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The crystal structure of (2a'S, 2a1'S, 3R, 5a'S, 7'R)-5-(furan-3-yl)-2a', 2a1'-dihydroxy-7'-methyldecahydro-2H-spiro[furan-3, 6'-naphtho [1,8-bc]furan]-2, 2'(2a'H)-dione, C₁₉H₂₂O₇

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Abstract

C₁₉H₂₂O₇, orthorhombic, *P*₂₁ (no. 4), *a* = 6.4154(1) Å, *b* = 7.8782(2) Å, *c* = 17.1555(4) Å, β = 91.021(2)°, *V* = 866.93(3) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0326, *wR*_{ref}(*F*²) = 0.1028, *T* = 150 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.10 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ:	0.89 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	73.8°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6173, 3151, 0.024
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3114
<i>N</i> (<i>param</i>) _{refined} :	238
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

Source of material

The dry roots of *Croton crassifolius* were collected in Yulin City, Guangxi province of China. The dried powder

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}
C1	0.1727 (3)	0.3226 (3)	0.26936 (11)	0.0229 (4)
H1A	0.286006	0.246140	0.283112	0.027*
H1B	0.121712	0.373189	0.316913	0.027*
C2	−0.0025 (3)	0.2241 (3)	0.22853 (11)	0.0241 (4)
H2A	−0.117621	0.300302	0.216859	0.029*
H2B	−0.052540	0.136171	0.263080	0.029*
C3	0.0720 (3)	0.1430 (3)	0.15290 (11)	0.0224 (4)
H3A	0.169994	0.052930	0.165631	0.027*
H3B	−0.046339	0.092233	0.125748	0.027*
C4	0.1755 (2)	0.2683 (2)	0.09926 (10)	0.0183 (4)
C5	0.3330 (2)	0.3896 (2)	0.13872 (10)	0.0176 (4)
C6	0.3459 (3)	0.5350 (3)	0.07844 (10)	0.0211 (4)
H6	0.450401	0.508575	0.039280	0.025*
C7	0.3889 (3)	0.7086 (3)	0.11422 (11)	0.0242 (4)
H7A	0.258524	0.761979	0.128173	0.029*
H7B	0.457381	0.780583	0.076640	0.029*
C8	0.5287 (3)	0.6879 (3)	0.18714 (11)	0.0220 (4)
H8	0.648223	0.618810	0.171873	0.026*
C9	0.4130 (3)	0.5866 (2)	0.25202 (10)	0.0195 (4)
C10	0.2514 (3)	0.4615 (3)	0.21503 (10)	0.0181 (4)
H10	0.129190	0.530203	0.200773	0.022*
C11	0.5708 (3)	0.5098 (3)	0.31072 (11)	0.0219 (4)
H11A	0.576543	0.387393	0.305068	0.026*
H11B	0.708946	0.555834	0.302609	0.026*
C12	0.4925 (3)	0.5583 (3)	0.39192 (11)	0.0241 (4)
H12	0.412775	0.463191	0.413010	0.029*
C13	0.6560 (3)	0.6092 (3)	0.44987 (12)	0.0270 (4)
C14	0.8309 (4)	0.7197 (4)	0.43960 (13)	0.0383 (6)
H14	0.865659	0.777525	0.394381	0.046*
C15	0.9344 (4)	0.7232 (4)	0.50838 (15)	0.0454 (7)
H15	1.054994	0.785795	0.518192	0.054*
C16	0.6696 (4)	0.5560 (4)	0.52442 (13)	0.0361 (5)
H16	0.575160	0.482772	0.547498	0.043*
C17	0.6152 (3)	0.8585 (3)	0.21458 (14)	0.0328 (5)
H17A	0.501939	0.934250	0.225111	0.049*
H17B	0.700397	0.906155	0.174686	0.049*
H17C	0.697741	0.842404	0.261226	0.049*
C18	0.0313 (3)	0.3946 (3)	0.05772 (10)	0.0197 (4)
C19	0.2889 (3)	0.7093 (3)	0.30239 (11)	0.0223 (4)
O1	0.8399 (3)	0.6234 (3)	0.56159 (10)	0.0464 (5)
O2	0.3517 (2)	0.7019 (2)	0.37804 (8)	0.0284 (3)
O3	0.1504 (2)	0.8027 (2)	0.28138 (9)	0.0315 (3)
O4	0.52663 (19)	0.31036 (19)	0.15480 (8)	0.0225 (3)
H4	0.573903	0.272281	0.114290	0.034*
O5	0.2892 (2)	0.18688 (19)	0.03852 (8)	0.0244 (3)
H5	0.223726	0.104077	0.022611	0.037*
O6	0.1360 (2)	0.5382 (2)	0.04224 (8)	0.0245 (3)
O7	−0.1491 (2)	0.37937 (19)	0.03756 (8)	0.0246 (3)

(10.0 kg) was extracted three times with 95% ethanol at room temperature. The solution was evaporated under reduced pressure to get a residue (1.5 kg). The crude extract was suspended in water and partitioned with

petroleum ether, dichloromethane and ethyl acetate. The dichloromethane extract was chromatographed on silica gel column chromatography using the petroleum ether-ethyl acetate solvent system, which afforded seven fractions (Fr. 1–7). The title compound was obtained from Fr. 6 and crystallized in dichloromethane.

Experimental details

Using OLEX2 [1], the structure was solved with the SHELXT program [2].

Comment

The plant *C. crassifolius* belongs to Euphorbiaceae family. It is widely distributed in the south and southwest area of China, such as Guangxi, Guangdong and Hainan provinces [5]. Previous chemical researches of *C. crassifolius* have revealed that clerodane-type diterpenoids are the main bioactive natural products of this plant [6, 7]. The roots of the *C. crassifolius* are used as a traditional medicine for treatment of joint pain, rheumatic arthritis, stomach ache, pharyngitis and jaundice [8]. Because of their interesting structural diversity and remarkable biological characteristics, clerodane-type diterpenoids have become increasing attractive research targets [9].

The title compound [10], is made of one clerodane diterpenoid which containing two lactones and two hydroxy groups. The C=O bond lengths 1.204–1.208 Å, C–C and C–O bond lengths are within normal range [11, 12].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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