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Isolation and crystal structure of 4-((2-(methoxycarbonyl)phenyl)amino)-2-methyl-4-oxobutanoic acid from *Delphinium Grandiflorum*, C₁₃H₁₅N₁O₅

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.24 × 0.24 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.86 mm ⁻¹
Diffractometer, scan mode:	Gemini S ultra, ω
$\theta_{\max}$, completeness:	62.8°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4205, 2102, 0.023
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1836
$N(\text{param})_{\text{refined}}$:	178
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

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Abstract

C₁₃H₁₅N₁O₅, triclinic, $P\bar{1}$ (no. 2), $a = 7.9457(7)$ Å, $b = 8.7190(9)$ Å, $c = 9.9964(10)$ Å, $\alpha = 100.254(8)^\circ$, $\beta = 100.036(8)^\circ$, $\gamma = 94.760(8)^\circ$, $V = 666.38(11)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0453$, $wR_{\text{ref}}(F^2) = 0.1243$, $T = 298$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The dry rhizomes of *Delphinium grandiflorum* were purchased from Guangzhou qingping medicine market, Guangdong,

China. The dry powder (10 kg) was extracted three times with 95% ethanol at room temperature. The solution was concentrated in vacuum to get a residue (492 g). The crude extract was suspended in water and adjusted pH to 4–5 with HCl, and then partitioned with chloroform. The aqueous layer was adjusted pH to 9–10 with ammonium hydroxide and extracted by chloroform. After evaporation of the solvent, the chloroform soluble fraction (63 g) was obtained. This fraction was chromatographed on a silica gel column eluted with CHCl₃-CH₃OH solvent system, afforded six fractions (Fr. 1–6). The title compound was obtained from Fr.5 and crystallized in methanol.

Experimental details

The C-bound H atoms were positioned geometrically and were included in the refinement in the riding-model approximation.

Comment

Delphinium grandiflorum is a perennial herb belonging to the Ranunculaceae family. It is native to southern Europe and widely distributed in almost all provinces of China [3, 4]. Previous investigations indicate alkaloids as the main chemical constituents of genus *Delphinium* [5, 6]. As a toxic plant, the toxicity is due [7]. But the dried roots and stems of *Delphinium grandiflorum* are often used as traditional Chinese medicine to treat stomachache, asthma [8], traumatic injury [9] and toothache [10]. The title compound which was originally synthesized for the preparation of pyridazino[6,1-*b*]quinazolinones [11] is an alkaloid isolated from *Delphinium grandiflorum*.

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
O1	0.2853(3)	−0.0576(2)	−0.36828(15)	0.0879(6)
O2	0.1968(2)	0.13506(19)	−0.23470(15)	0.0832(5)
O3	0.1965(3)	0.13901(18)	0.25536(14)	0.0884(6)
O4	0.27762(19)	0.46201(19)	0.51114(14)	0.0700(4)
H4	0.394(5)	0.484(5)	0.563(4)	0.161(14)*
O5	0.40732(17)	0.49284(18)	0.33690(14)	0.0664(4)
N1	0.19438(18)	0.11165(15)	0.02634(14)	0.0452(4)
H1	0.1655	0.1568	−0.0427	0.054*
C1	0.2869(2)	−0.0849(2)	−0.14020(17)	0.0448(4)
C2	0.26012(19)	−0.03218(18)	−0.00531(17)	0.0412(4)
C3	0.3020(2)	−0.1243(2)	0.09368(18)	0.0490(4)
H3	0.2846	−0.0914	0.1834	0.059*
C4	0.3691(2)	−0.2636(2)	0.0594(2)	0.0544(5)
H4A	0.3974	−0.3232	0.1267	0.065*
C5	0.3946(2)	−0.3157(2)	−0.0724(2)	0.0579(5)
H5	0.4390	−0.4103	−0.0947	0.070*
C6	0.3538(2)	−0.2266(2)	−0.1711(2)	0.0542(5)
H6	0.3713	−0.2618	−0.2605	0.065*
C7	0.2502(2)	0.0096(2)	−0.24936(19)	0.0539(5)
C8	0.2589(5)	0.0282(4)	−0.4805(3)	0.1086(11)
H8A	0.2872	−0.0323	−0.5619	0.163*
H8B	0.3313	0.1266	−0.4542	0.163*
H8C	0.1406	0.0471	−0.4996	0.163*
C9	0.1701(2)	0.1891(2)	0.14937(17)	0.0473(4)
C10	0.1076(2)	0.3475(2)	0.14634(18)	0.0488(4)
H10A	−0.0062	0.3335	0.0885	0.059*
H10B	0.1842	0.4096	0.1054	0.059*
C11	0.1002(2)	0.4355(2)	0.29040(19)	0.0506(4)
H11	0.0240	0.3710	0.3312	0.061*
C12	0.2758(2)	0.4640(2)	0.38131(18)	0.0503(4)
C13	0.0291(3)	0.5926(2)	0.2852(2)	0.0688(6)
H13A	0.1050	0.6587	0.2488	0.103*
H13B	0.0205	0.6428	0.3770	0.103*
H13C	−0.0828	0.5747	0.2266	0.103*

The title compound is composed of one phenyl moiety, one ester group, one carboxyl group and one amide group. The carbon atoms C1, C2, C3, C4, C5, C6 are coplanar (mean deviation 0.002 Å). The dihedral angle between this plane as expected and the ester group at C1 and the amide backbone of the molecule (N1–C9–C10–C11–C13) is nearly zero. In accord with the sp³ character of C11 the carbonyl group is not located in the aforementioned plane. A short intramolecular hydrogen bonding interaction between amide group and O acceptor of ester group is present.

Bond lengths and angles are all in the expected ranges [12]. Molecules are pairwise hydrogen bonded *via* the carboxylic group to form the typical dimers in the solid state [13].

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