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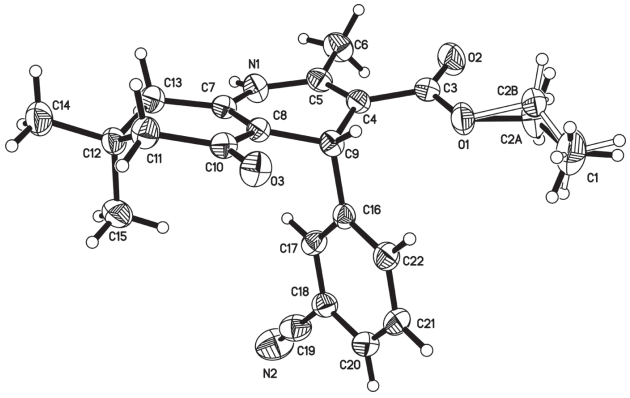
# Crystal structure of ethyl 4-(3-cyanophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{22}H_{24}N_2O_3$

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## Abstract

$C_{22}H_{24}N_2O_3$, orthorhombic, *Pbca* (no. 61), $a = 15.333(11)$ Å, $b = 14.146(11)$ Å, $c = 17.640(13)$ Å, $V = 3826(5)$ Å$^3$, $Z = 8$, $R_{gt}(F) = 0.0533$, $wR_{ref}(F^2) = 0.1431$, $T = 296$ K.

**CCDC no.:** 1848572

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 title compound was synthesized according to a reported procedure. A mixture of 1,1-dimethyl-3,5-cyclohexanedione (10 mmol), 3-cyanobenzaldehyde (10 mmol), ammonium acetate (10 mmol) and ethyl acetoacetate (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

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

| | |
|---|---|
| Crystal: | Colorless block |
| Size: | 0.36 × 0.25 × 0.21 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.09 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$-scans |
| $\theta_{max}$, completeness: | 25°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 18335, 3372, 0.079 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1943 |
| $N(param)_{refined}$: | 255 |
| Programs: | Bruker programs [1], SHELX [2], OLEX2 [3] |

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

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.32956(13) | 0.58901(13) | 0.10504(11) | 0.0514(6) |
| H1 | 0.320180 | 0.529233 | 0.101508 | 0.062* |
| N2 | 0.2605(2) | 0.5691(2) | 0.43772(15) | 0.0988(10) |
| O1 | 0.52090(12) | 0.83281(13) | 0.16794(11) | 0.0681(6) |
| O2 | 0.58406(12) | 0.70082(15) | 0.12926(12) | 0.0807(6) |
| O3 | 0.20893(11) | 0.89045(11) | 0.11989(9) | 0.0582(5) |
| C1 | 0.6046(2) | 0.9561(2) | 0.2156(2) | 0.1064(12) |
| H1BD[a] | 0.560016 | 0.999720 | 0.200229 | 0.160* |
| H1BE[a] | 0.660292 | 0.987079 | 0.214292 | 0.160* |
| H1BF[a] | 0.592902 | 0.934638 | 0.266236 | 0.160* |
| H1AA[b] | 0.587874 | 0.997048 | 0.174722 | 0.160* |
| H1AB[b] | 0.660845 | 0.974679 | 0.234329 | 0.160* |
| H1AC[b] | 0.562454 | 0.960645 | 0.255702 | 0.160* |
| C2B[a] | 0.6054(6) | 0.8804(10) | 0.1673(10) | 0.088(4) |
| H2BA[a] | 0.650600 | 0.836331 | 0.182637 | 0.106* |
| H2BB[a] | 0.618582 | 0.901759 | 0.116292 | 0.106* |
| C2A[b] | 0.6084(4) | 0.8642(5) | 0.1899(8) | 0.067(3) |
| H2AA[b] | 0.630977 | 0.823498 | 0.229599 | 0.080* |
| H2AB[b] | 0.647329 | 0.860282 | 0.146731 | 0.080* |
| C3 | 0.51850(18) | 0.74435(19) | 0.14325(13) | 0.0533(7) |
| C4 | 0.42901(15) | 0.70986(16) | 0.13576(12) | 0.0442(6) |
| C5 | 0.41401(16) | 0.61992(17) | 0.11569(13) | 0.0483(6) |
| C6 | 0.48126(18) | 0.54555(19) | 0.10128(16) | 0.0696(8) |
| H6A | 0.452950 | 0.487398 | 0.087794 | 0.104* |
| H6B | 0.518553 | 0.565305 | 0.060539 | 0.104* |
| H6C | 0.515449 | 0.536180 | 0.146244 | 0.104* |
| C7 | 0.26047(16) | 0.64904(15) | 0.09986(12) | 0.0412(6) |
| C8 | 0.27122(15) | 0.74053(14) | 0.11798(11) | 0.0386(5) |
| C9 | 0.35333(15) | 0.77550(15) | 0.15494(12) | 0.0424(6) |

**Table 2** (continued)

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| H9 | 0.366581 | 0.838262 | 0.134434 | 0.051* |
| C10 | 0.20137(16) | 0.80639(16) | 0.10442(12) | 0.0440(6) |
| C11 | 0.11860(16) | 0.76911(17) | 0.06928(14) | 0.0557(7) |
| H11A | 0.123122 | 0.774548 | 0.014607 | 0.067* |
| H11B | 0.070275 | 0.808446 | 0.085530 | 0.067* |
| C12 | 0.09845(16) | 0.66743(17) | 0.08919(13) | 0.0499(6) |
| C13 | 0.17782(15) | 0.60817(16) | 0.07083(14) | 0.0503(7) |
| H13A | 0.170139 | 0.545630 | 0.092368 | 0.060* |
| H13B | 0.182147 | 0.601201 | 0.016250 | 0.060* |
| C14 | 0.02069(18) | 0.6328(2) | 0.04277(17) | 0.0739(8) |
| H14A | 0.008253 | 0.568228 | 0.055667 | 0.111* |
| H14B | −0.029303 | 0.671272 | 0.053709 | 0.111* |
| H14C | 0.034244 | 0.637001 | −0.010257 | 0.111* |
| C15 | 0.07613(19) | 0.6596(2) | 0.17302(15) | 0.0717(9) |
| H15A | 0.063421 | 0.594967 | 0.185266 | 0.108* |
| H15B | 0.124774 | 0.680894 | 0.202745 | 0.108* |
| H15C | 0.026125 | 0.698137 | 0.183932 | 0.108* |
| C16 | 0.34158(14) | 0.78474(16) | 0.24036(12) | 0.0429(6) |
| C17 | 0.31607(16) | 0.70796(17) | 0.28251(13) | 0.0479(6) |
| H17 | 0.305503 | 0.650652 | 0.258362 | 0.057* |
| C18 | 0.30579(16) | 0.71433(19) | 0.36030(13) | 0.0516(7) |
| C19 | 0.2795(2) | 0.6339(2) | 0.40354(16) | 0.0696(8) |
| C20 | 0.32080(17) | 0.7986(2) | 0.39636(15) | 0.0644(8) |
| H20 | 0.314838 | 0.803093 | 0.448701 | 0.077* |
| C21 | 0.34453(19) | 0.8757(2) | 0.35491(17) | 0.0705(9) |
| H21 | 0.353759 | 0.933272 | 0.379089 | 0.085* |
| C22 | 0.35497(17) | 0.86915(18) | 0.27748(15) | 0.0601(7) |
| H22 | 0.371256 | 0.922381 | 0.249961 | 0.072* |

Occupancies: [a] = 0.49(3), [b] = 51(3)

## Experimental details

H atoms bonded to C were positioned geometrically and refined using a riding model, with C—H = 0.96 or 0.97 Å and with $U_{iso}$(H) = 1.2 times $U_{eq}$(C). There is a disorder of the ethyl group (*cf.* the figure).

## Discussion

4-Arylpolyhydroquinoline derivatives form an important class of compounds. These compounds may possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [4]. A large number of 4-arylpolyhydroquinoline derivatives have been reported to show substantial cytotoxic activity *in vitro* and *in vivo* [5, 6]. Recognizing the considerable importance of the compounds, the research focused on the synthesis of the 4-arylpolyhydroquinoline derivatives.

In the crystal structure of the title compound (*cf.* the figure), the six-membered ring containing nitrogen atom is nearly planar and the adjacent cyclohex-2-en-1-one adopts a flattened chair conformation. The 1,4-hydropyridine moiety is almost perpendicular to the cyanophenyl ring and is almost coplanar with the mean plane of the cyclohex-2-ene ring.

## References

1. Bruker. *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, WI, USA (2012).
2. Sheldrick, G. M.: A short history of *SHELX*. Acta Crystallogr. **A64** (2008) 112–122.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Crystallogr. **42** (2009) 339–341.
4. Smyth, T.; Ramachandran, V. N.; Smyth, W. F.: A study of the antimicrobial activity of naturally occurring and synthetic coumarins. Int. J. Antimicrob. Agents **33** (2009) 421–426.
5. Li, M.; Li, J.; Liu, B.; Zhou, Y.; Li, X.; Xue, X.; Hou, Z.; Luo, X.: Synthesis, crystal structures, and anti-drug-resistant Staphylococcus aureus activities of novel 4-hydroxycoumarin derivatives. Eur. J. Pharmacol. **721** (2013) 151–157.
6. Iranshahi, M.; Askari, M.; Sahebkar, A.; Hadjipavlou-Litina, D.: Evaluation of antioxidant, anti-inflammatory and lipoxygenase inhibitory activities of the prenylated coumarin umbelliprenin. J. Pharm. Sci. **17** (2009) 99–103.