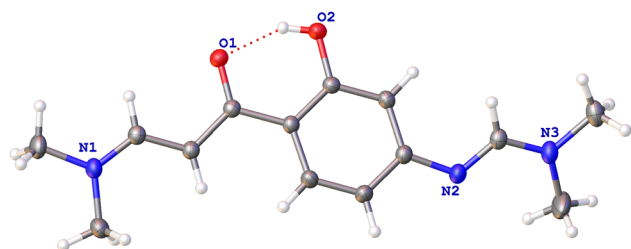


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Crystal structure of (*E*)-*N*'-(4-((*E*)-3-(dimethylamino)acryloyl)-3-hydroxyphenyl)-*N*, *N*-dimethylformimidamide, C₁₄H₁₉N₃O₂



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Abstract

C₁₄H₁₉N₃O₂, monoclinic, *P*2₁/*c* (no. 14), *a* = 8.62239(12) Å, *b* = 15.3515(3) Å, *c* = 10.86408(16) Å, β = 99.5170(14)°, *V* = 1418.25(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0452, *wR*_{ref}(*F*²) = 0.1354, *T* = 295.52(10) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

In our research on the identification of anti-rot genes of *pinellia ternata* in Guizhou Province, we selected the hydroxyl aromatic ketonether derivate present in *pinellia ternata* as the raw material to synthesize the title compound. 1-(4-amino-2-hydroxyphenyl)ethan-1-one (3.02 g, 20 mmol) and dimethylformamide dimethylacetal (9.0 mL) were combined. The colorless solution heated to 383 K. Thin layer chromatography tracked the progress of the reaction until

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.15 × 0.12 × 0.10 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ:	0.68 mm ⁻¹
Diffraction, scan mode:	XtaLAB synergy, ω
θ _{max} , completeness:	73.1°, >99 %
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	9567, 2757, 0.028
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 2251
<i>N</i> (<i>param</i>) _{refined} :	178
Programs:	Olex2 [1], SHELX [2, 3]

the 1-(4-amino-2-hydroxyphenyl)ethan-1-one is complete and the reaction is over. Then the reddish brown solution precipitated red solid when cooled to room temperature. The mixture was filtered and concentrated in vacuo to afford a solid. The solid was recrystallized from ethanol.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Enaminone compounds and their derivatives are widely used in the field of organic chemistry, pharmaceutical chemistry, which are used as intermediates to synthesize many active drugs [4, 5]. Some enaminone compounds can be used as potential anticonvulsants and as potential multidrug resistance regulator [6–8]. In the field of anti-inflammatory and antiepileptic drug research, they play an important role [9]. In addition, in our research of the identification of antiseptic genes of *pinellia*, an important national Chinese medicinal material, adopting functional genomics approach, collecting *pinellia* fine medicinal materials from Hezhang, Dafang, and other places in Guizhou Province, China. We find that enaminone compounds are

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.02771 (13)	0.32428 (8)	0.48362 (9)	0.0594 (3)
O2	0.27989 (13)	0.40933 (9)	0.51220 (10)	0.0647 (3)
H2	0.193449	0.391180	0.528105	0.097 [*]
N1	−0.32479 (15)	0.17818 (9)	0.28474 (13)	0.0582 (4)
N2	0.57911 (15)	0.41763 (10)	0.17527 (12)	0.0608 (4)
N3	0.81735 (16)	0.48806 (10)	0.19682 (15)	0.0654 (4)
C1	0.29913 (16)	0.38163 (10)	0.39756 (12)	0.0475 (3)
C2	0.42987 (16)	0.41112 (10)	0.35078 (13)	0.0508 (4)
H2A	0.502497	0.448749	0.400149	0.061 [*]
C3	0.45649 (16)	0.38653 (10)	0.23281 (14)	0.0506 (4)
C4	0.34603 (19)	0.33107 (12)	0.16210 (15)	0.0579 (4)
H4	0.361423	0.313230	0.081264	0.069 [*]
C5	0.21681 (17)	0.30253 (11)	0.20828 (14)	0.0541 (4)
H5	0.144600	0.264923	0.158454	0.065 [*]
C6	0.18717 (15)	0.32686 (9)	0.32664 (13)	0.0455 (3)
C7	0.04368 (16)	0.30043 (9)	0.37482 (13)	0.0461 (3)
C8	−0.07478 (16)	0.25117 (10)	0.29790 (13)	0.0484 (3)
H8	−0.064512	0.238499	0.214028	0.058 [*]
C9	−0.20297 (17)	0.22217 (10)	0.34463 (14)	0.0513 (4)
H9	−0.205108	0.234985	0.429883	0.062 [*]
C10	−0.3396 (2)	0.15943 (15)	0.15339 (17)	0.0749 (5)
H10A	−0.339274	0.214067	0.106787	0.112 [*]
H10B	−0.438454	0.128370	0.125522	0.112 [*]
H10C	−0.251089	0.123086	0.138512	0.112 [*]
C11	−0.4535 (2)	0.15026 (15)	0.3473 (2)	0.0783 (6)
H11A	−0.423166	0.157886	0.437602	0.117 [*]
H11B	−0.476865	0.088719	0.328674	0.117 [*]
H11C	−0.547121	0.185419	0.317551	0.117 [*]
C12	0.70478 (17)	0.44671 (10)	0.24378 (15)	0.0533 (4)
H12	0.717558	0.438264	0.331498	0.064 [*]
C13	0.9605 (2)	0.51704 (13)	0.2748 (2)	0.0769 (5)
H13A	0.965965	0.580770	0.272654	0.115 [*]
H13B	1.051425	0.492356	0.243670	0.115 [*]
H13C	0.960955	0.497617	0.360751	0.115 [*]
C14	0.7985 (3)	0.5093 (3)	0.0665 (2)	0.1249 (12)
H14A	0.840575	0.461815	0.021481	0.187 [*]
H14B	0.855554	0.563196	0.055677	0.187 [*]
H14C	0.686622	0.517256	0.033528	0.187 [*]

also present in *Guizhou's pinellia ternata*. Enaminone compounds are also used as raw material to synthesize flavonoids in *pinellia ternata*. Therefore, the synthesis and modification of enaminone compounds has been a hot topic. The structure of the title compound is a kind of enaminone, containing a benzene ring, a hydroxyl group, a ketone and two enamines. The bond lengths and angles derived from the title structure are within normal ranges [10, 11]. The keto group was confirmed by the distance of 1.2664(17) Å (C7–O1), the phenolic hydroxyl was confirmed by the distance of 1.3520(17) Å (C1–O2), the C8=C9 double bond adopts an *E*-configuration and the bond distance is 1.365(2) Å, the bond

distance of the N2=C12 double bond is 1.289(2) Å. The torsion angles of O2–C1–C2–C3 and N2–C3–C4–C5 are −179.04(14)° and −175.56(15)°, respectively.

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