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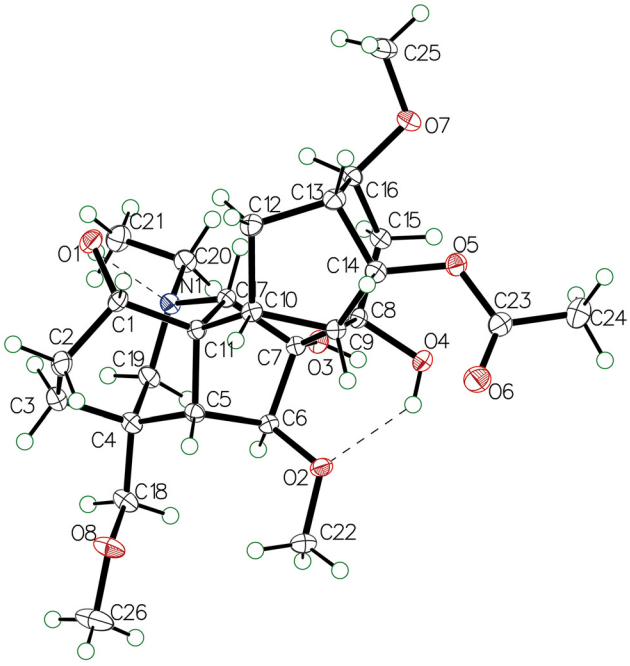
Crystal structure of 14-O-acetyldelcosine, $C_{26}H_{41}NO_8$



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Abstract

$C_{26}H_{41}NO_8$, orthorhombic, $P2_12_12_1$ (no. 19), a = 8.24370(10) Å, b = 13.1867(2) Å, c = 22.8490(4) Å, V = 2483.85(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0357, $wR_{ref}(F^2)$ = 0.0975, T = 169.99(10) 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.13 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.80 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.7°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,294, 4713, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4616
$N(param)_{refined}$:	325
Programs:	CrysAlis^{PRO} [1], Olex2 [2, 3], SHELX [4]

Source of material

The dry seeds of *Delphinium grandiflorum* were purchased from Kaiyuan, Liaoning Province, China. Dried powder (50.0 kg) was extracted three times with 95% ethanol at room temperature. The solution was evaporated under reduced pressure to get a residue (6.5 kg). The crude extract was suspended in water and we adjusted pH to 2–3 with HCl, and then partitioned with dichloromethane. The aqueous layer was adjusted to pH 9–10 with ammonium hydroxide and extracted by dichloromethane. After evaporation the dichloromethane soluble fraction (400 g) was obtained. This fraction was chromatographed by macroporous resins, eluted with EtOH/H_2O solvent system, which afforded four fractions (fr. 1–4). The title compound was obtained from fr.4 and crystallized in methanol.

Experimental details

A suitable crystal was selected and mounted on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer. Using Olex2 [2], the structure was solved with the ShelXT [3] and refined with ShelXL [4]. Refinement package using least squares.

Comment

The plant *D. grandiflorum* belongs to Ranunculaceae Juss. It is originated in southern Europe and is now widely

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7956 (2)	0.50370 (17)	0.39373 (10)	0.0256 (4)
H1A	0.847524	0.458957	0.365058	0.031*
C2	0.8512 (3)	0.4694 (2)	0.45373 (11)	0.0338 (5)
H2A	0.828855	0.397648	0.458469	0.041*
H2B	0.967391	0.479378	0.457309	0.041*
C3	0.7647 (3)	0.5288 (2)	0.50112 (10)	0.0344 (5)
H3A	0.801845	0.598540	0.500115	0.041*
H3B	0.793543	0.500744	0.538956	0.041*
C4	0.5766 (3)	0.52728 (18)	0.49463 (10)	0.0268 (5)
C5	0.5232 (2)	0.45535 (16)	0.44418 (9)	0.0217 (4)
H5	0.548326	0.384413	0.453196	0.026*
C6	0.3401 (2)	0.47056 (15)	0.43253 (9)	0.0208 (4)
H6	0.293106	0.505222	0.466480	0.025*
C7	0.3312 (2)	0.54438 (16)	0.37903 (9)	0.0194 (4)
C8	0.2940 (2)	0.48856 (15)	0.32082 (9)	0.0211 (4)
C9	0.4078 (2)	0.39600 (16)	0.31399 (9)	0.0215 (4)
H9	0.363592	0.337474	0.335077	0.026*
C10	0.5832 (2)	0.41749 (16)	0.33411 (9)	0.0224 (4)
H10	0.631842	0.352317	0.344934	0.027*
C11	0.6071 (2)	0.49028 (16)	0.38642 (9)	0.0206 (4)
C12	0.6715 (3)	0.45519 (18)	0.27704 (9)	0.0272 (5)
H12A	0.715712	0.522637	0.282732	0.033*
H12B	0.759170	0.409579	0.266608	0.033*
C13	0.5408 (3)	0.45607 (18)	0.22913 (9)	0.0266 (5)
H13	0.588187	0.441289	0.190727	0.032*
C14	0.4326 (3)	0.36978 (17)	0.24931 (10)	0.0257 (4)
H14	0.491369	0.305424	0.246022	0.031*
C15	0.2974 (3)	0.56062 (16)	0.26763 (9)	0.0242 (4)
H15A	0.203764	0.545568	0.243457	0.029*
H15B	0.284484	0.629387	0.281961	0.029*
C16	0.4488 (3)	0.55801 (18)	0.22823 (9)	0.0257 (4)
H16	0.523110	0.611627	0.240981	0.031*
C17	0.5075 (2)	0.58758 (16)	0.37614 (9)	0.0196 (4)
H17	0.529234	0.613961	0.336820	0.024*
C18	0.5034 (3)	0.49664 (19)	0.55309 (10)	0.0312 (5)
H18A	0.536570	0.544193	0.583183	0.037*
H18B	0.385926	0.497936	0.550526	0.037*
C19	0.5151 (3)	0.63504 (17)	0.48061 (9)	0.0271 (5)
H19A	0.398644	0.637759	0.486562	0.033*
H19B	0.565066	0.683011	0.507263	0.033*
C20	0.4967 (3)	0.76764 (16)	0.40648 (10)	0.0273 (5)
H20A	0.387847	0.776995	0.421675	0.033*
H20B	0.492477	0.776065	0.364331	0.033*
C21	0.6074 (3)	0.84748 (18)	0.43235 (12)	0.0350 (5)
H21A	0.574192	0.913334	0.419032	0.053*
H21B	0.717002	0.834855	0.420135	0.053*
H21C	0.601303	0.844901	0.474276	0.053*
C22	0.2288 (3)	0.3225 (2)	0.47661 (11)	0.0367 (6)
H22A	0.172823	0.260315	0.468307	0.055*
H22B	0.165421	0.362483	0.503286	0.055*
H22C	0.332150	0.307652	0.493932	0.055*
C23	0.1832 (3)	0.28837 (18)	0.22702 (10)	0.0302 (5)
C24	0.0282 (3)	0.2997 (2)	0.19410 (13)	0.0417 (6)
H24A	−0.009197	0.368431	0.197165	0.063*
H24B	−0.051791	0.254807	0.210245	0.063*
H24C	0.045827	0.283149	0.153682	0.063*
C25	0.5133 (4)	0.6211 (2)	0.13365 (11)	0.0407 (6)
H25A	0.465460	0.645563	0.098029	0.061*
H25B	0.589279	0.568153	0.124785	0.061*
H25C	0.568554	0.675751	0.152987	0.061*
C26	0.5076 (5)	0.3691 (3)	0.62518 (13)	0.0576 (9)
H26A	0.391674	0.373603	0.628317	0.086*
H26B	0.556884	0.413509	0.653298	0.086*
H26C	0.541259	0.300577	0.632617	0.086*
N1	0.5524 (2)	0.66417 (13)	0.42028 (8)	0.0223 (4)
O1	0.85168 (19)	0.60412 (13)	0.38212 (8)	0.0311 (4)
H1	0.794405	0.645016	0.399559	0.047*
O2	0.25227 (18)	0.37757 (12)	0.42371 (7)	0.0262 (3)
O3	0.21858 (17)	0.62386 (11)	0.38845 (7)	0.0232 (3)
H3	0.126230	0.602606	0.383232	0.035*
O4	0.12791 (17)	0.45398 (12)	0.32342 (7)	0.0255 (3)
H4	0.114672	0.419847	0.353031	0.038*
O5	0.2857 (2)	0.36506 (12)	0.21506 (7)	0.0282 (3)
O6	0.2158 (3)	0.22038 (14)	0.26007 (9)	0.0428 (4)
O7	0.3905 (2)	0.58278 (14)	0.17064 (7)	0.0351 (4)
O8	0.5563 (2)	0.39794 (15)	0.56787 (7)	0.0390 (4)

distributed throughout China [5]. Previous chemical investigations of *D. grandiflorum* have revealed that diterpene alkaloids are the main chemical constituents of this plant [6]. The single crystals were obtained by slow evaporation from a methanol solution.

The title compound was determined structurally as 14-O-acetyldelcosine [7], which is belonging to a class of diterpene alkaloids. The C=O bond length is 1.203 Å, the C–O bond lengths have values of 1.346(2)–1.444(8) Å, and the C–N and C–C bond lengths are within normal ranges [8, 9].

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

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