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Crystal structure of (4*S*,4*aS*,6*aR*,6*bR*,12*aS*,12*bR*,14*aS*,14*bR*)-3,3,6*a*,6*b*,9,9,12*a*-heptamethyloctadecahydro-1*H*,3*H*-4,14*b*-ethanophenanthro[1,2-*h*]isochromene-1(6*bH*)-one, C₃₀H₄₈O₂

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Abstract

C₃₀H₄₈O₂, orthorhombic, *P*₂₁₂₁₂₁ (no. 19), *a* = 10.8192(12) Å, *b* = 16.4915(17) Å, *c* = 28.832(3) Å, *V* = 5144.4(10) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0582, *wR*_{ref}(*F*²) = 0.1169, *T* = 293(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 powdered whole plants of *Pyrrosia lingua* were extracted with 90° EtOH under reflux for 3 times, 1 h each time. The combined EtOH extracts were concentrated under vacuum to give a residue, which was suspended in water, then extracted with AcOEt for 3 times. The AcOEt extract was chromatographed on silica gel using gradient elution petroleum ether/AcOEt (volume ratio 9:1–1:1) to yield seven fractions (fractions A–E). The title compound (70 mg) was isolated from the fraction B by the silica gel column.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.24 × 0.23 × 0.21 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:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	39401, 9050, 0.071
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 5738
<i>N</i> (<i>param</i>) _{refined} :	581
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1'	0.1559(4)	0.4989(2)	0.88140(14)	0.0488(11)
H1'A	0.0792	0.5226	0.8705	0.059*
H1'B	0.1724	0.5207	0.9120	0.059*
C1	0.1376(4)	0.3195(2)	1.11456(15)	0.0543(12)
H1A	0.1410	0.3358	1.1469	0.065*
H1B	0.0640	0.3434	1.1011	0.065*
C2	0.1266(5)	0.2274(3)	1.11219(17)	0.0675(14)
H2A	0.1959	0.2029	1.1283	0.081*
H2B	0.0512	0.2104	1.1276	0.081*
C2'	0.1393(5)	0.4068(2)	0.88525(15)	0.0613(13)
H2'A	0.2125	0.3832	0.8992	0.074*
H2'B	0.0694	0.3951	0.9052	0.074*
C3'	0.1182(4)	0.3693(3)	0.83825(15)	0.0535(12)
H3'A	0.0399	0.3886	0.8262	0.064*
H3'B	0.1120	0.3110	0.8419	0.064*
C3	0.1249(4)	0.1983(3)	1.06231(17)	0.0639(14)
H3A	0.0504	0.2183	1.0475	0.077*
H3B	0.1212	0.1395	1.0622	0.077*
C4	0.2373(4)	0.2255(3)	1.03366(16)	0.0524(12)
C4'	0.2192(4)	0.3879(2)	0.80295(14)	0.0406(11)
C5	0.2504(4)	0.3186(2)	1.03907(13)	0.0392(10)
H5	0.1742	0.3403	1.0256	0.047*
C5'	0.2393(4)	0.4812(2)	0.80135(12)	0.0345(10)
H5'	0.1601	0.5028	0.7904	0.041*
C6'	0.3327(4)	0.5107(2)	0.76547(13)	0.0402(10)
H6'A	0.4158	0.5015	0.7768	0.048*
H6'B	0.3225	0.4803	0.7369	0.048*
C6	0.3527(4)	0.3560(2)	1.00992(14)	0.0438(11)
H6A	0.4320	0.3452	1.0244	0.053*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H6B	0.3529	0.3311	0.9794	0.053*
C7'	0.3146(4)	0.6005(2)	0.75584(13)	0.0434(11)
H7'A	0.2335	0.6083	0.7422	0.052*
H7'B	0.3755	0.6176	0.7332	0.052*
C7	0.3351(4)	0.4465(2)	1.00500(13)	0.0430(11)
H7A	0.2592	0.4565	0.9880	0.052*
H7B	0.4028	0.4683	0.9868	0.052*
C8	0.3294(3)	0.4919(2)	1.05125(13)	0.0321(9)
C8'	0.3258(3)	0.6550(2)	0.79896(12)	0.0336(10)
C9'	0.2478(4)	0.6181(2)	0.83938(11)	0.0320(9)
H9'	0.1618	0.6252	0.8295	0.038*
C9	0.2384(4)	0.4474(2)	1.08507(12)	0.0324(10)
H9	0.1567	0.4554	1.0712	0.039*
C10'	0.2604(4)	0.5244(2)	0.84876(13)	0.0351(10)
C10	0.2522(4)	0.3535(2)	1.08924(13)	0.0389(10)
C11'	0.2590(4)	0.6700(2)	0.88308(12)	0.0422(11)
H11A	0.2086	0.6465	0.9074	0.051*
H11B	0.3443	0.6696	0.8935	0.051*
C11	0.2318(4)	0.4929(2)	1.13164(12)	0.0382(10)
H11C	0.1726	0.4660	1.1517	0.046*
H11D	0.3120	0.4906	1.1466	0.046*
C12'	0.2185(4)	0.7570(2)	0.87486(13)	0.0416(11)
H12A	0.2344	0.7887	0.9026	0.050*
H12B	0.1303	0.7581	0.8690	0.050*
C12	0.1941(4)	0.5811(2)	1.12567(13)	0.0378(10)
H12C	0.1958	0.6080	1.1556	0.045*
H12D	0.1102	0.5836	1.1139	0.045*
C13'	0.2860(3)	0.7952(2)	0.83401(12)	0.0324(10)
H13'	0.3737	0.7908	0.8421	0.039*
C13	0.2806(3)	0.6250(2)	1.09223(12)	0.0312(10)
H13	0.3633	0.6149	1.1048	0.037*
C14'	0.2728(3)	0.7442(2)	0.78856(12)	0.0329(10)
C14	0.2818(3)	0.5828(2)	1.04363(12)	0.0310(10)
C15'	0.3471(4)	0.7859(2)	0.74933(14)	0.0503(12)
H15A	0.4346	0.7805	0.7560	0.060*
H15B	0.3310	0.7577	0.7204	0.060*
C15	0.3694(4)	0.6285(2)	1.00989(13)	0.0418(11)
H15C	0.4542	0.6173	1.0187	0.050*
H15D	0.3573	0.6073	0.9788	0.050*
C16'	0.3171(4)	0.8758(2)	0.74277(14)	0.0543(13)
H16A	0.3678	0.8983	0.7182	0.065*
H16B	0.2310	0.8822	0.7342	0.065*
C16	0.3507(4)	0.7203(2)	1.00882(13)	0.0449(11)
H16C	0.4105	0.7450	0.9882	0.054*
H16D	0.2685	0.7329	0.9975	0.054*
C17'	0.3426(4)	0.9195(2)	0.78781(15)	0.0435(11)
H17'	0.4284	0.9067	0.7959	0.052*
C17	0.3669(3)	0.7530(2)	1.05743(12)	0.0343(10)
H17	0.4470	0.7328	1.0683	0.041*
C18	0.2703(3)	0.7185(2)	1.09068(12)	0.0304(9)
C18'	0.2641(4)	0.8871(2)	0.82760(13)	0.0334(10)
C19	0.3042(4)	0.7598(2)	1.13740(13)	0.0420(11)
H19C	0.2304	0.7736	1.1548	0.050*
H19D	0.3557	0.7246	1.1562	0.050*
C19'	0.3096(4)	0.9387(2)	0.86941(15)	0.0482(12)
H19A	0.2418	0.9513	0.8902	0.058*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H19B	0.3733	0.9103	0.8866	0.058*
C20	0.3628(4)	0.8425(2)	1.07029(13)	0.0378(10)
H20	0.4343	0.8707	1.0571	0.045*
C20'	0.3285(4)	1.0114(3)	0.79543(17)	0.0518(12)
H20'	0.3898	1.0408	0.7769	0.062*
C21'	0.3617(4)	1.0164(3)	0.84698(17)	0.0625(14)
H21A	0.3249	1.0641	0.8609	0.075*
H21B	0.4506	1.0191	0.8510	0.075*
C21	0.3757(4)	0.8373(2)	1.12298(14)	0.0481(11)
H21C	0.3402	0.8849	1.1377	0.058*
H21D	0.4620	0.8332	1.1318	0.058*
C22	0.2432(4)	0.8821(2)	1.05344(15)	0.0470(11)
C22'	0.1983(4)	1.0401(3)	0.78307(17)	0.0544(13)
C23	0.2097(5)	0.2046(3)	0.98266(16)	0.0720(16)
H23D	0.1380	0.2339	0.9726	0.108*
H23E	0.2793	0.2193	0.9638	0.108*
H23F	0.1948	0.1474	0.9798	0.108*
C23'	0.1731(5)	0.3585(3)	0.75561(15)	0.0678(15)
H23A	0.2348	0.3693	0.7325	0.102*
H23B	0.1574	0.3012	0.7569	0.102*
H23C	0.0982	0.3865	0.7477	0.102*
C24'	0.3368(4)	0.3397(3)	0.81432(16)	0.0600(13)
H24A	0.3996	0.3519	0.7918	0.090*
H24B	0.3655	0.3543	0.8447	0.090*
H24C	0.3188	0.2827	0.8135	0.090*
C24	0.3524(5)	0.1768(3)	1.04778(19)	0.0712(15)
H24D	0.4219	0.1944	1.0296	0.107*
H24E	0.3694	0.1856	1.0801	0.107*
H24F	0.3380	0.1202	1.0424	0.107*
C25'	0.3848(4)	0.5038(3)	0.87230(14)	0.0492(11)
H25A	0.4092	0.5479	0.8920	0.074*
H25B	0.4468	0.4953	0.8490	0.074*
H25C	0.3757	0.4555	0.8905	0.074*
C25	0.3665(4)	0.3288(2)	1.11789(14)	0.0542(12)
H25D	0.3772	0.3660	1.1431	0.081*
H25E	0.4385	0.3298	1.0985	0.081*
H25F	0.3550	0.2750	1.1299	0.081*
C26	0.4632(3)	0.4920(2)	1.07159(14)	0.0442(11)
H26D	0.5011	0.4403	1.0661	0.066*
H26E	0.4597	0.5022	1.1043	0.066*
H26F	0.5110	0.5337	1.0568	0.066*
C26'	0.4646(3)	0.6591(2)	0.81171(14)	0.0465(11)
H26A	0.4955	0.6053	0.8167	0.070*
H26B	0.4747	0.6905	0.8395	0.070*
H26C	0.5095	0.6842	0.7868	0.070*
C27'	0.1374(4)	0.7410(2)	0.77235(13)	0.0453(11)
H27A	0.0878	0.7155	0.7958	0.068*
H27B	0.1078	0.7951	0.7670	0.068*
H27C	0.1321	0.7104	0.7441	0.068*
C27	0.1512(4)	0.5847(2)	1.02128(13)	0.0440(11)
H27D	0.0937	0.5570	1.0411	0.066*
H27E	0.1254	0.6400	1.0174	0.066*
H27F	0.1539	0.5584	0.9916	0.066*
C28	0.1440(4)	0.7518(2)	1.07785(13)	0.0357(10)
C28'	0.1306(4)	0.9119(3)	0.82084(13)	0.0367(10)
C29	0.2524(5)	0.9066(3)	1.00260(16)	0.0710(15)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H29D	0.3198	0.9438	0.9987	0.107*
H29E	0.2665	0.8593	0.9840	0.107*
H29F	0.1768	0.9321	0.9932	0.107*
C29'	0.1835(5)	1.0556(3)	0.73178(18)	0.0843(18)
H29A	0.2421	1.0959	0.7221	0.126*
H29B	0.1978	1.0063	0.7150	0.126*
H29C	0.1012	1.0746	0.7257	0.126*
C30'	0.1575(5)	1.1150(3)	0.80956(19)	0.0811(17)
H30A	0.2139	1.1587	0.8035	0.122*
H30B	0.0760	1.1303	0.7998	0.122*
H30C	0.1569	1.1034	0.8422	0.122*
C30	0.2021(4)	0.9546(3)	1.08161(19)	0.0737(16)
H30D	0.2656	0.9953	1.0812	0.110*
H30E	0.1276	0.9765	1.0685	0.110*
H30F	0.1868	0.9382	1.1130	0.110*
O1	0.1383(2)	0.82509(17)	1.05705(10)	0.0548(8)
O2	0.0464(2)	0.71958(17)	1.08627(9)	0.0461(8)
O3	0.1064(3)	0.97853(17)	0.79499(11)	0.0568(9)
O4	0.0422(3)	0.87826(17)	0.83700(10)	0.0501(8)

chromatography with petroleum ether/AcOEt (volume ratio 85:15) and then the sephadex LH-20 with dichloromethane/methanol (volume ratio 1:1). After dissolving the sample with AcOEt colorless needle crystals of the title compound were obtained within 1 day [5].

Experimental details

The crystal structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 using SHELXTL-2014 [3]. All hydrogen atoms were positioned geometrically, with the $d(C-H) = 0.96-0.98$ Å, $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ and $U_{iso}(H) = 1.5$ times $U_{eq}(O)$.

Comment

Pyrrosia lingua is a traditional Chinese herb recorded in Chinese Pharmacopoeia for treating swelling, urocystitis, urinary calculus, bloody urine, cough, bronchitis, etc. and has also been used to cure infection and diarrhoea in minority regions of Guizhou Province. In previous reports, hopane triterpenoids had been isolated from this plant [6]. In order to discover more active hopane triterpenoids from this plant, phytochemical investigation led to the isolation of the title compound. This contribution is part of our continuing interest in natural occurring compounds [7, 8].

The bond lengths and angles derived from the title structure are within normal ranges [8]. The title compound contains one five-membered ring which shows an envelope conformation at C17 and five six-membered rings which all present chair conformations (*cf.* the figure). At the same time, the title compound contains an lactone and seven methyl groups, of which C24, C25, C26, C30 are in β -configuration, and C23, C27, C29 are in α -configuration. The lactone was confirmed by the distances $d(C22-O1) = 1.477(5)$, $d(C28-O1) = 1.351(5)$, $d(C28-O2) = 1.207(5)$. The intermolecular connection was achieved by van der Waals forces.

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