Kang He, Tian-Tai Wu, Lin-Yan Shen, Lin-Lin Fan, Jiang-Hai Ye and Juan Zou*

Crystal structure of (4*aR*,6*aR*,6*bR*,10*S*,12*aR*)-10-acetoxy-1,2,3,4, 4*a*,5,6, 6*a*,6*b*,7,8,8*a*,9,10,11,12,12*a*, 12*b*,13,14*b*-icosahydro-2,2,4*a*,6*b*,9,9,12*a*-heptamethylpicene-6*a*-carboxylic acid, C₃₂H₅₀O₄

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.35 × 0.16 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.07 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	56475, 10029, 0.064
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 8128
$N(\text{param})_{\text{refined}}$:	669
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

C₃₂H₅₀O₄, orthorhombic, $P2_12_12_1$ (no. 19), $a = 14.090(3)$ Å, $b = 15.642(3)$ Å, $c = 25.956(6)$ Å, $V = 5721(2)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0679$, $wR_{\text{ref}}(F^2) = 0.1764$, $T = 250(2)$ K.

CCDC no.: 1940948

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 roots of *Astilbe grandis* Stapf ex Wils. 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

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.8311(3)	0.9976(3)	0.32722(18)	0.0553(11)
O2	0.9013(3)	0.8728(3)	0.3098(2)	0.0692(13)
O3	0.3918(3)	0.7188(2)	0.37349(13)	0.0404(9)
O4	0.3716(3)	0.7115(2)	0.45846(14)	0.0448(9)
H4	0.389(5)	0.661(4)	0.4533(5)	0.067*
O5	−0.0571(3)	0.2600(2)	0.33663(15)	0.0478(10)
O6	−0.1106(3)	0.3660(3)	0.2866(2)	0.0740(14)
O7	0.3900(3)	0.5373(2)	0.36666(13)	0.0399(8)
H7	0.379(4)	0.596(4)	0.3692(8)	0.060*
O8	0.4188(3)	0.5510(2)	0.45070(14)	0.0462(10)
C1	0.5749(4)	0.9482(4)	0.30111(19)	0.0399(12)
H1A	0.5777	0.8859	0.2978	0.048*
H1B	0.5336	0.9700	0.2737	0.048*
C2	0.6747(4)	0.9849(4)	0.2939(2)	0.0448(13)
H2A	0.7001	0.9664	0.2606	0.054*
H2B	0.6714	1.0475	0.2938	0.054*
C3	0.7399(4)	0.9560(4)	0.3363(2)	0.0470(14)
H3	0.7489	0.8934	0.3330	0.056*
C4	0.7055(4)	0.9753(4)	0.3916(2)	0.0470(14)
C5	0.6021(4)	0.9422(3)	0.39597(19)	0.0359(11)
H5	0.6074	0.8796	0.3912	0.043*
C6	0.5577(4)	0.9527(4)	0.44965(19)	0.0432(13)
H6A	0.6046	0.9382	0.4761	0.052*
H6B	0.5384	1.0123	0.4547	0.052*
C7	0.4718(4)	0.8944(3)	0.45470(19)	0.0381(12)
H7A	0.4454	0.9007	0.4894	0.046*
H7B	0.4925	0.8350	0.4507	0.046*
C8	0.3935(3)	0.9128(3)	0.41528(17)	0.0286(10)
C9	0.4383(3)	0.9164(3)	0.36011(17)	0.0285(10)
H9	0.4576	0.8569	0.3523	0.034*

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C10	0.5311(3)	0.9711(3)	0.35386(18)	0.0291(10)
C11	0.3610(4)	0.9378(3)	0.32039(19)	0.0383(12)
H11A	0.3480	0.9993	0.3217	0.046*
H11B	0.3851	0.9246	0.2859	0.046*
C12	0.2705(3)	0.8905(3)	0.32864(18)	0.0333(11)
H12	0.2253	0.8927	0.3020	0.040*
C13	0.2480(3)	0.8452(3)	0.37037(17)	0.0263(9)
C14	0.3145(3)	0.8387(3)	0.41669(17)	0.0263(10)
C15	0.2559(4)	0.8433(3)	0.46740(16)	0.0312(10)
H15A	0.2969	0.8268	0.4962	0.037*
H15B	0.2363	0.9027	0.4730	0.037*
C16	0.1675(4)	0.7865(3)	0.46791(17)	0.0341(11)
H16A	0.1336	0.7949	0.5005	0.041*
H16B	0.1872	0.7265	0.4662	0.041*
C17	0.1001(3)	0.8056(3)	0.42306(18)	0.0331(11)
C18	0.1559(3)	0.7943(3)	0.37164(17)	0.0273(10)
H18	0.1151	0.8171	0.3438	0.033*
C19	0.1741(3)	0.6992(3)	0.35915(19)	0.0312(10)
H19A	0.2063	0.6955	0.3257	0.037*
H19B	0.2172	0.6757	0.3852	0.037*
C20	0.0846(4)	0.6441(3)	0.3573(2)	0.0385(12)
C21	0.0348(4)	0.6529(4)	0.4095(2)	0.0473(14)
H21A	−0.0247	0.6205	0.4087	0.057*
H21B	0.0752	0.6279	0.4363	0.057*
C22	0.0134(4)	0.7451(4)	0.4234(2)	0.0414(13)
H22A	−0.0150	0.7463	0.4579	0.050*
H22B	−0.0339	0.7672	0.3992	0.050*
C23	0.7702(5)	0.9248(5)	0.4290(3)	0.074(2)
H23A	0.7593	0.8640	0.4246	0.110*
H23B	0.7557	0.9410	0.4642	0.110*
H23C	0.8361	0.9378	0.4216	0.110*
C24	0.7184(5)	1.0714(4)	0.4048(3)	0.0609(17)
H24A	0.6914	1.1060	0.3774	0.091*
H24B	0.7854	1.0841	0.4082	0.091*
H24C	0.6863	1.0840	0.4370	0.091*
C25	0.3459(4)	0.9984(3)	0.4292(2)	0.0421(13)
H25A	0.3886	1.0451	0.4211	0.063*
H25B	0.3314	0.9992	0.4658	0.063*
H25C	0.2877	1.0047	0.4096	0.063*
C26	0.5116(4)	1.0681(3)	0.3534(2)	0.0443(13)
H26A	0.5597	1.0967	0.3330	0.067*
H26B	0.5133	1.0899	0.3884	0.067*
H26C	0.4496	1.0788	0.3386	0.067*
C27	0.3632(3)	0.7506(3)	0.41469(18)	0.0300(10)
C28	0.0630(4)	0.8968(4)	0.4268(2)	0.0473(14)
H28A	0.1154	0.9366	0.4233	0.071*
H28B	0.0326	0.9051	0.4600	0.071*
H28C	0.0173	0.9069	0.3996	0.071*
C29	0.1154(5)	0.5515(4)	0.3487(3)	0.0670(19)
H29A	0.1498	0.5472	0.3165	0.101*
H29B	0.0598	0.5150	0.3475	0.101*
H29C	0.1562	0.5336	0.3768	0.101*
C30	0.0191(4)	0.6703(4)	0.3130(2)	0.0467(14)
H30A	0.0544	0.6699	0.2810	0.070*
H30B	−0.0054	0.7273	0.3194	0.070*
H30C	−0.0333	0.6303	0.3107	0.070*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C31	0.9044(4)	0.9487(4)	0.3127(2)	0.0439(13)
C32	0.9890(4)	1.0029(5)	0.3009(3)	0.0626(18)
H32A	1.0173	0.9844	0.2687	0.094*
H32B	1.0351	0.9973	0.3285	0.094*
H32C	0.9696	1.0622	0.2980	0.094*
C33	0.1995(4)	0.3013(3)	0.30419(18)	0.0378(12)
H33A	0.1981	0.3630	0.2977	0.045*
H33B	0.2390	0.2749	0.2775	0.045*
C34	0.0988(4)	0.2661(4)	0.29984(19)	0.0422(13)
H34A	0.0725	0.2809	0.2661	0.051*
H34B	0.1005	0.2036	0.3025	0.051*
C35	0.0360(4)	0.3017(3)	0.3415(2)	0.0374(12)
H35	0.0280	0.3639	0.3357	0.045*
C36	0.0722(4)	0.2873(3)	0.39691(19)	0.0386(12)
C37	0.1762(3)	0.3196(3)	0.39847(17)	0.0307(11)
H37	0.1714	0.3815	0.3909	0.037*
C38	0.2232(4)	0.3157(4)	0.45178(19)	0.0412(13)
H38A	0.1779	0.3343	0.4781	0.049*
H38B	0.2417	0.2566	0.4595	0.049*
C39	0.3105(4)	0.3729(3)	0.45325(19)	0.0389(12)
H39A	0.3382	0.3706	0.4879	0.047*
H39B	0.2910	0.4320	0.4467	0.047*
C40	0.3869(3)	0.3475(3)	0.41370(16)	0.0281(10)
C41	0.3399(3)	0.3381(3)	0.35966(17)	0.0292(10)
H41	0.3220	0.3969	0.3494	0.035*
C42	0.2450(4)	0.2851(3)	0.35705(18)	0.0300(10)
C43	0.4133(4)	0.3097(3)	0.31987(18)	0.0378(12)
H43A	0.4256	0.2485	0.3243	0.045*
H43B	0.3869	0.3182	0.2853	0.045*
C44	0.5050(4)	0.3571(3)	0.32385(18)	0.0340(11)
H44	0.5485	0.3503	0.2967	0.041*
C45	0.5304(3)	0.4077(3)	0.36213(16)	0.0264(10)
C46	0.4687(3)	0.4195(3)	0.41059(17)	0.0269(10)
C47	0.5335(4)	0.4167(3)	0.45885(17)	0.0327(11)
H47A	0.4965	0.4345	0.4889	0.039*
H47B	0.5537	0.3575	0.4646	0.039*
C48	0.6218(4)	0.4732(3)	0.45492(18)	0.0358(11)
H48A	0.6021	0.5331	0.4524	0.043*
H48B	0.6595	0.4670	0.4864	0.043*
C49	0.6838(4)	0.4509(3)	0.4084(2)	0.0385(12)
C50	0.6230(3)	0.4583(3)	0.35904(18)	0.0318(11)
H50	0.6607	0.4321	0.3310	0.038*
C51	0.6058(4)	0.5513(3)	0.3432(2)	0.0365(11)
H51A	0.5713	0.5515	0.3104	0.044*
H51B	0.5646	0.5779	0.3690	0.044*
C52	0.6944(4)	0.6065(4)	0.3372(2)	0.0433(13)
C53	0.7492(4)	0.6012(4)	0.3882(2)	0.0521(15)
H53A	0.8091	0.6325	0.3846	0.062*
H53B	0.7120	0.6293	0.4152	0.062*
C54	0.7705(4)	0.5101(4)	0.4045(2)	0.0475(14)
H54A	0.8020	0.5116	0.4382	0.057*
H54B	0.8153	0.4852	0.3798	0.057*
C55	0.0583(4)	0.1933(4)	0.4137(2)	0.0526(15)
H55A	−0.0086	0.1826	0.4198	0.079*
H55B	0.0937	0.1828	0.4452	0.079*
H55C	0.0810	0.1556	0.3868	0.079*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C56	0.0113(4)	0.3438(4)	0.4332(2)	0.0558(16)
H56A	0.0222	0.4036	0.4254	0.084*
H56B	0.0289	0.3327	0.4687	0.084*
H56C	−0.0554	0.3305	0.4284	0.084*
C57	0.4332(4)	0.2628(3)	0.4308(2)	0.0443(13)
H57A	0.3875	0.2167	0.4275	0.067*
H57B	0.4533	0.2674	0.4664	0.067*
H57C	0.4878	0.2510	0.4092	0.067*
C58	0.2616(4)	0.1874(3)	0.3613(2)	0.0465(14)
H58A	0.2105	0.1573	0.3440	0.070*
H58B	0.2631	0.1709	0.3973	0.070*
H58C	0.3216	0.1727	0.3452	0.070*
C59	0.4230(3)	0.5087(3)	0.41039(18)	0.0310(10)
C60	0.7206(4)	0.3588(4)	0.4148(2)	0.0526(15)
H60A	0.6679	0.3192	0.4124	0.079*
H60B	0.7510	0.3529	0.4481	0.079*
H60C	0.7662	0.3464	0.3878	0.079*
C61	0.6640(5)	0.6986(4)	0.3277(3)	0.0651(18)
H61A	0.6287	0.7019	0.2957	0.098*
H61B	0.7198	0.7347	0.3254	0.098*
H61C	0.6243	0.7178	0.3559	0.098*
C62	0.7540(5)	0.5779(4)	0.2912(2)	0.0590(17)
H62A	0.7149	0.5772	0.2604	0.088*
H62B	0.7785	0.5209	0.2975	0.088*
H62C	0.8064	0.6172	0.2864	0.088*
C63	−0.1203(4)	0.2967(4)	0.3050(2)	0.0459(14)
C64	−0.2051(4)	0.2409(5)	0.2972(2)	0.0597(17)
H64A	−0.2373	0.2573	0.2656	0.090*
H64B	−0.2481	0.2473	0.3261	0.090*
H64C	−0.1850	0.1817	0.2947	0.090*

extract was chromatographed on silica gel using gradient elution dichloromethane/methanol (volume ratio 100:1–8:2) to yield three fractions (fractions A–C). The fraction B was chromatographed on silica gel using gradient elution petroleum ether/AcOEt (volume ratio 100:1–1:1) to yield five fractions (fractions B1–B5). The title compound (6 mg) was isolated from the fraction B2 by the silica gel column chromatography with dichloromethane/AcOEt (volume ratio 100:1–8:2) and then the sephadex LH-20 with dichloromethane/methanol (volume ratio 1:1). After dissolving the sample with dichloromethane colorless needle crystals of the title compound were obtained within 2 day [5].

Experimental details

All hydrogen atoms were positioned geometrically, with the $d(\text{C}–\text{H}) = 0.96–0.98 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{O})$.

Comment

Astilbe grandis Stapf ex Wils., which belongs to the *Astilbe* genus of the family Saxifragaceae, is well-known in Miao Nationality Areas of Guizhou Province under the name “Ma Sang Gou Bang”. The investigation of the Miao nationality medicine in Guizhou reveals that “Ma Sang Gou Bang” traditionally known as “Da Yao” has been used for treating bruises, back pain, aching pain and rheumatism in Miao Nationality Areas of Southeast Guizhou. In order to discover active compounds from this plant, phytochemical investigation led to the isolation of the title compound.

The bond lengths and angles derived from the title structure are within normal ranges [6, 7]. There are two independent molecules in the asymmetric unit of the title structure. The title compound contains five six-membered rings which all present chair conformations (cf. the figure). At the same time, the title compound contains one double bond, one ester group, one carboxyl group and eight methyl groups, of which C23, C25, C26, C28, C29 are in β -bond, and C24, C30 are in α -bond. The C12–C13 distance is 1.332(6). The ester group was confirmed by the distances of $d(\text{C31}–\text{O1}) = 1.339(7)$, $d(\text{C31}–\text{O2}) = 1.191(7)$, $d(\text{C3}–\text{O1}) = 1.459(7) \text{ \AA}$. The carboxyl group was confirmed by the distance of $d(\text{C27}–\text{O3}) = 1.246(6)$, $d(\text{C27}–\text{O4}) = 1.295(6)$, $d(\text{H4}–\text{O4}) = 0.85(7) \text{ \AA}$.

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