	0.028457	0.088*
H6B	1.052558	0.670666	0.072060	0.088*
C7	1.0935(6)	0.5039(5)	0.08381(16)	0.0651(13)
H7A	1.213379	0.513142	0.084282	0.078*
H7B	1.069055	0.433460	0.068542	0.078*
C8	1.0290(5)	0.4957(3)	0.13215(14)	0.0446(9)
C9	1.0942(6)	0.3835(4)	0.14877(17)	0.0600(12)
H9	1.027005	0.320386	0.144252	0.072*
C10	1.0935(5)	0.5921(3)	0.16117(14)	0.0470(9)
H10A	1.214020	0.593473	0.159935	0.056*
H10B	1.052992	0.663740	0.149641	0.056*
C11	1.0383(4)	0.5793(3)	0.20922(13)	0.0416(8)
H11A	1.072037	0.646461	0.225451	0.050*
H11B	1.096254	0.515174	0.222074	0.050*
C12	0.8500(4)	0.5621(3)	0.21583(12)	0.0359(7)
C13	0.7663(5)	0.6797(3)	0.21153(14)	0.0457(9)
H13A	0.786014	0.722796	0.238005	0.068*
H13B	0.648758	0.670097	0.207392	0.068*
H13C	0.812150	0.719120	0.186517	0.068*
C14	0.8180(4)	0.5071(3)	0.26341(13)	0.0375(8)
C15	0.9107(6)	0.3931(3)	0.26690(17)	0.0565(11)
H15A	0.888884	0.359525	0.295297	0.085*
H15B	1.027988	0.405735	0.263688	0.085*
H15C	0.872868	0.343178	0.243892	0.085*
C16	0.8841(5)	0.5859(3)	0.30022(13)	0.0442(9)
H16A	1.004633	0.582660	0.300248	0.053*
H16B	0.852028	0.663178	0.293358	0.053*
C17	0.8215(5)	0.5566(4)	0.34638(13)	0.0472(9)
H17A	0.863975	0.611323	0.367537	0.057*
H17B	0.862189	0.482274	0.354822	0.057*
C18	0.6310(5)	0.5570(3)	0.34757(13)	0.0425(8)
H18	0.598773	0.628525	0.333434	0.051*
C19	0.5580(6)	0.5637(4)	0.39479(14)	0.0525(10)
C20	0.6114(7)	0.4666(5)	0.42609(17)	0.0749(16)
H20A	0.583274	0.486062	0.456074	0.112*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H20B	0.729330	0.455388	0.423810	0.112*
H20C	0.554814	0.398197	0.417833	0.112*
C21	0.6118(9)	0.6761(5)	0.41656(18)	0.0816(17)
H21A	0.722165	0.668336	0.428271	0.122*
H21B	0.536330	0.694533	0.440159	0.122*
H21C	0.610202	0.735510	0.394809	0.122*
C22	0.3676(6)	0.5670(4)	0.39004(15)	0.0572(11)
H22	0.336137	0.640098	0.377104	0.069*
C23	0.1716(8)	0.6269(6)	0.44458(19)	0.0778(16)
C24	0.0973(10)	0.5983(8)	0.4889(2)	0.115(3)
H24A	0.167981	0.626538	0.512042	0.172*
H24B	0.087044	0.517542	0.491610	0.172*
H24C	−0.010607	0.632523	0.491339	0.172*
C25	0.2962(5)	0.4737(5)	0.36153(15)	0.0587(12)
H25A	0.176726	0.483220	0.359244	0.070*
H25B	0.317699	0.400935	0.375360	0.070*
C26	0.3722(5)	0.4753(4)	0.31554(14)	0.0498(10)
H26A	0.342812	0.545786	0.300975	0.060*
H26B	0.325643	0.413617	0.298262	0.060*
C27	0.5638(4)	0.4636(3)	0.31646(13)	0.0394(8)
C28	0.6046(6)	0.3421(4)	0.33215(17)	0.0595(12)
H28A	0.518481	0.315839	0.351650	0.089*
H28B	0.708957	0.342147	0.347618	0.089*
H28C	0.611804	0.292676	0.306925	0.089*
C29	0.6283(4)	0.4898(3)	0.26880(12)	0.0390(8)
H29	0.578518	0.562867	0.260954	0.047*
C30	0.5648(6)	0.4066(4)	0.23369(15)	0.0594(12)
H30A	0.581607	0.329930	0.244389	0.071*
H30B	0.446199	0.417836	0.229921	0.071*
C31	0.6477(5)	0.4184(4)	0.18958(15)	0.0536(11)
H31	0.605904	0.374517	0.166524	0.064*
C32	0.7747(4)	0.4854(3)	0.18020(13)	0.0390(8)

groups were allowed to rotate with a fixed angle around the C—C bonds to best fit the experimental electron density (HFIX 137 in the SHELXL program [3]), with *U*_{iso}(H) set to 1.5*U*_{eq}(C).

Comment

The title compound, in the above figure, is a pentacyclic triterpene of the ursane derivative main group. These pentacyclic triterpenes are commonly found in most plants with a vast spectrum of biological activities and some may be medicinally useful [8]. To the best of our knowledge, only fourteen examples of these pentacyclic triterpene ursane derivatives have been reported [9–11]. This contribution is part of our continuing interest in natural products, isolation, characterisation and biological activity.

The asymmetric unit cell contains one molecule of the titled compound with all bond lengths and angles in expected ranges and similar to those of a related compound published

in the literature [9–11]. Compared to Ursolic acid the carboxyl group has been replaced with a formyl group and the hydroxyl group with an acetoxy group. Ignoring the functional groups the RMSD is 0.1427 with a MaxD of 0.2937 Å compared to the Ursolic acid structure recorded at room temperature [12].

All the hydrogen interactions are weak with the shortest intramolecular interaction being C22–H22...O3 of length 2.702(7) Å, and the shortest intermolecular interaction C30–H30B...O1 of length 3.443(6) Å. The analyses of the Hirshfeld surface with Crystal Explorer [13] shows the dominance of the H...H contact area at 86.6% while the O...H reciprocal contact surface area is only 13.2%.

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