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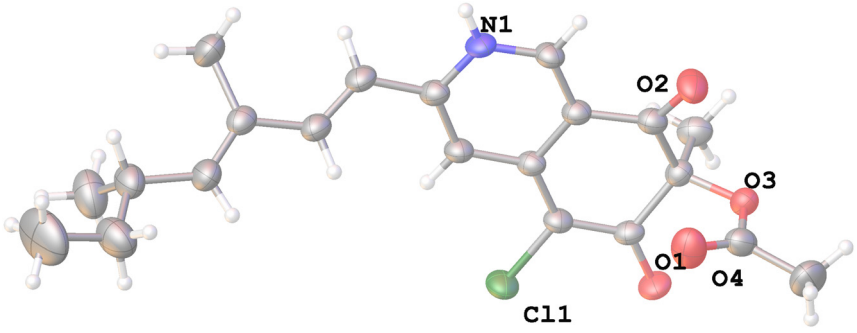
Crystal structure of (*R*)(*R*)-5-chloro-3-((*S*,1*E*,3*E*)-3,5-dimethyl-hepta-1,3-dien-1-yl)-7-methyl-6,8-dioxo-2,6,7,8-tetrahydroisoquinolin-7-yl acetate, $C_{21}H_{24}ClNO_4$



https://doi.org/10.1515/ncrs-2024-0295
Received July 11, 2024; accepted August 23, 2024;
published online September 6, 2024

Abstract

$C_{21}H_{24}ClNO_4$, monoclinic, $P2_1$ (no. 14), a = 8.6813(3) Å, b = 7.3821(2) Å, c = 16.0325(6) Å, β = 105.282(2)°, V = 991.13(6) Å^3, Z = 2, $R_{gt}(F)$ = 0.0405, w$R_{ref}(F^2)$ = 0.1014, T = 296.15 K.

CCDC no.: 2348404

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.

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Table 1: Data collection and handling.

Crystal:	Red needle
Size:	0.28 × 0.15 × 0.10 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.22 mm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
θ_{max}, completeness:	27.9°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15860, 4621, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3735
$N(param)_{refined}$:	252
Programs:	Olex2,[1,2] SHELX[3,4]

1 Source of material

The title compound is a natural product, which has been totally synthesized in 2011.[5] We got it by extraction and isolation from fungal fermentation products.[6]

2 Experimental details

The structure was treated with the Olex2 crystallographic software package,[1,2] solved with the SHELXT structure solution program and refined with the SHELXL refinement package.[3,4] Carbon-bound hydrogen atoms were placed in calculated positions and refined with riding coordinates, with U_{iso}(H) fixed at 1.2 times of U_{eq}(C) (Tables 1 and 2).

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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	0.8155 (3)	0.3956 (5)	0.36708 (17)	0.0336 (6)
C3	0.8946 (3)	0.4085 (5)	0.51986 (16)	0.0332 (6)
C4	0.7381 (3)	0.4134 (5)	0.52163 (17)	0.0340 (6)
C5	0.4504 (3)	0.4150 (5)	0.44303 (16)	0.0328 (6)
C6	0.3242 (3)	0.4171 (5)	0.36775 (16)	0.0313 (6)
C7	0.3690 (3)	0.4414 (4)	0.28128 (17)	0.0317 (6)
C8	0.5378 (3)	0.3845 (4)	0.28142 (17)	0.0337 (6)
C9	1.0284 (3)	0.4089 (5)	0.59771 (17)	0.0367 (6)
C10	1.0275 (3)	0.3142 (4)	0.66721 (19)	0.0375 (7)
C11	1.1521 (3)	0.3023 (4)	0.7484 (2)	0.0367 (7)
C12	1.1324 (4)	0.1892 (5)	0.8089 (2)	0.0451 (8)
C13	1.2414 (4)	0.1551 (6)	0.8979 (2)	0.0516 (9)
C14	1.2179 (5)	−0.0362 (7)	0.9276 (3)	0.0710 (12)
C15	1.3464 (7)	−0.0961 (11)	1.0063 (4)	0.115 (2)
C16	1.2133 (6)	0.2974 (7)	0.9604 (3)	0.0772 (13)
C17	1.2947 (4)	0.4251 (6)	0.7595 (2)	0.0539 (9)
C18	0.3543 (4)	0.6407 (4)	0.2567 (2)	0.0435 (8)
C19	0.2457 (4)	0.1689 (5)	0.2252 (2)	0.0404 (7)
C20	0.1074 (5)	0.0876 (6)	0.1604 (3)	0.0656 (11)
C21	0.6103 (3)	0.4091 (5)	0.44447 (15)	0.0301 (5)
C22	0.6582 (3)	0.3972 (5)	0.36516 (16)	0.0316 (6)
Cl1	0.39620 (8)	0.41451 (13)	0.54013 (4)	0.0456 (2)
H1	1.023 (4)	0.408 (6)	0.4420 (19)	0.043*
H7	0.714105	0.419778	0.574764	0.041*
H9	0.844461	0.388791	0.315300	0.040*
H10	1.117772	0.478740	0.598111	0.044*
H11	0.935853	0.246350	0.664171	0.045*
H13	1.038800	0.121459	0.794599	0.054*
H14	1.351979	0.166268	0.894354	0.062*
H15A	1.115170	−0.042867	0.940769	0.085*
H15B	1.215475	−0.119588	0.880576	0.085*
H16A	1.306118	−0.192321	1.034882	0.173*
H16B	1.377033	0.004212	1.045210	0.173*
H16C	1.437546	−0.138294	0.988604	0.173*
H17A	1.371654	0.397295	0.812935	0.081*
H17B	1.261343	0.548981	0.760272	0.081*
H17C	1.341920	0.407420	0.712265	0.081*
H18A	1.233217	0.415402	0.940354	0.116*
H18B	1.284197	0.276321	1.016557	0.116*
H18C	1.104755	0.290498	0.963866	0.116*
H20A	0.125220	−0.039885	0.155616	0.098*
H20B	0.096197	0.144363	0.105278	0.098*
H20C	0.011777	0.105937	0.178691	0.098*
H21A	0.373926	0.656168	0.200940	0.065*
H21B	0.431065	0.709387	0.298927	0.065*
H21C	0.248674	0.682383	0.254724	0.065*
N1	0.9305 (3)	0.4036 (4)	0.44192 (14)	0.0360 (5)
O1	0.18009 (19)	0.4126 (4)	0.36458 (12)	0.0441 (5)
O2	0.5699 (2)	0.3456 (4)	0.21459 (14)	0.0495 (7)
O3	0.2532 (2)	0.3499 (3)	0.21400 (13)	0.0357 (5)
O4	0.3378 (3)	0.0903 (3)	0.28234 (17)	0.0552 (6)

3 Comment

The title compound of $C_{21}H_{24}ClNO_4$ has been reported with various biological activities, such as neuroprotective effects, and cytotoxic against hepatoma cells.[7,8] The structure of $C_{21}H_{24}ClNO_4$ is composed of 4,6-dimethylocta-2,4-diene, pyridine ring, and *m*-benzoquinone ring moiety.[9] The geometry of title structure was characterized with the bond angles and lengths. In particular, the bond angles for C6···C7···C8, C5···C6···C7, C6···C5···Cl1 and C1···N1···C3 are 115.8(2)°, 116.7 (2)°, 115.85(17)° and 121.5(2)°, respectively. For another, the bond lengths of Cl1···C5, O1···C6, O2···C8, C5···C6 and C1···N1, are 1.741(2) Å, 1.239(3) Å, 1.435(3) Å, 1.400(4) Å, 1.345(3) Å. Furthermore, the molecules are connected by intermolecular hydrogen bonds N1-H1···O1, C9-H10···Cl1, C10-H11···O1, which are 2.07 Å/145°, 2.85 Å/133°, 2.66 Å/142°, respectively. These bond lengths are within the normal range.[9–11] In the structure, the pyridine ring and benzoquinone formed a rare isoquinoline quinone skeleton, and it was further unequivocally secured that the stereo configurations of C-7 and C-13 are 7R and 13S (coincident with the reported).[9]

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Huangshan University school-enterprise cooperation project (hxkt2024095, hxkt2020118), Natural Science Foundation for Colleges and Universities of Jiangsu Province (No. 20KLB350007).

Competing interests: The authors declare no conflicts of interest regarding this article.

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