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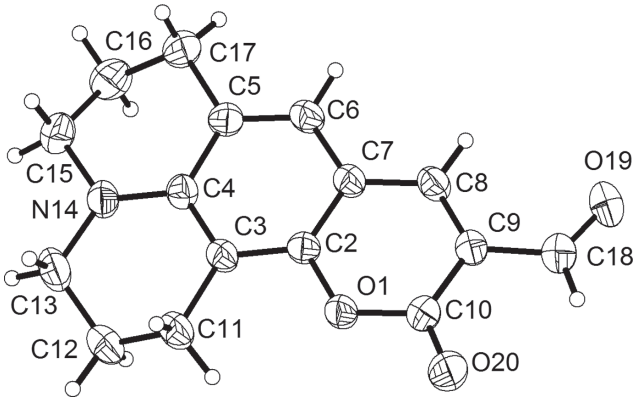
# Crystal structure of 11-oxo-2,3,6,7-tetrahydro-1*H*, 5*H*,11*H*-pyrano[2,3-*f*]pyrido[3,2,1-*ij*]quinoline-10-carbaldehyde - a julolidine derivative, $C_{16}H_{15}NO_3$

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## Abstract

$C_{16}H_{15}NO_3$, monoclinic, $P2_1/c$ (no. 14), $a = 9.802(1)$ Å, $b = 14.492(1)$ Å, $c = 9.667(1)$ Å, $\beta = 112.027(1)°$, $V = 1272.9$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0422$, $wR_{ref}(F^2) = 0.1249$, $T = 296$ K.

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The crystal structure is shown in the figure. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. 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 title compound was synthesized according to standard Vilsmeier-Haack conditions, under which formylation at 3-position of julolidine [2,3]quinolone occurred efficiently [5, 6]. All chemicals used were commercially available of AR grade, and were used as received without further purification. The purple crystals of the title compound were obtained by slow evaporation of an ethanol solution at room temperature.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | purple block |
| Size: | 0.22 × 0.19 × 0.18 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.10 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART, $\varphi$ and $\omega$-scans |
| $2\theta_{max}$, completeness: | 28.3°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 34108, 3173, 0.062 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2240 |
| $N(param)_{refined}$: | 186 |
| Programs: | Bruker [1], SHELX [2], Diamond [3], ORTEP, WinGX [4] |

## Experimental details

The hydrogen atoms were placed geometrically and refined using a riding model with d(C—H) = 0.93 Å (aromatic), 0.97 Å (-$CH_2$-), 0.925 Å (-COH). $U_{iso}(H) = 1.2\ U_{eq}(C)$ for COH and $CH_2$ groups.

## Discussion

Fluorescent molecules fascinate the physicists and chemists owing to the application of fluorescence signal in smart material artificial intelligence. Intensive effort and development have been made and engineered according to requirement of device fabrication and material research [7–10].

The coumarin moiety (O1/C2/C3/C4/C5/C6/C7/C8/C9/C10) is planar with the mean deviation 0.043 Å. However, the adjacent aliphatic moiety is twisted out of the molecular plane due to the envelope conformation. Two carbonyl oxygen atoms are present in molecule, which are polarized and are gathered by more electron density. Chances are that oxygen atoms may involve H···O interactions. Actually, C—H···O, C–H···$\pi$, and $\pi$···$\pi$ interactions are predominant weak forces that joint the molecule into a crystal. All the oxygen atoms including O1, O19, and O20 are involved

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

| Atom | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.47668(10) | 0.11771(5) | 0.18064(10) | 0.0376(2) |
| C2 | 0.55368(13) | 0.18839(8) | 0.14740(13) | 0.0311(3) |
| C3 | 0.66213(13) | 0.16271(8) | 0.09744(13) | 0.0325(3) |
| C4 | 0.74475(13) | 0.23394(8) | 0.06400(12) | 0.0320(3) |
| C5 | 0.70775(14) | 0.32889(9) | 0.07403(13) | 0.0351(3) |
| C6 | 0.60049(14) | 0.34942(8) | 0.12724(13) | 0.0359(3) |
| H6 | 0.5789 | 0.4110 | 0.1367 | 0.043* |
| C7 | 0.52149(13) | 0.28073(8) | 0.16821(13) | 0.0326(3) |
| C8 | 0.41771(13) | 0.29746(8) | 0.23347(14) | 0.0348(3) |
| H8 | 0.3985 | 0.3580 | 0.2525 | 0.042* |
| C9 | 0.34402(13) | 0.22727(9) | 0.26989(13) | 0.0346(3) |
| C10 | 0.36769(14) | 0.13314(9) | 0.23612(14) | 0.0383(3) |
| C11 | 0.69726(15) | 0.06282(9) | 0.08249(17) | 0.0450(3) |
| H11A | 0.6368 | 0.0407 | −0.0162 | 0.054* |
| H11B | 0.6749 | 0.0261 | 0.1552 | 0.054* |
| C12 | 0.85793(16) | 0.05188(10) | 0.10690(17) | 0.0496(4) |
| H12A | 0.9181 | 0.0650 | 0.2103 | 0.060* |
| H12B | 0.8768 | −0.0113 | 0.0859 | 0.060* |
| C13 | 0.89856(17) | 0.11638(9) | 0.00698(17) | 0.0484(4) |
| H13A | 1.0034 | 0.1119 | 0.0291 | 0.058* |
| H13B | 0.8471 | 0.0982 | −0.0962 | 0.058* |
| N14 | 0.86116(12) | 0.21214(7) | 0.02647(12) | 0.0389(3) |
| C15 | 0.94382(16) | 0.28225(10) | −0.01761(16) | 0.0454(3) |
| H15A | 0.9008 | 0.2901 | −0.1250 | 0.054* |
| H15B | 1.0444 | 0.2613 | 0.0085 | 0.054* |
| C16 | 0.94457(15) | 0.37357(10) | 0.05624(17) | 0.0506(4) |
| H16A | 0.9925 | 0.4195 | 0.0170 | 0.061* |
| H16B | 0.9999 | 0.3682 | 0.1627 | 0.061* |
| C17 | 0.78955(15) | 0.40408(9) | 0.02897(17) | 0.0455(3) |
| H17A | 0.7386 | 0.4184 | −0.0759 | 0.055* |
| H17B | 0.7920 | 0.4595 | 0.0861 | 0.055* |
| C18 | 0.24370(16) | 0.24397(11) | 0.34589(15) | 0.0435(3) |
| H18 | 0.2000(19) | 0.1914(12) | 0.3647(18) | 0.069(5)* |
| O19 | 0.21677(11) | 0.31875(7) | 0.38520(11) | 0.0536(3) |
| O20 | 0.30346(12) | 0.06559(7) | 0.25281(13) | 0.0588(3) |

in C—H···O interactions: C11–H11A···O1$^{i1}$ ($^{i1}$: $x$, $y$, $1+z$), C12—H12B···O19$^{i2}$ ($^{i2}$: $x$, $1/2-y$, $1/2+z$), C15–H15B···O19$^{i2}$, C16—H16B···O19$^{i2}$, C6–H6···O20$^{i3}$ ($^{i3}$: $x$, $y$, $1+z$), and C17—H17B···O20$^{i3}$. The distance of C···O varied from 3.53 to 3.75 Å, which falls inside the normal C···O interval of C–H···O interactions [11]. The H···O distances range from 2.50 to 2.78 Å and the C—H···O angles vary from 138° to 153°, indicating very weak H···O interactions. It is worthy to note that the H···O interactions with O19 and O20 involve in the crystal are more complicated than the interaction with O1, in which one oxygen atom involve two or more hydrogen bonds. The carbonyl C18 = O19, hovering above the carbon ring (O1/C2/C7/C8/C9C10), configure the $\pi\cdots\pi$ interactions.

Apart from the interactions mentioned above, more weaker interactions that cannot be neglected are C—H···$\pi$ interactions, to which hydrogen atoms of methylene contribute and interact with the coumarin ring.

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