Crystal structure of methyl 4a*R**,4b*R**,8a*S**,9*R**-1-cyano-4b-hydroxy-2,4a,4b,5,6,7,8,8a,9,10-decahydrophenanthrene-9-carboxylate, $C_{17}H_{21}NO_3$

C. U. Dinesh[I], H.-U. Reißig[I] and O. Rademacher*[,II]

[I] Technische Universität Dresden, Institut für Organische Chemie, D-01062 Dresden, Germany
[II] Technische Universität Dresden, Institut für Anorganische Chemie, D-01062 Dresden, Germany

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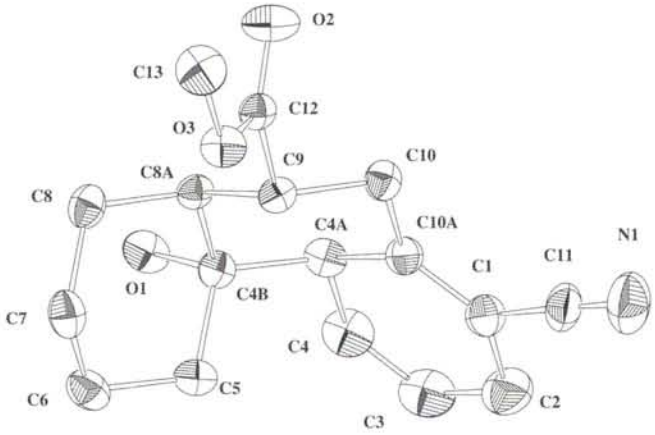

Abstract

$C_{17}H_{21}NO_3$, monoclinic, $P12_1/c1$ (No. 14), a = 10.084(1) Å, b = 13.767(2) Å, c = 11.783(3) Å, β = 113.15(1)°, V = 1504.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.133, T = 293 K.

Source of material

The title compound was obtained by samarium diiodide induced reaction of methyl 1*S**,2*R**-(2-cyanophenyl)-2-(24-oxocyclohexyl)propanoate in 60% yield (purified by chromatography on silica gel, colourless crystals, mp 424 K – 426 K, recrystalized from hexane/ethyl acetate) [1].

Table 1. Data collection and handling.

Crystal:	colourless, flate plate, size 0.04 × 0.29 × 0.58 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.87 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	42.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1827, 1725
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1263
$N(param)_{refined}$:	191
Programs:	SHELXS-97 [2], SHELXL-97 [3], ZORTEP [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.6311	0.1642	0.1780	0.080
H(2A)	4*e*	1.1275	0.1183	0.0210	0.091
H(2B)	4*e*	1.0679	0.2177	–0.0443	0.091
H(3)	4*e*	1.1136	0.2336	0.1691	0.088
H(4)	4*e*	0.9266	0.2097	0.2158	0.076
H(4A)	4*e*	0.7769	0.0806	0.1002	0.053
H(5A)	4*e*	0.7499	0.3345	0.0464	0.054
H(5B)	4*e*	0.6827	0.2963	–0.0899	0.054
H(6A)	4*e*	0.5243	0.3777	0.0386	0.065
H(6B)	4*e*	0.5450	0.4263	–0.0738	0.065
H(7A)	4*e*	0.4006	0.3097	–0.2076	0.066
H(7B)	4*e*	0.3141	0.3545	–0.1348	0.066
H(8A)	4*e*	0.2979	0.1837	–0.1394	0.059
H(8B)	4*e*	0.3677	0.2220	–0.0033	0.059
H(8A)	4*e*	0.5020	0.0921	–0.0139	0.045
H(9)	4*e*	0.5360	0.1749	–0.2227	0.045
H(10A)	4*e*	0.6481	–0.0067	–0.1116	0.053
H(10B)	4*e*	0.6716	0.0383	–0.2246	0.053
H(13A)	4*e*	0.1247	0.1010	–0.5004	0.101
H(13B)	4*e*	0.1213	0.0453	–0.3856	0.101
H(13C)	4*e*	0.2030	0.0006	–0.4622	0.101

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.9359(4)	0.0416(3)	–0.2787(3)	0.098(3)	0.107(3)	0.087(2)	0.028(2)	0.059(2)	0.011(2)
O(1)	4*e*	0.6405(2)	0.2167(1)	0.1492(2)	0.083(2)	0.050(1)	0.029(1)	–0.002(1)	0.024(1)	–0.0019(9)
O(2)	4*e*	0.3696(2)	–0.0193(2)	–0.2297(2)	0.073(2)	0.058(2)	0.049(1)	–0.023(1)	0.010(1)	0.006(1)
O(3)	4*e*	0.3099(2)	0.1093(2)	–0.3531(2)	0.045(1)	0.057(1)	0.038(1)	–0.004(1)	0.004(1)	0.001(1)

* Correspondence author
(e-mail: otto.rademacher@chemie.tu-dresden.de)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.9134(3)	0.1141(2)	–0.0851(3)	0.047(2)	0.051(2)	0.059(2)	0.005(2)	0.019(2)	0.005(2)
C(2)	4*e*	1.0474(4)	0.1635(3)	–0.0011(4)	0.051(2)	0.077(3)	0.093(3)	–0.004(2)	0.020(2)	0.005(2)
C(3)	4*e*	1.0356(4)	0.1997(3)	0.1129(3)	0.052(2)	0.073(3)	0.065(2)	–0.008(2)	–0.009(2)	0.002(2)
C(4)	4*e*	0.9224(4)	0.1871(3)	0.1400(3)	0.056(2)	0.071(2)	0.043(2)	–0.003(2)	–0.002(2)	–0.002(2)
C(4A)	4*e*	0.7878(3)	0.1386(2)	0.0564(2)	0.049(2)	0.044(2)	0.033(2)	–0.001(2)	0.008(1)	0.003(1)
C(4B)	4*e*	0.6525(3)	0.2014(2)	0.0323(2)	0.048(2)	0.044(2)	0.029(2)	–0.001(2)	0.017(1)	–0.001(1)
C(5)	4*e*	0.6666(3)	0.3023(2)	–0.0143(3)	0.053(2)	0.043(2)	0.035(2)	–0.008(2)	0.013(1)	0.000(1)
C(6)	4*e*	0.5335(3)	0.3646(2)	–0.0389(3)	0.071(2)	0.042(2)	0.050(2)	0.005(2)	0.025(2)	0.005(2)
C(7)	4*e*	0.3973(3)	0.3153(2)	–0.1267(3)	0.054(2)	0.059(2)	0.053(2)	0.017(2)	0.020(2)	0.004(2)
C(8)	4*e*	0.3825(3)	0.2152(2)	–0.0795(3)	0.046(2)	0.062(2)	0.042(2)	–0.000(2)	0.021(2)	–0.006(2)
C(8A)	4*e*	0.5144(3)	0.1507(2)	–0.0558(2)	0.045(2)	0.041(2)	0.031(2)	–0.005(1)	0.019(1)	0.001(1)
C(9)	4*e*	0.5279(3)	0.1175(2)	–0.1766(2)	0.039(2)	0.044(2)	0.030(2)	–0.005(2)	0.012(1)	–0.002(1)
C(10)	4*e*	0.6611(3)	0.0537(2)	–0.1482(3)	0.044(2)	0.050(2)	0.039(2)	–0.001(2)	0.016(1)	–0.006(1)
C(10A)	4*e*	0.7960(3)	0.1030(2)	–0.0616(3)	0.038(2)	0.043(2)	0.044(2)	0.003(2)	0.013(2)	0.003(2)
C(11)	4*e*	0.9215(4)	0.0736(3)	–0.1949(4)	0.054(2)	0.069(2)	0.074(3)	0.018(2)	0.036(2)	0.014(2)
C(12)	4*e*	0.3956(3)	0.0608(2)	–0.2545(3)	0.048(2)	0.044(2)	0.032(2)	–0.004(2)	0.020(2)	–0.002(2)
C(13)	4*e*	0.1786(3)	0.0598(3)	–0.4320(3)	0.050(2)	0.077(2)	0.054(2)	–0.007(2)	–0.003(2)	–0.012(2)

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