Crystal structure of (9a*RS*,10*SR*,10a*RS*)-8-methoxy-10-methyl-1,2,3,5,7,9a,10,10a-octahydropyrrolo[1,2-*b*]isoquinoline-10-ol, C₁₄H₂₁NO₂

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Table 1. Data collection and handling.

Crystal:	colorless cube, size 0.07 × 0.07 × 0.07 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.81 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω with $\Delta\omega = 0.3^\circ$
$2\theta_{\max}$:	61.08°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	15403, 3884
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3491
$N(\text{param})_{\text{refined}}$:	239
Programs:	SHELXS-97 [3], SHELXL-97 [4], PLATON [5]

Abstract

C₁₄H₂₁NO₂, monoclinic, $P12_1/c1$ (no. 14),
 $a = 10.597(2)$ Å, $b = 12.684(3)$ Å, $c = 9.790(2)$ Å,
 $\beta = 104.466(4)^\circ$, $V = 1274.2$ Å³, $Z = 4$,
 $R_{\text{gt}}(F) = 0.042$, $wR_{\text{ref}}(F^2) = 0.126$, $T = 173$ K.

Source of material

The title compound was obtained by samarium diiodide induced reaction of 1-[1-(4-methoxybenzyl)-2-pyrrolidinyl]ethanone analogously to the procedure described in [1,2], purified by chromatography on silica gel and recrystallized from dichloromethane/hexane; m.p. 361–368 K.

Discussion

The crystal structure proves the constitution and configuration of the title compound and also shows interesting details of the conformation. The 10-methyl group and the bridgehead hydrogens at C9A and C10A occupy equatorial positions. Bonding distances and bond angles show normal values.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1A)	4e	0.985(1)	0.359(1)	0.100(1)	0.036(3)
H(1B)	4e	0.956(1)	0.395(1)	0.243(1)	0.040(3)
H(2A)	4e	0.896(2)	0.513(1)	0.009(2)	0.060(4)
H(2B)	4e	0.837(2)	0.535(1)	0.138(2)	0.062(4)
H(3A)	4e	0.642(1)	0.457(1)	0.027(1)	0.037(3)
H(3B)	4e	0.688(1)	0.471(1)	−0.115(1)	0.038(3)
H(5A)	4e	0.554(1)	0.288(1)	0.004(1)	0.038(3)
H(5B)	4e	0.560(1)	0.287(1)	−0.160(1)	0.038(3)
H(6A)	4e	0.528(1)	0.113(1)	−0.246(1)	0.036(3)
H(7A)	4e	0.515(1)	−0.067(1)	−0.168(2)	0.049(4)
H(7B)	4e	0.649(2)	−0.055(1)	−0.222(2)	0.051(4)
H(9A)	4e	0.796(1)	−0.028(1)	0.187(1)	0.042(3)
H(9B)	4e	0.671(1)	0.1365(9)	0.162(1)	0.030(3)
H(10A)	4e	0.754(1)	0.3124(9)	0.187(1)	0.027(2)
H(11A)	4e	0.908(1)	0.165(1)	−0.047(1)	0.040(3)
H(11B)	4e	1.027(1)	0.188(1)	0.083(1)	0.038(3)
H(11C)	4e	0.963(1)	0.076(1)	0.064(2)	0.050(4)
H(12A)	4e	0.720(2)	−0.198(1)	0.201(2)	0.056(4)
H(12B)	4e	0.851(2)	−0.206(1)	0.147(2)	0.066(4)
H(12C)	4e	0.744(2)	−0.296(2)	0.105(2)	0.068(5)
H(1)	4e	0.853(1)	0.175(1)	0.327(2)	0.044(3)

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4e	0.91582(8)	0.38263(6)	0.14344(8)	0.0336(4)	0.0206(3)	0.0275(3)	−0.0052(3)	0.0072(3)	−0.0005(2)
C(2)	4e	0.8450(1)	0.48087(7)	0.0692(1)	0.0413(5)	0.0186(4)	0.0571(6)	−0.0023(3)	0.0081(4)	0.0027(3)
C(3)	4e	0.71385(9)	0.44098(6)	−0.01869(9)	0.0370(4)	0.0197(3)	0.0314(4)	0.0047(3)	0.0113(3)	0.0034(3)
C(5)	4e	0.60862(8)	0.26783(6)	−0.06312(8)	0.0263(3)	0.0258(4)	0.0285(3)	0.0000(3)	0.0040(3)	0.0036(3)
C(5A)	4e	0.63538(7)	0.15168(6)	−0.05112(8)	0.0254(3)	0.0245(3)	0.0233(3)	−0.0037(3)	0.0059(3)	0.0006(2)
C(6)	4e	0.58628(8)	0.08548(7)	−0.15628(8)	0.0305(4)	0.0338(4)	0.0253(3)	−0.0078(3)	0.0053(3)	−0.0031(3)
C(7)	4e	0.6069(1)	−0.03085(7)	−0.1468(1)	0.0415(5)	0.0308(4)	0.0344(4)	−0.0102(4)	0.0088(3)	−0.0111(3)
C(8)	4e	0.68715(9)	−0.06602(6)	−0.0069(1)	0.0345(4)	0.0210(4)	0.0401(4)	−0.0060(3)	0.0138(3)	−0.0057(3)
C(9)	4e	0.74021(8)	−0.00100(6)	0.09944(9)	0.0353(4)	0.0195(3)	0.0306(4)	−0.0031(3)	0.0084(3)	−0.0001(3)
C(9A)	4e	0.71863(7)	0.11640(6)	0.08982(7)	0.0283(3)	0.0187(3)	0.0216(3)	−0.0025(2)	0.0075(2)	−0.0003(2)
C(10)	4e	0.84718(7)	0.18195(5)	0.13425(7)	0.0259(3)	0.0182(3)	0.0178(3)	−0.0007(2)	0.0058(2)	−0.0001(2)
C(10A)	4e	0.80859(7)	0.29907(5)	0.11831(7)	0.0267(3)	0.0179(3)	0.0195(3)	−0.0008(2)	0.0079(2)	−0.0003(2)
C(11)	4e	0.94475(8)	0.14955(6)	0.05187(8)	0.0314(4)	0.0245(4)	0.0273(3)	0.0024(3)	0.0123(3)	−0.0012(3)
C(12)	4e	0.7586(1)	−0.22243(8)	0.1246(2)	0.0561(7)	0.0220(4)	0.0789(9)	0.0004(4)	0.0081(6)	0.0026(5)
O(1)	4e	0.90864(6)	0.16174(4)	0.27960(6)	0.0305(3)	0.0261(3)	0.0186(2)	0.0006(2)	0.0045(2)	0.0017(2)
O(2)	4e	0.69546(8)	−0.17396(5)	−0.00505(9)	0.0489(4)	0.0203(3)	0.0612(5)	−0.0054(3)	0.0135(3)	−0.0097(3)
N(4)	4e	0.73167(6)	0.32615(5)	−0.02545(6)	0.0276(3)	0.0183(3)	0.0223(3)	0.0008(2)	0.0068(2)	0.0022(2)

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