Crystal structure of 1,2,3α,9,10,10a-hexahydro-10aβ-methyl-3-phenyl-phenanthrene, $C_{21}H_{22}$

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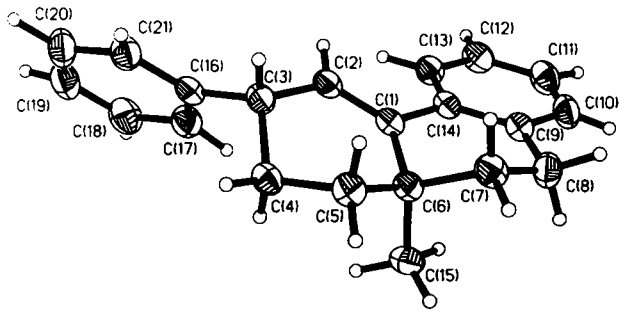

Source of material: The title compound was synthesized in 51% yield in five steps (1. H_2/Pd/C, 2. $K_2Cr_2O_7/H_2SO_4$, 3. PhMgBr, 4. TsOH, 5. *tert*-BuOK/HMPA) from the α,β-unsaturated phenanthrenone (see ref. 1) to evaluate relative differences in entropy and enthalpy to its double bond isomers (see ref. 2). A colorless single crystal, mp 386 K, was grown from ethanol at room temperature by slow solvent evaporation.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless needle, size 0.35 x 0.15 x 0.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.66 cm^{-1}
Diffractometer:	Siemens CCD
Scan mode:	ω
$T_{measurement}$:	213 K
$2\theta_{max}$:	57°
$N(hkl)_{unique}$:	2518
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	279
Programs:	SHELXS-86, SHELXL-93

$C_{21}H_{22}$, monoclinic, *C*1*c*1 (No. 9), a =7.346(2) Å, b =32.20(1) Å, c =6.646(2) Å, β =100.35(2)°, V =1546.5 $Å^3$, Z =4, $R(F)$ =0.066, $R_w(F^2)$ =0.161.

Table 2. Final atomic coordinates and displacement parameters (in $Å^2$)

Atom	Site	x	y	z	U_{iso}
H(2)	4*a*	0.670(5)	0.3909(9)	–0.188(4)	0.022(6)
H(3)	4*a*	0.483(5)	0.373(1)	–0.530(5)	0.035(8)
H(4B)	4*a*	0.265(5)	0.3234(9)	–0.543(5)	0.028(7)
H(4A)	4*a*	0.314(5)	0.314(1)	–0.305(5)	0.032(8)
H(5B)	4*a*	0.040(5)	0.358(1)	–0.382(5)	0.040(9)
H(5A)	4*a*	0.181(4)	0.391(1)	–0.453(5)	0.027(7)
H(7B)	4*a*	–0.014(5)	0.4252(9)	–0.175(5)	0.023(6)
H(7A)	4*a*	0.159(5)	0.447(1)	–0.234(5)	0.035(8)
H(8B)	4*a*	0.111(7)	0.474(1)	0.087(7)	0.06(1)
H(8A)	4*a*	0.103(7)	0.433(2)	0.184(7)	0.06(1)
H(10)	4*a*	0.357(6)	0.481(1)	0.418(6)	0.043(9)
H(11)	4*a*	0.655(8)	0.483(2)	0.579(8)	0.07(1)
H(12)	4*a*	0.872(6)	0.444(1)	0.425(5)	0.041(9)
H(13)	4*a*	0.777(5)	0.403(1)	0.135(5)	0.032(8)
H(15C)	4*a*	0.237(6)	0.366(1)	0.163(6)	0.047(9)
H(15B)	4*a*	0.039(5)	0.354(1)	0.000(5)	0.034(8)
H(15A)	4*a*	0.228(6)	0.327(1)	–0.010(7)	0.06(1)
H(17)	4*a*	0.678(5)	0.304(1)	–0.122(5)	0.034(8)
H(18)	4*a*	0.920(6)	0.255(1)	–0.143(6)	0.043(9)
H(19)	4*a*	1.012(7)	0.244(2)	–0.469(7)	0.07(1)
H(20)	4*a*	0.869(5)	0.279(1)	–0.764(5)	0.035(8)
H(21)	4*a*	0.627(6)	0.328(2)	–0.737(7)	0.06(1)

Table 3. Final 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}
C(1)	4*a*	0.4306(3)	0.39526(6)	–0.0989(3)	0.029(1)	0.0209(9)	0.028(1)	–0.0015(7)	0.0015(8)	0.0013(8)
C(2)	4*a*	0.5454(3)	0.38271(6)	–0.2213(4)	0.030(1)	0.0231(9)	0.034(1)	–0.0038(8)	0.0038(9)	–0.0027(8)
C(3)	4*a*	0.4927(3)	0.35585(7)	–0.4095(4)	0.035(1)	0.024(1)	0.029(1)	–0.0012(8)	0.0054(9)	–0.0015(8)
C(4)	4*a*	0.3032(4)	0.33571(7)	–0.4107(4)	0.036(1)	0.027(1)	0.030(1)	–0.0031(9)	0.0026(9)	–0.0034(8)
C(5)	4*a*	0.1672(3)	0.36853(8)	–0.3635(4)	0.029(1)	0.034(1)	0.030(1)	–0.0010(9)	–0.0007(9)	–0.0013(9)
C(6)	4*a*	0.2229(3)	0.38593(7)	–0.1457(4)	0.029(1)	0.0261(9)	0.031(1)	0.0001(8)	0.0030(8)	0.0006(8)
C(7)	4*a*	0.1216(4)	0.42778(8)	–0.1304(4)	0.030(1)	0.031(1)	0.040(1)	0.0028(9)	0.0016(9)	0.0018(9)
C(8)	4*a*	0.1653(4)	0.44617(8)	0.0834(4)	0.034(1)	0.034(1)	0.043(1)	0.0043(9)	0.008(1)	–0.007(1)
C(9)	4*a*	0.3689(3)	0.44507(7)	0.1748(4)	0.033(1)	0.0251(9)	0.033(1)	0.0001(8)	0.0069(9)	–0.0002(8)
C(10)	4*a*	0.4330(4)	0.46803(8)	0.3518(4)	0.048(2)	0.030(1)	0.038(1)	0.002(1)	0.010(1)	–0.008(1)
C(11)	4*a*	0.6167(4)	0.46788(8)	0.4441(4)	0.052(2)	0.034(1)	0.033(1)	–0.002(1)	0.003(1)	–0.008(1)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(12)	4*a*	0.7427(4)	0.44418(8)	0.3593(4)	0.036(1)	0.036(1)	0.035(1)	−0.0042(9)	−0.002(1)	−0.003(1)
C(13)	4*a*	0.6818(4)	0.42096(7)	0.1845(4)	0.032(1)	0.029(1)	0.036(1)	−0.0009(8)	0.003(1)	−0.0017(9)
C(14)	4*a*	0.4950(3)	0.42099(6)	0.0876(3)	0.032(1)	0.0214(9)	0.029(1)	−0.0008(7)	0.0042(9)	0.0001(7)
C(15)	4*a*	0.1709(4)	0.35481(8)	0.0114(4)	0.040(1)	0.032(1)	0.037(1)	−0.008(1)	0.008(1)	0.0023(9)
C(16)	4*a*	0.6399(3)	0.32353(7)	−0.4264(4)	0.032(1)	0.0223(9)	0.034(1)	−0.0050(8)	0.0043(9)	−0.0036(8)
C(17)	4*a*	0.7208(4)	0.30046(8)	−0.2556(4)	0.048(2)	0.030(1)	0.037(1)	0.003(1)	0.010(1)	0.0022(9)
C(18)	4*a*	0.8530(5)	0.27014(9)	−0.2712(5)	0.053(2)	0.031(1)	0.051(2)	0.009(1)	0.006(1)	0.004(1)
C(19)	4*a*	0.9059(5)	0.26287(9)	−0.4570(5)	0.048(2)	0.032(1)	0.058(2)	0.005(1)	0.009(1)	−0.011(1)
C(20)	4*a*	0.8279(4)	0.28529(9)	−0.6276(5)	0.050(2)	0.039(1)	0.043(2)	0.002(1)	0.011(1)	−0.014(1)
C(21)	4*a*	0.6939(4)	0.31559(8)	−0.6120(4)	0.042(1)	0.032(1)	0.033(1)	−0.0003(9)	0.006(1)	−0.0047(9)

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